Faculty of Engineering – Department of Civil Engineering

Nottingham Transportation Engineering Centre

A NEW METHODOLOGY FOR THE MEASUREMENT OF THE RECLAIMED ASPHALT DEGREE OF BINDER ACTIVATION

Thesis submitted to the University of Nottingham for the degree of Doctor of Philosophy in Civil Engineering

by

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ABSTRACT

Transportation on highways, mostly on asphalt pavements, is crucial for many countries around the globe. It is also known that the maintenance costs are high, pushing companies and researchers towards seeking new alternatives to improve the utilisation of materials, reduce energy consumption, streamline maintenance operations, among others. Another key factor that stands out is the environmental issues, which are directly affected by the construction and maintenance of highways, such as in the consumption of natural resources and the fuel/energy costs. Thus, it becomes realistic to reuse deteriorated materials by recycling the pavement, since all the material can be used for building a new pavement layer.

Numerous studies have been reported concerning methods of using reclaimed asphalt (RA) and the performance of mixtures containing RA. It goes without saying that in the case of high-content RA mixtures, correctly predicting the real amount of RA binder available from a selected RA is crucial to obtain asphalt mixtures which comply with design and performance standards. If the RA binder does not blend with the virgin binder as predicted, pavement performance could be compromised. Thus, this thesis is concerned with the amount of RA binder that is available, or activated, for a new mixture. This property is called the Degree of Binder Activation (DoBA) in this thesis, with the thesis divided into three stages:

- Assessing the DoBA of the selected RA's, which is the amount of "old" binder that is re-activated in new mixtures, by proposing a practical method based on the Indirect Tensile Test (ITT). The analysis is performed by comparing results between RA samples and an artificial RA produced in the lab. The method includes the evaluation of the DoBA through the conditioning and manufacturing of RA samples over a range of temperatures, from 70°C to 170°C, and mixing times, from 30 seconds to 180 seconds. Results showed that variations in production temperatures are an important factor when considering 100% RA. The same influence was not

found by increasing mixing times. The DoBA index proposed can be used to improve the binder and mixture design and is an easy tool/parameter to be determined.

- Developing design procedures for the resulting RA binders obtained by mixing old RA binder, new bitumen and an additive, while considering the DoBA. The conventional, rheological and performance-related properties of these binders were evaluated. The optimum additive dosage was defined using the penetration and the softening point tests. The performance-related properties are characterised by means of rutting, fatigue and thermal cracking resistance. Moreover, using the DoBA results, the recycled binders outcomes were used to correlate with the recycled mixtures test results in order to validate the DoBA study.

- Developing asphalt concrete mixture containing 100% RA and manufacturing mixtures in order to analyse the behaviour in terms of rutting, resistance to fatigue, stiffness modulus and moisture sensitivity. Considering the binder design, four RA mixtures were produced, assuming 100%, 75%, 50% and 15%DoBA. Part of the RA binder was assumed to be black rock (for DoBA% less than 100%) and was compensated in the mixtures by adding virgin binder. Relations are identified and established between binders and mixtures using the tests results to verify the DoBA study. The final mechanical tests proved that they can be well correlated with the designed binders. It was found that the bitumen disregarded during the DoBA design is still potentially active in the mixtures. However, the DoBA methodology can still be considered as a useful tool providing good correlation between binders and mixtures and becoming quite promising to transform the RA into a material with its own characteristics.

In conclusion, is evident that the bitumen disregarded during the DoBA design is still potentially active in the mixtures. It can also be said that other sources of RA materials may act more like a Black rock than the RA under investigation in this research due to its own characteristics, although this could only be proved by undertaking an extense experimental programme.

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DECLARATION

I declare that the contents and the work described in this thesis were performed at the University of Nottingham, Faculty of Engineering from March 2015 to August 2018. I hereby certify that this thesis is my own and has not been submitted in whole or in part to any other university or any other educational association for another degree.

Gustavo Menegusso Pires

Nottingham, 2018

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1. INTRODUCTION

1.1. Background

In developing nations, transportation has a fundamental role. For many people, from every point of view, whether economic, political or military, transportation is the most important industry in the world. An inadequate transportation system affects the entire agriculture sector, reduces national integration, compromises security, slows growth, etc. Thus, knowledge of the transport system is shown as a determining factor in the search for the best performance by everyone in today's globalised economy.

Pavement can be defined as a structure formed by multiple layers capable of withstanding the damaging action of the traffic and the environment. The performance of pavement is conditioned by a complex set of factors, among them the physical and mechanical properties of the layers that constitute it. Surface layers comprise high-cost components and deserve particular attention. Among the factors that influence the performance of an asphalt layer, can be mentioned the characteristics of the materials, dosage, compaction conditions, construction process and maintenance and restoration management.

With the frequent use of roads, pavement degradation is inevitable and mainly caused by the high volume of traffic on overloaded roads which results in the accelerated degradation of the pavement layers. Thus, any pavement restoration aims to prepare the structure and surface layers to resist the climate changes and traffic loads acting over a new life cycle, ensuring safety, comfort, and economy for drivers in the new period.

One aggravating factor in the case of highways is the high consumption of virgin materials that are required for most of the interventions to be carried out, for both maintenance and new construction. Virgin materials for pavements are mainly aggregates and bitumen. In the case of aggregates, although materials sources are extensive, they are finite. Quality materials sources that are conveniently located tend to run out quickly. Also, environmental regulations, land use policies and urban sprawl further limit the access to natural aggregate sources. In this sense, while still available, these sources are becoming increasingly scarce.

The increased awareness of the correct use of natural resources, the necessity of environmental preservation and reduction of pollutant emissions has stimulated the search for environmentally friendly alternatives to conventional pavement techniques. Although the applicable methods must conform to issues such as durability and preservation of highways, when dealing with environmental issues with its proper merit, these methods can now be classified ahead of the traditional techniques. Consequently, recycling pavement methods have gained the support of management authorities and society, for being technical alternatives that are politically correct and economically viable if appropriately exploited.

Therefore, highway authorities together with the asphalt paving industry have been working together to develop new methods and design approaches to increase recycling in pavement construction and rehabilitation. Economic and environmental benefits are the driving forces behind research in asphalt pavement recycling. Economic benefits are mainly associated with the reduction of virgin materials (aggregates and binder). Furthermore, the term "recycling" expresses a concept established in the minds of the population as a correct practice.

In recent years in Europe, transport authorities together with the asphalt paving industry are promoting research initiatives to develop guidelines, mixtures design approaches and fundamental research to define strategies and technologies to increase the use of asphalt recycling in pavement construction and rehabilitation (Mollenhauer, 2010; Kalman, 2013; Nicholls *et al.*, 2014; Di Mino *et al.*, 2015; Molenhauer *et al.*, 2015; SUP&R ITN, 2017), proving that the reuse of the material in new mixtures can present satisfactory performance. Furthermore, throughout the world, there is the inclination to produce asphalt mixtures with 100% of reclaimed asphalt (RA) as the available amount of RA increases over the time. In this context, the amount of reuse of RA in new asphalt pavements has grown to the point that it is no longer a simple green construction alternative but a common practice in many countries around the globe.

1.2. Problem statement

The highway, especially the pavement, due to its importance for transport and socio-economic activity over the long-term, should permanently present satisfactory performance. This satisfactory performance translates into the user's offer of safe, comfortable and economic traffic conditions, taking into account the precepts of optimisation of the total cost of transportation. Furthermore, there is a real need to provide infrastructure so that the flow of people and products happen in a socially and economically efficient way.

In Brazil, general indications show that 46% of roads are classed as regular, bad or very bad. If the pavement of all highways were rated good or excellent in 2012, it would have been possible to save approximately 616 million litres of diesel, or R$ 1.29 billion (about US$ 430 million), and reduce CO_2 emissions by 1.6 megatons – the main potent greenhouse gas (CNT *et al.*, 2014). Another critical issue concerning highways are accidents that are directly related to the quality of the highway, considering or not the driver's reckless imprudence, the CNT announced that nearly 187,000 accidents occurred in 2013 on federally policed Brazilian highways alone. The costs of these accidents that year was R$ 17.7 billion (about US$ 8.5 billion), a high value that if applied to the maintenance of those highways, could reduce the number of victims.

The consumption of about 1.52 billion tons of virgin aggregates and 80 million tons of binder for the production of 1.6 billion tons of asphalt around the world shows the importance of an environmentally sustainable approach regarding reducing environmental effects and natural resource consumption. The spread of pavement recycling has the potential to create about a billion $/year in economic value worldwide. Decreasing the consumption will only be possible with the application of correct processes to recycle milled asphalt material, not wasting it and also using effective techniques. Thus, the effects of environmental and natural resources consumption in production processes will be minimised (Gencer *et al.*, 2012).

When dealing with the economic and environmental aspects of the reuse of materials, recycling of pavements comes with the high potential to meet these needs. The material derived from the milling of the asphalt pavements is another material that needs to be studied because tons of this material is deposited along the highways for every restoration of a highway, often without an appropriate destination. The correct use of this material serves as an alternative to the consumption of natural aggregates now widely used in pavements.

Numerous research studies have been reported in the literature concerning methods of using reclaimed asphalt and the performance of asphalt mixtures containing RA. One of the characteristics of RA bitumen is that it is usually much stiffer than typical virgin binders due to the oxidative ageing that occurs during asphalt mixture production. It goes without saying that in the case of high-content RA asphalt mixtures, correctly predicting the real amount RA binder available of a selected RA is crucial to obtain asphalt mixtures which comply with specific design standards. If the RA binder does not blend with the virgin binder as predicted, pavement performance could be compromised. Therefore, an important question remains to be answered: How much RA binder is actually available, or activated, for a new mixture during the mixing process? A better understanding of this process may lead to an increase in the RA content in recycled mixtures.

1.3. Research aims and objectives

This research aims to propose an innovative and practical methodology to determine the range of the Degree of Binder Activation (DoBA) for selected RA's as a function of the specific material and the recycled asphalt mixture manufacturing processes, developing procedures to understand better the role of RA as a material within new mixtures, mainly connected to the old binder available for the binder blends and mixture design approaches. Moreover, the study evaluates the feasibility of using an asphalt surface (single) course for an extra-urban pavement which maximises recycling and re-use of potential waste by incorporating the maximum amount possible of RA by adding an additive for 100% RA-content.

For this purpose, the main tasks and objectives of the thesis are:

1. A literature review on recycling asphalt pavements, current design methodologies for the binders and mixtures containing RA and, the DoBA and Degree of Blending (DOB) as a technical aspect to consider for the design of high-content RA mixtures.

2. Characterise the RA's and virgin materials, obtaining the conventional and rheological properties of the binders and, RA properties to be used in the mixtures such as the grading curves and bitumen content;

3. Develop a methodology to characterise the RA to assess the proposed property "degree of binder activation" (DoBA) of the RA binder, which is the amount of "old" binder that is re-activated in new asphalt mixtures, in order to apply in further binders and mixtures design;

4. Perform a design procedure for the resulting recycled binders obtained by mixing RA binder, additive and virgin bitumen, applying the DoBA assessed in the previous objective (3);

5. Produce RA asphalt concrete mixtures with 100% RA conditioned at different temperatures, using virgin binder and an additive, considering the RA characteristics, the estimate DoBA and the designed binders for the mixtures design;

6. Characterise the recycled and control binders stiffness, fatigue and rutting resistance using different test methods and parameters and, investigate the mechanical and performance-related properties of RA mixtures regarding the main distresses affecting asphalt pavements: rutting, fatigue, stiffness and moisture damage;

7. Identify and establish relations between the binders properties tested by different methods and their RA mixture performance in order to analyse and validate the DoBA study.

1.4. Intellectual contribution of this research

Based on the literature and the willing to investigate the RA as a material with its own properties for the recycled asphalt mixtures, a methodology to characterise the RA to assess the property "degree of binder activation" (DoBA) of the RA is proposed. The DoBA is defined in this thesis as the amount of "old" binder that is re-activated in new asphalt mixtures, according to different RA manufacturing conditions. On this basis, the present investigation tried coupling the potential of the methodology to be a wide-spread method to characterise RA with the need of having a label for the DoBA of RA at different processing temperatures and times. In this sense, it is believed that identifying the DoBA can support the maximisation and optimisation of RA content in new asphalt mixtures. Thus, becomes essential to have a reasonable assumption of the DoBA of a selected RA for a specific asphalt manufacturing process.

1.5. Outline of the thesis

Chapter One: Introduction with an overview and background information about the recycling of asphalt pavements. Also includes the problem statement associated with the use of RA and detailing the research aims and objectives;

Chapter two: Presents an up-to-date literature review of the topics discussed in this thesis. These topics contributed to understanding the research field and included: methods of recycling asphalt pavements, current methodologies to design RA binders and mixtures, the RA degree of blending and activation, mechanical and rheological performance of mixtures and binders.

Chapter three: Illustrates the experimental work programme as an essential step to achieve the goal of this thesis. Laboratory work included the ageing of a virgin bitumen to reproduce an RA in the lab and the cohesion test procedures for the DoBA investigation. Also, it describes the methods used in this research for testing recycled binders and mixtures. These tests access the conventional and rheological properties from binders, and performance-related properties from binders and mixtures by means of complex/stiffness modulus, rutting, fatigue and water sensitivity.

Chapter four: In this chapter, the RA and virgin materials are characterised. The DoBA investigation is presented with the Indirect Tensile Test data analysed from three sources of RA and their respective lab created RA. Also, the influence of RA material conditioning, compaction temperature and time is evaluated. The DoBA of each source of RA is assessed, and two DoBA labelling methods are proposed. The results obtained from this chapter are to be used in Chapter 5.

Chapter five: this chapter is dedicated to the binders recycling investigation. Results from the previous chapter are applied, and the RA binders blend design is presented, one source of RA is used only. An additive was considered in the design, and four different recycled binders were produced, their conventional, rheological and performance-related properties are tested and presented along with two control bitumens (virgin binder and the RA binder). The results obtained from this chapter are to be used in Chapter 6 for the final DoBA verification.

Chapter six: The chapter presents the recycled asphalt mixtures containing 100% RA. The four recycled binders from the previous chapter are used to produce four recycled mixtures, the DoBA labels from Chapter 4 are considered together with the binders design from Chapter 5. A brief description of the mixtures design associated with the manufacturing of the recycled asphalt samples is presented. The mixtures are compared with two reference materials, a virgin control mixture and an RA control. The total of six mixtures had their mechanical performance evaluated for stiffness, rutting, fatigue and water sensitivity. The binders parameters from Chapter 5 were correlated with the results found in the recycled mixtures for the final evaluation of the DoBA.

Chapter seven: In this chapter, the main conclusions and recommendations for future research are introduced.

2. LITERATURE REVIEW

2.1. History of the use of RA

Pavement recycling is the process of reusing deteriorated asphalt mixtures to produce new compounds, taking advantage of the remaining aggregates and bitumen, removed from the road through milling, by adding rejuvenating agents, foamed bitumen, virgin binders or emulsions when needed.

According to the publication of the Federal Highway Administration – FHWA (Sullivan, 1996), the reuse or recycling of the structure of a deteriorated pavement is not new. The earliest forms of pavement recycling are dated in mid-1915 in the USA. However, recycling of asphalt pavements in its current form occurred for the first time in the 1970s, when the interest in recycling was caused by the inflation of construction prices and the oil embargo by the Organization of the Petroleum Exporting Countries - OPEC.

In response to these economic pressures, the FHWA initiated the Demonstration Project 39 - Asphalt Pavement Recycling in 1976 (Cassellius and Olsen, 1979). The DP 39, as called, showed that the recycling of asphalt pavement was a viable rehabilitation technique, and estimated that the use of RA would be approximately 15% of total production of Hot Mix Asphalt (HMA) in the 1980s. Therefore, it was expected that most of the asphalt material removed would be reused in the construction of new pavements.

According to the National Asphalt Pavement Association (Hansen and Copeland, 2016), currently, the most recycled material in the United States are asphalt mixtures with about 80 million tons per year. This is approximately two times more than the four most notably waste recycling materials, which are paper, glass, plastics and aluminium, which together add up to 40 million tons per year recycled. In Europe, the situation is variable, according to (EAPA, 2013), Germany and Italy have about 10 million tons available per year for recycling, 6.5 million tons in France and 4 million tons in Holland. From these amounts of material, the quantity reused for HMA and Warm Mix Asphalt (WMA) varies, 87% in Germany, 20% in Italy, 62% in France and 80% in Holland.

The recycling of materials from an existing pavement to produce new paving materials, results in reducing the consumption of virgin materials, cuts energy use and minimises air and water pollution (Beer *et al.*, 2015). At the same time, recycling assists in solving problems that would be the disposal of construction waste resulting from maintenance processes. Furthermore, due to the reuse of materials, the geometry and thickness of the original pavement can be maintained during the building process, and in some cases, the traffic interruption is smaller than in other rehabilitation techniques. Thus, the recycling technique has specific advantages: Reduction of construction costs; Use of aggregates and binders; Preservation of existing geometry, preservation of the environment, saving energy in the stages of production, transportation and extraction of virgin material and shorter execution time (Kandhal and Mallick, 1997).

One important consideration must be made when dealing with the possibility of using RA in new mixtures, which is the limitation imposed by each local authority or country of application. Table 1 is presented by Mollenhauer (2010) and shows the maximum allowed percentage of RA, for HMA, in recycled mixtures in some countries in Europe.

It is notable that a range from 10 to 100% has been used in different countries, depending on the pavement layer. The most significant restrictions are usually in surface courses, gradually increasing the RA content for lower layers. Alternatively, some countries (e.g. Austria, France and Germany) permit the same maximum content in all pavement layers, with Austria and Germany allowing up to 100% reclaimed asphalt to be incorporated.

Table 1: Maximum RA content for HMA in European countries (Mollenhauer, 2010)

Country	Maximum allowed percentage of RA in HMA		
	Surface Course	Binder Course	Base Course
Austria	100%	100%	100%
Belgium	25%	-	50%
Denmark	30%	30%	100%
France	40%	40%	40%
Germany	100%	100%	100%
Hungary	10%	20%	20%
Ireland	10%	50%	50%
Poland	20%	30%	30%
Portugal	10%	50%	50%
Spain	-	10% - 50%	10% - 50%
Sweden	20%	30%	30%
United Kingdom	10%	50%	50%

2.1.1. Brazilian overview

In most countries of the world the pavements wearing courses are built with asphaltic materials. In Brazil, about 95% of the paved highways are made with these materials (e.g. asphalt concrete, bituminous surface treatments, etc.) (CNT *et al.*, 2014).

The Pavement Restoration Manual from DNIT/BR (Departamento Nacional de Infraestrutura de Transportes) deals with recycling as a good solution for different problems in pavements and presents application techniques according to some pre-established design criteria: Observe failures; Determination of the probable causes of failures, based on laboratory and field studies; Design information and history of restorations; Costs; Pavement performance history; Restrictions on the geometry of the road (horizontal and vertical); Environmental factors; and Traffic (DNIT, 2006).

In Brazil, recycling was first used in 1960 in the City Hall of Rio de Janeiro, where the asphalt material was removed from urban streets with hammers and transported to asphalt plants to be remixed (Castro, 2003).

In 1980, through encouragement from DNER (Departamento Nacional de Estradas de Rodagem - National Department of Roads), in situ recycling processes, which consisted in the use of equipment for milling the pavement, then produce a new mixture and spread on the highway, started.

The first Brazilian recycling of a highway, after incentives of DNER in 1980, was in 1985 on a stretch of 100 km of the Anhanguera Highway, between São Paulo and Campinas. This consisted of milling the surface course for subsequent recycling in a drum mixer asphalt plant (Campos, 1987).

In the 90s, according to Bonfim and Domingues (1995), the first cold-recycling in situ was undertaken in urban areas in Brazil, on a stretch at Via Anchieta, São Paulo. However, according to (Pinto *et al.*, 1994), the cold-recycling in situ was first held in Brazil by the DNER in 1993 at BR-393 highway in Rio de Janeiro, using a special emulsion.

In 2004, an experimental stretch was executed on SP-147 highway, between Piracicaba and Limeira. Due to the success of this technology, it was chosen for the restoration of a further 35 km of this highway. The project included, among others, assessment of the viability of the system, mixture design, performance tests on samples prepared in the laboratory and an emulsion formulated to ensure cohesion, initial stiffness and durability (Liberatori *et al.*, 2005).

According to Domingues and Balbo (2006), drum mixers asphalt mini-plants were put in operation for asphalt recycling with regards to corrective tasks. Tests disclosed particular characteristics of an RA mixture such as high resilient modulus and tensile strength when processing 100% RA without adding rejuvenator. Better results were achieved when using rejuvenator with results close to the control mixture. The paper emphasises the recycled material potential and intrinsic difficulties and issues in recycling RA in HMA.

Sachet and Gonçalves (2008) presented a paper on technological control of cold-recycling in situ for granular bases. The research was implemented in monitored highway stretches in the state of Rio Grande do Sul in 2006, highlighting the determination of the CBR through the Dynamic Cone Penetrometer (DCP) with positive results.

Oliveira *et al.* (2010) conducted a project analysis, which adopted cold-recycling as the maintenance solution for some stretches of the BR-282, between the cities of Florianópolis and Lages in Santa Catarina, of approximately 215 km. For 76 km, where the situation was more critical, it was found that Full Depth Reclamation with grading correction and addition of Portland cement was the most suitable alternative. The authors also mention the recycling economy that was evident and presented satisfactory performance.

Paiva and Oliveira (2014) undertook a laboratory study using two samples of milled material (conventional and rubberised bitumen) to evaluate the fatigue resistance of these recycled materials stabilised with 3% of Portland cement. The tested materials were influenced by the percentage of RA and bitumen activity level in fatigue resistance. The authors concluded that recycled materials with percentages above 30% (rubber RA) by weight are detrimental to the behaviour of the recycled layer.

From these brief reports, it is possible to see that the Brazilian reality is focused on laboratory investigation of recycled mixtures. When discussing applications in the field, it is evident that the most significant works are related to cold-recycling. Therefore, the Brazilian expectation for the future is positive, studies have evolved in recent decades, and the support of companies through collaboration has developed projects with satisfactory results.

2.2. Traditional RA technologies

During the years, some valuable state-of-the-art reports and papers on asphalt recycling have been published (Kandhal and Mallick, 1997; ARRA, 2001; Karlsson and Isacsson, 2006; Nielsen, 2012; Zaumanis and Mallick, 2014). Recycling asphalt pavements is a more demanding and complex task, requiring extra knowledge and experience than producing virgin asphalt mixtures. In this sense, methods and strategies for recycling roads are needed for the reconstruction of the pavement layers. Over the last few years, different methods have been developed for RA, each one being specialised over time. Furthermore, variations in the classification as well as the descriptions of asphalt recycling methods are currently found in the literature.

The two most common classifications (Karlsson and Isacsson, 2006; D'Angelo *et al.*, 2008; Vaitkus *et al.*, 2009; Nicholls and James, 2011; Rubio *et al.*, 2012; EAPA, 2014) are based on the location of the recycling process, in-place and in-plant; or a classification parameter subdividing the recycling methods to the RA temperature and energy consumption of manufacturing (Figure 1). In this case being arbitrarily divided into:

- Cold mix asphalt (CMA): usually manufactured at ambient temperature using bitumen emulsions or foams;
- Half-Warm mix asphalt (HWMA): mixture produced at a temperature below water vaporisation, in a range of 70°C to 100°C;
- Warm mix asphalt (WMA): asphalt mixture produced at a temperature range of 100°C to 140°C;
- Hot mix asphalt (HMA): mixture produced at a temperature range of 150°C to 190°C depending on the mixture and binder types.

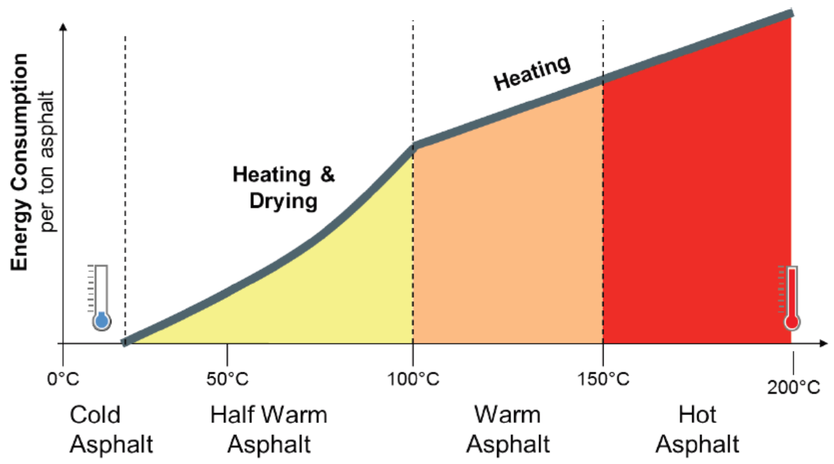


Figure 1: Classification by temperature range (EAPA, 2014)

The above description serves as an introduction to the review of recycled asphalt methods presented in the coming sections, divided into cold, warm and hot recycled mixtures.

2.2.1. Cold-mix asphalt recycling

Researchers in the asphalt industry are continually looking for methods to decrease energy consumption, greenhouse gas emissions and to improve cost-efficiency. Cold mix asphalt (CMA) is the result of various studies in this area over the last few decades. CMA usually refers to an asphalt mixture obtained by mixing bitumen emulsion, aggregates and filler at ambient temperature (Fang *et al.*, 2016). By using CMA, up to 95% of energy savings can be achieved if compared to HMA manufacturing processes (Chehovits and Galehouse, 2010; Goyer *et al.*, 2013).

The environmental benefits of cold recycling have been well documented by Jenkins (2000):

- Significantly lower energy consumption for bitumen-stabilised material production than HMA because for those aggregates, the treatment is at ambient temperature;

- Opportunity to use high percentages (up to 100%) of RA, with very minimal wastage;

- Fewer emissions during bitumen-stabilised material production than HMA due to lower aggregate temperatures;
- Health safety and environment risks due to worker exposure to the bitumen-stabilised material;
- Fewer traffic interventions and conflicts during restoration, and;
- Enhanced durability of the material, reducing future preservation requirements.

Cold mix recycling is the procedure applied to recover and reuse the material of an existing pavement without the use of heat as an artifice to perform the job. According to some researchers (Rogge *et al.*, 1992; Kandhal and Mallick, 1997), the term cold recycling is often misunderstood because it has been used to describe different procedures used with considerably different design concepts and outcomes. The field of cold recycling covers various types of applications, such as for relatively thin layers (in-site), mainly composed of asphaltic material; or for thick layers, which incorporate, besides the asphalt layer, the granular unbound layer of the pavement, a method known as "full depth reclamation" (ARRA, 2001).

The use of cold-mix recycling has been applied to correct pavement distress that involves both surface and base courses, though the method has generally been used for base courses. One of the most prevalent methods is stabilisation with emulsified bitumen, foamed bitumen, emulsion and cutbacks. In addition to asphalt materials, the other types of additives include Portland cement and fly ash (Kandhal and Mallick, 1997).

To perform the procedure of this type of recycling, the method consists of milling the asphalt layer to a certain depth. The milled asphalt material is mixed with a recycling agent which may be bitumen emulsion, foam bitumen or emulsified recycle agent, which complements the amount of binder of the blend, enabling its reuse with the characteristics defined in the project. When necessary, virgin aggregate may be added to the mixture. After the blending operation, the material is laid and the compaction of the recycled blend is carried out. Figure 2 shows the basic layout of the partial recycling process, and Figure 3 the process represented with the machines in the field.

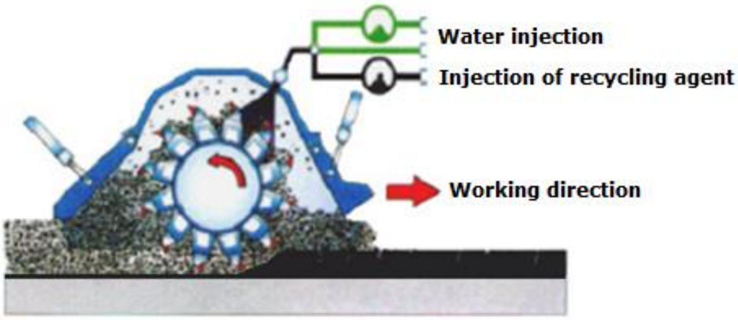


Figure 2: Basic scheme of the cold recycling in-place (WIRTGEN GmbH, 2016)

According to Kandhal and Mallick (1997) and ARRA (2001), the steps for the implementation of recycling of cold mixtures, in a general overview, are: Preparation of the area to be recycled; Milling of existing pavement; Addition of recycling agents and virgin materials; Mixing and material spreading; Levelling; and, compaction.

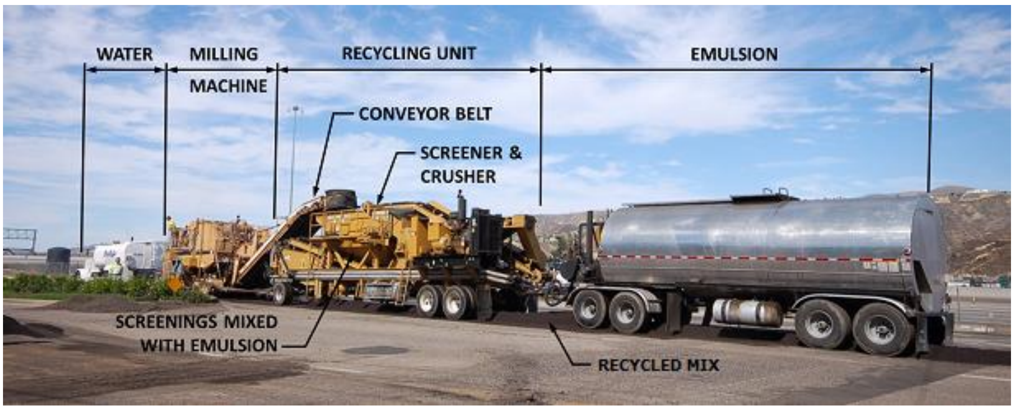


Figure 3: Cold recycling in-place with emulsion (LA County, 2015)

2.2.2. Warm-mix asphalt recycling

To reduce the energy costs and emissions from asphalt plants, the asphalt industry has developed warm mix technologies to reduce the mixing and compaction temperatures of HMA without detrimentally affecting the properties of the mixture (Rogers, 2011). Since the mid-1990s a range of techniques have been developed to reduce the mixing and laying temperatures and energy of manufacture of HMA. According to EAPA (2014), the first WMA technologies were developed in the late 1990's, where some additives were utilised in Germany, while in Norway the WAM-Foam process was developed.

Warm mix additives are used to produce WMA, which is a term given to a series of asphalt concrete mixtures which are produced at temperatures 20 to 55°C lower than the typical temperatures at which conventional HMA is produced. While these technologies allow the viscosity of the binder to be reduced at laying and compaction temperatures, they provide a reasonable and adequate coating of aggregates and are designed to give comparable performance to HMA at operating temperatures (Bonaquist, 2011; Rubio *et al.*, 2012). According to Zaumanis and Haritonovs (2010), one of the primary objectives of WMA technologies is the possibility to reduce the carbon footprint of asphalt thus supporting the demands of the Kyoto protocol for lowering greenhouse gas emissions in the atmosphere.

Regarding the recycling asphalt, the limit in the use of RA proportion is restricted due to stiffness and workability issues related to the old material. This problem can be addressed with the help of WMA additives which increase the proportion of RA used by producing mixes having the same or better properties than HMA. Those mixtures are usually provided with better workability and reduced viscosity than HMA at lower temperatures.

The WMA additives can be classified based on the technology that is used to produce the mixture, three main categories are presented:

Organic or wax additives: Waxes are a class of chemical compounds that are plastic (malleable) near ambient temperatures. Characteristically, they melt above 45°C to give a low viscosity liquid. Waxes are insoluble in water but soluble in organic, nonpolar solvents. The processes that use organic additives, waxes, amides and sulphur produce a decrease in viscosity above the melting point of the wax making it possible to produce asphalt mixtures at lower temperatures (D'Angelo *et al.*, 2008; Zaumanis and Haritonovs, 2010), consequently helping to increase the RA amount. Organic additives typically give a temperature reduction of between 20–40°C while they also improve the rutting resistance (EAPA, 2014).

Chemical additives: Chemical additives do not change the bitumen viscosity. As surfactants, they work at the microscopic interface of the aggregates and the bitumen. They regulate and reduce the frictional forces at that interface over a range of temperatures, typically between 85 and 140°C (EAPA, 2014). These surfactants improve the ability of the bitumen to coat the aggregate particles at lower temperatures rather than reduce the bitumen viscosity. The chemical additive package is used either in the form of an emulsion or added to bitumen in the mixture production and then mixed with hot aggregates (D'Angelo *et al.*, 2008; Zaumanis and Haritonovs, 2010). Chemical additives may reduce the mix and compaction temperatures by about 20 - 40°C (EAPA, 2014).

Foaming techniques: Foaming technologies use small amounts of cold water injected into the hot binder or directly in the asphalt mixing chamber. The water rapidly evaporates and is encapsulated in the binder, producing a large volume of foam. The foaming action in the binder temporally increases the volume of the binder and lowers the viscosity, which improves coating and workability. In the foaming processes, enough water must be added to cause foaming action but without adding so much that stripping problems are created (D'Angelo *et al.*, 2008; EAPA, 2014). Liquid antistripping additives are recommended for WMA production processes (D'Angelo *et al.*, 2008; Zaumanis and Haritonovs, 2010).

2.2.3. Hot-mix asphalt recycling

Hot mix recycling is the process in which RA materials are combined with new materials (virgin binders, aggregates and recycling agents), to produce HMA. The term "hot" does not denote the way that the old pavement is removed for recycling, as this process can be carried out by heating the surface to the depth to be detached and then removing the layer, or undertaken cold, using the milling process. The hot process refers to the temperature at which the mixing and compaction of the components from the RA take place as well as to the "virgin" materials to be added. To produce the recycled mixture, both drum and batch type plants can be used, and the mixtures placement and compaction equipment and procedures are the same as for regular HMA (Kandhal and Mallick, 1997).

According to Epps (1980), the proportion of RA to new aggregates depends on the mixture design, on the type of the asphalt plants and the quality of stack emission generated. Typical RA contents vary between 10% to 30%, though a maximum of 50% was already reported for drum mix plants. According to the author, the advantages of hot mix recycling are as follows:

- Important structural improvements with little or no change in thickness by improving the existing materials;
- The further right-of-way is not needed;
- Surface/binder course and base distortion problems can be corrected;
- Performance of recycled mixture is as good as conventional HMA.

The hot recycling methods are typically divided in two, Hot-in-place recycling (with some subcategories) and Hot-recycling in asphalt plant. Both methods are discussed as follows.

2.2.3.1. Hot in-place recycling

Button *et al.* (1994) define hot in-place recycling as the process of removal and in-place processing of the asphalt and granular material of a flexible pavement using heat, transforming it into a new asphalt mixture and subsequent hot compaction. Hot in-situ recycling is, therefore, a technique used to solve problems arising from defects in pavements, and thus works alongside efficiency and economy.

WIRTGEN GmbH (2016) describes the major pavement distresses and how they interfere in the performance. Also, how the recycling technique is used to solve the problem:

Permanent deformation: The probable reasons for this defect are related to the binder, where its content is too high for the mixture, known as a "fat mixture", and also with mixtures with low content of large aggregates in the grading curve. The pavement is commonly affected by the occurrence of wheel tracks, consequently, the users' safety mainly because of the water accumulated in raining days. This film of water reduces the area of contact between the pavement surface and the car tyres, compromising the vehicles stability. The hot recycling addresses this problem by adding corrective mixtures containing stiffer bitumen or a high proportion of high-quality aggregates.

Low skid resistance: When the bitumen content in the wearing course is very high, the surface of the highway becomes slippery when wet. Pavement adhesion may also be adversely affected by an insufficient percentage of aggregate. If this is the case, a high-quality corrective asphalt mixture can be added to the recycling process or by adding large aggregates in the recycled blend.

Cracking: Cracking occurs on the surface of a highway when the pavement becomes rigid or brittle. The possible causes are related to errors in the dosage of the bitumen, poor construction, overly thin surface course thickness or deficient bond between the layers of the pavement. In the recycling process, the addition of soft binders protects the new wearing course from cracking in the medium term.

According to the DNIT - Pavement Restoration Manual (DNIT, 2006), the variability of the removed materials is considered high when compared to new materials. This requires special care to be taken in designing and constructing layers with hot recycled blends.

Kandhal and Mallick (1997) highlighted the necessary steps for the hot-in-place recycling process as follows:

- Milling and/or mechanical removal of surface material;
- Material mixing with recycling agents and bitumen;
- Addition of virgin aggregates when necessary, for particle size correction;
- Laying and compacting the recycled mixture on the pavement surface.

The procedure can be performed in a single-pass or multiple-pass operations. In the single-pass process, virgin materials are mixed with the RA that comes from the milled asphalt pavement (milled in a single pass), while in the multiple-pass process, a new surface layer is added after the recycling operation with the recycled layer becoming a binder or base course.

ARRA (2001) recognises three basic types of hot in-place recycling processes: Surface recycling; Repaving; and, Remixing.

2.2.3.1.1. Surface recycling

Surface recycling is defined as a suitable rehabilitation process for cracked, brittle and uneven pavements when requiring a thin final course. The ideal pavements for this process are those whose base is stable and adequately designed. The primary purpose of the method is to eliminate surface irregularities and cracks, as well as to restore the pavement surface to the desired line, grade and cross sections to ensure proper drainage (Button *et al.*, 1994).

According to ARRA (2001), compared to the other in-place recycling processes, Surface Recycling is the least technologically complex process. Virgin aggregates and new HMA are not added during this process, so the existing pavement is only modified by the rejuvenation of the aged bitumen.

The process works as follows: the preheating unit heats the surface of the pavement, the heating and recycling unit applies more heat and scarifies the surface with a set of non-rotating teeth and recycling agents. After that, the old pavement material and the recycling agents are mixed, dispensed with a standard auger and stabilised with a screed. A pneumatic tyred roller is used to compact the RA (Button *et al.*, 1994; Kandhal and Mallick, 1997; ARRA, 2001). Figure 4 shows a surface recycling process.

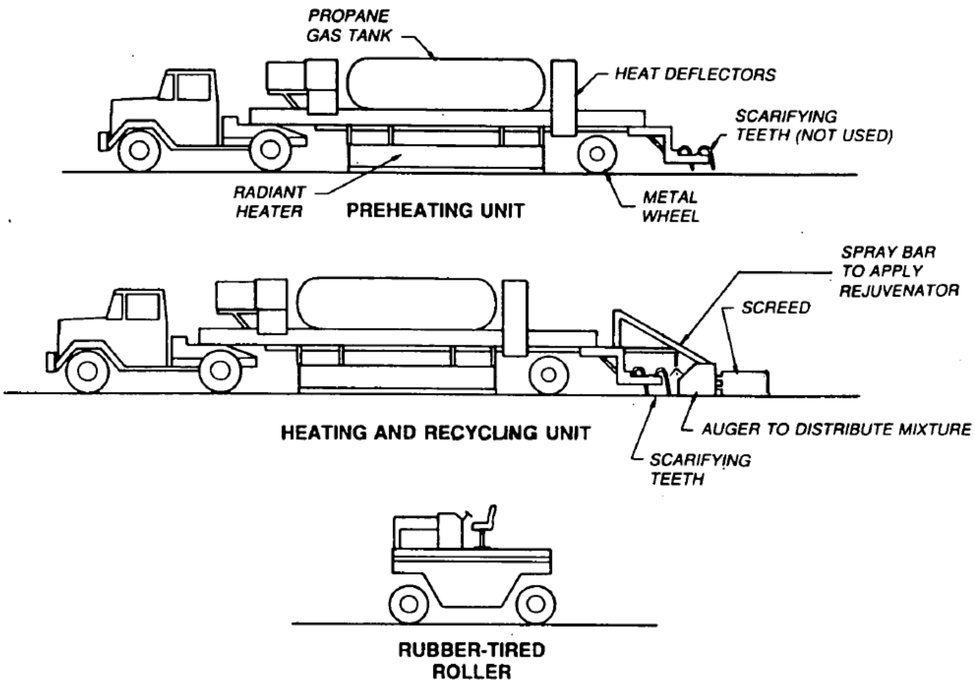


Figure 4: Surface recycling process (Button *et al.*, 1994)

2.2.3.1.2. Repaving

According to ARRA (2001), repaving is the surface recycling method combined with a simultaneous overlay of a new layer of HMA to form a thermal bond between the new and the recycled layer. Defects in the pavement, such as minor rutting, shrinkage cracking and disintegration of the mixture can be eliminated by this method. For Button *et al.* (1994) the repaving process is beneficial when the surface recycling process is not enough to restore the required comfort and safety characteristics of the pavement, or when a conventional HMA overlay is impractical or not needed.

The repaving process consists of the following steps: heating, scarifying and/or rotary milling, applying and mixing the recycling agent with scarified material, placing the recycled mixture as a levelling course using primary screed, and simultaneously placing a new HMA wearing course. Figure 5 shows the multiple-pass repaving scheme according to Button *et al.* (1994).

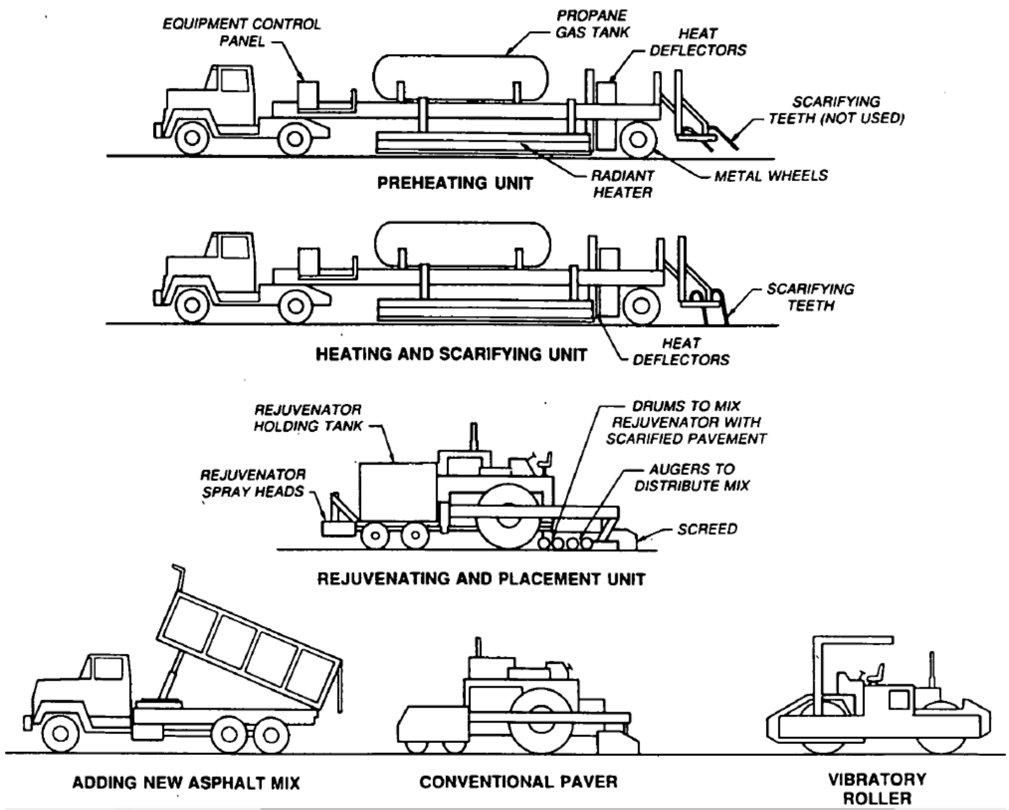


Figure 5: Repaving recycling process (Button *et al.*, 1994)

2.2.3.1.3. Remixing

This method is usually used when the repaving process is not sufficient to restore the desirable properties of the pavement concerning the comfort and safety of the highway user. In this process, aggregates and new HMA are added, which guarantees the restoration of the support capacity and stability for the recycled pavement (ARRA, 2001). Through this method, it is possible to efficiently eliminate rutting and cracks as well as correct the problem of oxidation (hardening) of the binder in the top 50 mm of the pavement surface. The simplified scheme of the remixing process is shown in Figure 6.

In the execution process explained by Button *et al.* (1994), the pavement is first heated and softened by infrared heaters in preheating units. The temperature of the asphalt is raised to levels ranging from 85°C to 105°C, the material when softened is scarified and collected. The removal can be done by fixed scarifiers, which can be followed by an additional set of milling machines and reaches depths ranging from 25 mm to 50 mm. The collected material is mixed with recycling agents and virgin aggregates to recompose the granulometry, then a set of augers spreads the material. A vibrating table is then used to partially compact the material; the final compaction process is performed by conventional methods and procedures.

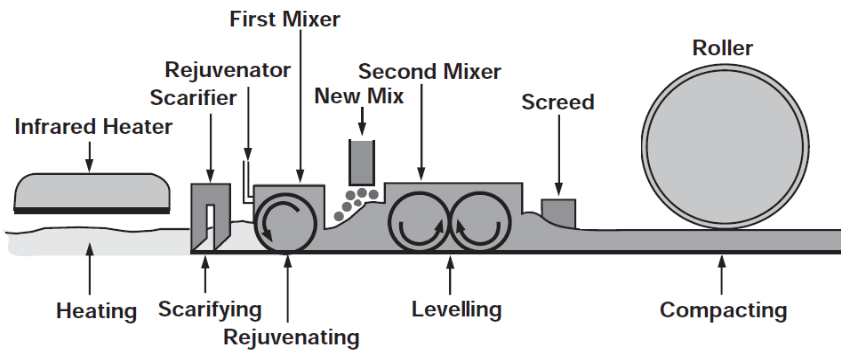


Figure 6: Remixing recycling process (adapted from ARRA, 2001)

2.2.3.2. Hot recycling in asphalt plant

According to DNIT (2006), hot mix recycled asphalt is a mixture produced in an asphalt plant with specific characteristics. The asphalt material removed from existing pavement, bitumen, additional aggregates and, if necessary, filler materials, are usually used for this method together with the recycling agent. All the materials are mixed, spread and compacted at high temperatures to recycle the pavement. This recycled material can be used as surface course, base course, regularisation or reinforcement of the subgrade.

Regarding the applications of the technique, hot mix recycling in a stationary plant consists of removal of the existing pavement by cold milling, where the milled material is loaded into dump trucks that transport the material to a hot mix plant. The recycled mixture is then produced by adding new materials such as virgin aggregates, binder and recycling agents. After that, the RA mixture is transported to the construction site to be paved.

Figure 7 shows the basic process of hot mix recycling using an asphalt plant.

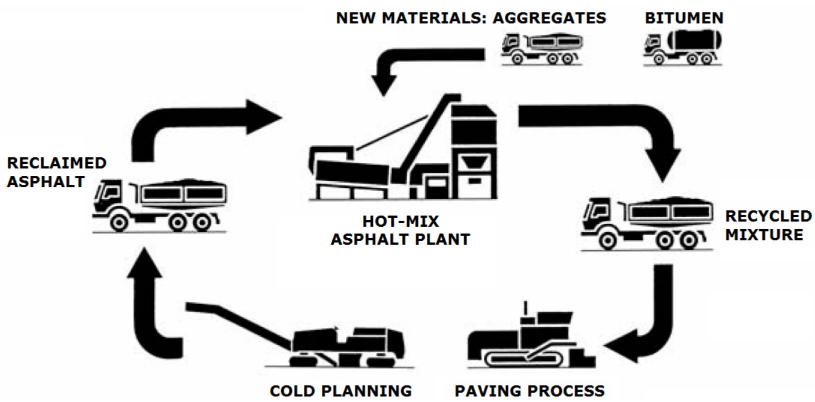


Figure 7: Hot recycling basic processes (adapted from Baptista, 2006)

According to Kandhal and Mallick (1997), there are two possible processes for obtaining hot recycled asphalt mixtures in the plant: Batch plants and Drum Mixer plants. Hot mix recycling facilities are basically modified forms of typical hot mix asphalt plants with an extra process of introducing RA. This is needed because it is not possible to dry RA along with virgin material at the same temperature. Therefore, RA is mixed with preheated virgin aggregates halfway through the drum mixing facility, or RA is added to the pug mill mixer or weighs box in batch plants.

Although batch plants present a smaller margin of error in the mixing process than the volumetric plants (drum plants) due to the intermediate control of aggregates carried out during the process, the hot mix recycling procedures using drum mixer plants have been shown to be more advantageous than batch plants. Some critical advantages of drum mixer plants in relation to batch mixing plants are:

- Portability: more portable and require shorter setup time;
- Versatility: moderately higher percentage of RA can be used;
- Production: production rates are reasonably unaffected by RA percentages;
- Mixing: A more homogeneous mixture is produced in a drum mix plant since the RA is heated and mixed with the virgin aggregate and bitumen for a more extended period compared to the mix in a batch plant.

2.2.3.2.1. Batch plants

Asphalt batch plants are the most common type in the UK. These plants have capacities ranging from 100 to over 400 tonnes per hour and produce a single batch of material at a time. The major components of a batch plant are; the cold-feed system, the bitumen supply system, the aggregate dryer, mixing tower and emission-control system (Moore, 2015).

According to Kandhal and Mallick (1997), modifications are required in the batch plants to accommodate RA since attempts to introduce RA directly with the virgin aggregates results in excessive smoke and material build-up problems in the dryer, hot elevator and screen tower. In the process, generally, a separate cold feed bin introduces the RA into the weigh hopper or the pugmill by a chute and belt conveyor. There the RA is combined with the virgin aggregate coming through the dryer and the screen decks. The temperature to which the virgin aggregates are heated depends on the characteristics of the RA material. The exhaust capacity of the pugmill or weigh hopper is essential as a significant amount of vapour is generated when the dry virgin aggregates come in contact with relatively moist and cold RA. The vapour produced can either be exhausted by the RA charging chute or an exhaust duct fitted to the pugmill or weigh hopper.

According to EAPA (2005), in batch plants there are four recycling variants: The RA can be cold fed along with the aggregates or directly into the mixing unit (cold method). To achieve higher recycling rates, the RA can be heated and introduced into the mixing unit (hot method) or heated together with the aggregates (*recyclean* method) before being raised to the mixing unit. When the RA is not heated, the aggregates are overheated so that the recycled blend has a suitable final temperature. Therefore, in these methods, only under special conditions is it possible to go beyond incorporations of 30% RA content. On the other hand, when the RA is heated separately or together with the new aggregates (*recyclean* method), recycling rates can be achieved, in the first case 70% and in the second at least 35%.

2.2.3.2.2. Drum mixer plants

According to Kandhal and Mallick (1997), the process of producing asphalt mixtures in drum mixer plants, described briefly, works as follows: the aggregates to be used are arranged, previously dosed, according to design specifications, and stored in silos according to their grading curves. They are launched on a conveyor belt that transports them to the drum. A flame continuously heats the interior of the drum from an oil or natural gas burner. The aggregates are, in a first step, heated and dried to then be mixed with the bitumen, which is injected into the downstream drum of the flame in the parallel flow drums and upstream in the counterflow drums. The recycled mixture exits the drum ready to be employed on site.

The RA material cannot be processed in conventional Drum Mixer plants, since the contact of the RA with the flame of the drum, which is intended to generate heat for drying the virgin aggregates that will be incorporated into the mixture, will result in burning the old binder from the RA. This burning results in the production of the so-called "blue smoke", which damages the functioning of the system (ARRA, 2001).

Various solutions are possible to solve this problem, for example, including higher moisture content in RA, so that it was only heated and not burned. However, measures like this would decrease production, since more energy would be needed to heat the mixture that would be wetter and more time would be spent undertaking this task. In this sense, drum mixer plants have evolved, due to the need to adapt them to even more stringent regulatory requirements (Kandhal and Mallick, 1997).

The drum mix recycling offers many advantages including portability and versatility. The facilities need minor modification to existing plants and have rapidly become one of the most popular recycling processes. The most widely used method is that with the centre entry type drum mix plant (Figure 8). Special fixtures such as flights or rings are provided to force the RA to mix with the virgin aggregates before being subjected to the high gas temperature, thus avoiding generation of the blue smoke.

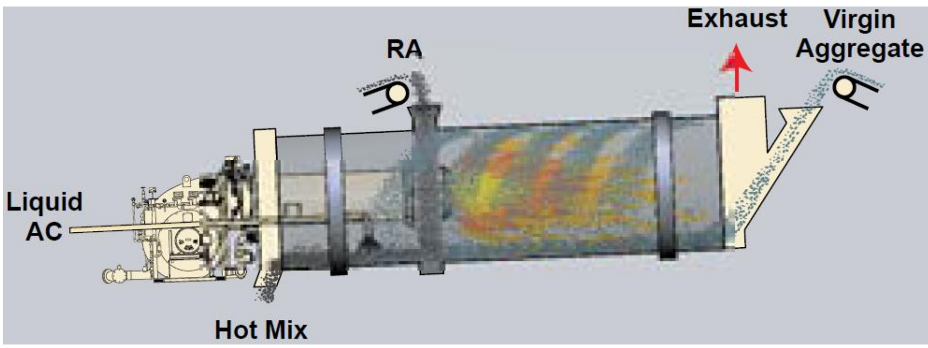


Figure 8: Counterflow drum mixer plant (Brock and Richmond, 2006)

2.3. Current practices in RA design

2.3.1. Current practices to design RA and Virgin Binders

Given that RA comes from the recovery of pavements that have been in service, considerations about its properties have to be taken into account. In most countries, the mixtures design which contain RA refers to the design procedures of mixtures containing only virgin materials. Due to this, additional specifications are defined. In order to meet these specifications and when the desired percentage of RA in the mixture is high, blending models for the RA binder and the virgin binder have to be used to design the final content of RA or to design a final binder with specific desired properties. For estimating the resulting binder consistency properties, based on the properties and binder content of the RA and the properties of the virgin binder, equations are applied according to the following methodologies.

Most countries have established their blending laws for the use of RA binder and virgin binders in RA mixtures. For example, European countries use conventional properties to design blends (EN 13108-1, 2016), while USA is exploring performance-related properties (McDaniel and Anderson, 2001).

2.3.1.1. European Methodology

Mollenhauer (2010) presents a review of the design methodology used in Europe, in accordance with the European standard (EN 13108-1, 2016). Blending models are based on the penetration and softening point properties of the RA recovered binder and the virgin binders. When RA proportions are higher than 20%, the properties of the binder blend are to be determined using Equations 1 or 2 for Penetration and Softening Point (SP), respectively:

$$a \log pen_1 + a \log pen_2 = (a + b) \log pen_{mix} \qquad \text{(Equation 1)}$$

Where,

pen_{mix} = calculated penetration of the binder in the mixture containing RA;
pen_1 = penetration of the binder recovered from the RA;
pen_2 = penetration of the added binder;
a and *b* = portions by mass of the binder from the RA (*a*) and from the added binder (*b*) in the mixture; $a + b = 1$.

$$T_{R\&B\ mix} = a \times T_{R\&B\ 1} + b \times T_{R\&B\ 2} \qquad \text{(Equation 2)}$$

Where,

$T_{R\&Bmix}$ = calculated softening point of the binder in the mixture containing RA;
$T_{R\&B\ 1}$ = softening point of the binder recovered from the RA;
$T_{R\&B\ 2}$ = softening point of the added binder;
a and *b* = portions by mass of the binder from the RA (*a*) and from the added binder (*b*) in the mixture; $a + b = 1$.

Jiménez del Barco Carrión *et al.* (2015) presented an overview of those blending methods and included another law considering the Fraass Breaking Point for the design (Equation 3). In this sense, together with penetration and SP, a different range of temperatures from conventional tests can be analysed for the design.

$$T_{Fraass\ mix} = a \times T_{Fraass\ 1} + b \times T_{Fraass\ 2} \qquad \text{(Equation 3)}$$

Where,

$T_{Fraassmix}$ = calculated Fraass BP of the binder in the mixture containing RA;
$T_{Fraass\ 1}$ = Fraass BP of the binder recovered from the RA;
$T_{Fraass\ 2}$ = Fraass BP point of the added binder;
a and *b* = portions by mass of the binder from the RA (*a*) and from the added binder (*b*) in the mixture; $a + b = 1$.

2.3.1.2. USA Methodology

The National Cooperative Highway Research program (NCHRP) Report 452 (McDaniel and Anderson, 2001) is a reference guide for mixture design and field testing technicians who deal with RA in Superpave mixtures. If a high percentage of RA is used (greater than 15 to 30 percent, depending on virgin binder grade), the RA binder will have to be considered when choosing the virgin binder grade. For lower RA contents it is not necessary to do tests because there is not enough of the oxidised RA binder present in the mixture to change the properties. Table 2 shows the selection guidelines for RA mixtures according to the recovered RA grades:

Table 2: Binder selection for RA Superpave mixtures (McDaniel and Anderson, 2001)

	Recovered RA Grade - Percentage		
Recommended Virgin Asphalt Binder Grade	**PG xx-22 or lower**	**PG xx-16**	**PG xx-10 or higher**
No change in binder selection	<20%	<15%	<10%
Select virgin binder one grade softer than normal	20-30%	15-25%	10-15%
Follow recommendations from blending charts	>30%	>25%	>15%

McDaniel and Anderson (2001) describe the procedure to obtain blending charts according to Superpave. This procedure is based on critical temperatures (high, intermediate and low) of materials and follows two methods:

Blending at a known RA content, virgin binder grade unknown (Equation 4):

$$T_{VB}=\frac{T_{blend}-(\%RA\times T_{RA})}{(1-\%RA)} \qquad \text{(Equation 4)}$$

Blending a known virgin binder, RA content unknown (Equation 5):

$$\%RA=\frac{T_{blend}-T_{VB}}{T_{RA}-T_{VB}} \qquad \text{(Equation 5)}$$

In which T_{RA}, T_{VB} and T_{blend} are the critical temperatures of RA binder, Virgin Binder and blended binder.

To construct a blending chart, the desired final binder grade and the physical properties (and critical temperatures) of the recovered RA binder are needed, plus one of the following pieces of information: The physical properties (and critical temperatures) of the virgin binder, or the percentage of RA in the mixture. Once the RA binder has been extracted and recovered, its properties need to be determined. The binder must be tested in the Dynamic Shear Rheometer (DSR) at a high temperature as if it were an original, unaged binder. Then the remaining RA binder is aged in the Rolling Thin Film Oven Test (RTFOT) and is tested in the DSR and Bending Beam Rheometer (BBR) to determine the critical temperatures.

2.3.1.2.1. High Critical Temperatures

The $T_{c(High)}$ is based on DSR testing on the original bitumen or aged at RTFOT. $T_{C(High)}$ is chosen between $T_{C(High)1}$ and $T_{C(High)2}$ as the most restrictive value (the lower one).

The slope (a) of the stiffness-temperature curve of the unaged binder is determined as ΔLog(G*/sin δ)/ΔT and Equation 6 is applied:

$$T_{c(High)1} = \left(\frac{\text{Log}(1.00)-\text{Log}(G_1)}{a}\right) + T_1 \qquad \text{(Equation 6)}$$

Where:

G_1 = the G*/sinδ value in kPa at a specific temperature T_1

a = the slope of the stiffness-temperature curve

The slope (a) of the stiffness-temperature curve of the RTFOT binder is determined as ΔLog(G*/sinδ)/ΔT and Equation 7 is used:

$$T_{c(High)2} = \left(\frac{\text{Log}(2.20)-\text{Log}(G_1)}{a}\right) + T_1 \qquad \text{(Equation 7)}$$

Where:

G_1 = the G*/sinδ value in kPa at a specific temperature T_1

a = the slope of the stiffness-temperature curve

Although any temperature (T_1) and the corresponding stiffness (G_1) can be selected, it is advisable to use the G*/sinδ value closest to the criterion (1.0/2.2 kPa) to minimise extrapolation errors.

2.3.1.2.2. Intermediate Critical Temperatures

The critical intermediate temperature is determined by performing intermediate temperature DSR testing on the RTFOT RA binder. The slope of the stiffness-temperature curve of the RTFOT binder is determined as ΔLog (G*/sinδ)/ΔT and Equation 8 is used:

$$T_{c(Int)} = \left(\frac{\text{Log}(5000)\text{-Log}(G_1)}{a}\right) + T_1 \qquad \text{(Equation 8)}$$

Where:

G_1 = the G*/sinδ value in kPa at a specific temperature T_1

a = the slope of the stiffness-temperature curve

Although any temperature (T_1) and the corresponding stiffness (G_1) can be selected, it is advisable to use the G*/sinδ value closest to the criterion (5000 kPa) to minimise extrapolation errors.

2.3.1.2.3. Low Critical Temperatures

The critical low temperature is determined by performing BBR testing on the RTFOT RA binder at low temperatures, the stiffness and m-value results obtained are used to calculate the $T_{c(Low)S}$ or $T_{c(Low)m}$. The higher of the two calculated temperatures is selected to represent the low critical temperature. The $T_{c(Low)S}$ and $T_{c(Low)m}$ are defined by the Equations 9 and 10, respectively:

$$T_{c(Low)S} = \left(\frac{\text{Log}(300)\text{-Log}(S_1)}{a}\right) + T_1 \qquad \text{(Equation 9)}$$

Where:

S1= the stiffness value in MPa at a specific temperature T1

a = the slope of the stiffness-temperature

$$T_{c(Low)m} = \left(\frac{0.300\text{-}m_1}{a}\right) + T_1 \qquad \text{(Equation 10)}$$

Where:

S1= the stiffness value in MPa at a specific temperature T1

a = the slope of the stiffness-temperature

Once the critical temperatures of the recovered RA binder are known, two blending approaches may be used as showed previously, applying Equation 4 or 5. Both approaches use the performance grade to determine the final blended binder.

2.3.1.3. Binders specification

According to Southern (2015): "A specification can be considered to be a detailed description of a given material, enabling parties on either side of a commercial transaction to understand what can be expected in performance terms at the point of transfer of ownership. In the context of bituminous binder specifications, the specification should address properties that are relevant to the end user of construction products in which they are used."

As briefly mentioned previously in this thesis, different bitumen specifications are used. The specification systems used can be described as either empirical or performance based. Those based on empirical testing such as penetration, softening point (e.g. Europe, Africa, Brazil, many parts of Asia) and viscosity (e.g. Australia, New Zealand, Mexico, Chile) have been used by many countries over the years, and the performance-based system (PG grading) used in USA and Canada which is well established in product standards (Hunter *et al.*, 2015).

As mentioned, in Europe, bitumens are typically specified by their penetration value (e.g. pen 40/60, pen 150/200, etc.) expressed in decimillimeters which are known as penetration grading (EN 12591, 2009) – Appendix 1 presents the EU specification for paving grade bitumens. The reason for the penetration test comes from its simplicity and the low cost of its apparatus.

Recognising the deficiencies in the system based on empirical testing, where the specifications classify different bitumens with the same grading when in fact these bitumens may have different temperature and performance characteristics, in 1987 the Strategic Highway Research Program (SHRP) started developing new tests for measuring the physical properties of bitumens. The significant result of this research is the Superpave binder specification (Asphalt Institute, 1994).

The Superpave tests measure physical properties that can be related directly to field performance. The bitumen tests are conducted at temperatures that are encountered by in-service pavements. These tests and specifications are specifically designed to address pavement performance parameters such as rutting, fatigue and thermal cracking. Then, critical temperatures for those phenomena are calculated, and the bitumens are denominated by Performance Grade (PG), followed by two temperatures: high critical temperature and low critical temperature (usually negative value). The Appendix 2 shows the Performance Graded Asphalt Binder Specification from MP-1 (AASHTO MP-1, 1998).

2.3.2. Current practices to design RA mixtures

Historically, specifications limiting RA in HMA have been based on RA-percentage by weight of aggregate or by weight of the total mix. However, the primary issue with higher RA content in asphalt mixes is the amount of binder replacement available since the use of RA can reduce the need for a virgin binder and impact the binder properties. For this reason, most of the recommendations for recycling are based on binder methodologies and testing, and some of them have already been presented.

Although the design methodologies to use RA in asphalt mixtures are based on bitumen properties, some considerations and recommendations can be observed in the mixtures design. According to Copeland (2011), the standard practice and specifications for designing asphalt mixtures according to the Superpave mix design system are the standards AASHTO M323 (2012) and AASHTO R35 (2012). The standards specify the quality requirements for the bitumen, aggregate, and HMA for Superpave mixture design as well as the design evaluation based on volumetric properties of the HMA such as air voids, voids of mineral aggregate and voids filled with bitumen (Al-Qadi *et al.*, 2007).

According to McDaniel and Anderson (2001), one decision that must be made primarily in the process is the approximate amount of RA that is intended to use. This decision is made based on the current state specifications, the aggregate gradation and properties, economics, and, sometimes, the binder properties. The amount of RA to include in the new mixture may be limited by different factors such as specification limits for mixture and plant type; gradation; aggregates and bitumen properties; heating, drying and exhaust capacity of the asphalt plant; moisture content of the RA and aggregates; conditioning temperature of the aggregates and RA.

The mixture design process incorporating RA is similar to the mixture design for all virgin materials. Once RA has been characterised, it can be combined with virgin aggregate for calculation of the gradation for mixture design purposes. During the analysis, RA is treated as a material with its proper stockpile of aggregates. The compound properties for gradation, specific gravity and characteristics are used in defining suitability of the combined aggregates (Copeland, 2011).

To account for the presence of binder in the RA material, the weight of RA aggregate is calculated using Equation 11 (McDaniel and Anderson, 2001; Al-Qadi *et al.*, 2007):

$$M_{dryRA} = \frac{M_{RAAgg}}{(100 - RA_{Bit\%})} \times 100 \qquad \text{(Equation 11)}$$

Where,

M_{dryRA} = mass of dry RA;
M_{RAAgg} = mass of RA aggregate and binder;
$RA_{Bit\%}$ = RA binder content.

Indicated as being of critical importance by Al-Qadi *et al.* (2007), in the mixture design procedure of HMA incorporating RA materials the required amount of bitumen at 4% air voids is reduced by the amount of binder in the RA stockpile based on solvent extraction or ignition. For example, if the design binder content is 4.5% and the bitumen in the RA is estimated at 3.3%, the virgin binder to be added is 1.2%. This has been reported to be inaccurate and could result in a wrong HMA mixture formula. The authors mention that, since many volumetric calculations are based on the bitumen content, the values of voids of mineral aggregate, voids filled with bitumen, and effective bitumen content may also be erroneous. Hence, the HMA may be vulnerable to durability cracking and premature failure.

For Copeland (2011), the percentage of bitumen in RA needs to be considered when determining the trial binder content. The mixture trial bitumen content is calculated or estimated by experience during the trial blend analysis. Thus, the amount of binder in RA is considered when determining how much virgin binder is required. Authors conclude that it may be necessary to adjust the virgin binder grade (Superpave binder specs) when RA is used in the new mixture to achieve the appropriate grade.

According to West *et al.* (2013), one essential property that must be determined for the RA is the specific gravity of the RA aggregate (Gsb). The RA aggregate Gsb is critical to an accurate determination of voids of mineral aggregate. For high-RA content mixture designs, the best method to recover the aggregate to determine the RA specific gravity is to use a solvent extraction method and then test the coarse and fine parts of the recovered aggregate. McDaniel and Anderson (2001) present another approach to estimate the specific gravity, which involves determining the maximum theoretical specific gravity and the RA bitumen content. Important to know that all methods are likely to cause small errors in the specific gravity results, especially when the RA-content gets closer or above 50%, where the net effect increases as does the errors. This uncertainty is one reason why it may be suitable to perform additional performance related tests on high-RA mixture designs to assure resistance to rutting, moisture damage, fatigue cracking and thermal cracking.

Copeland (2011) highlights that the gradation of the RA particles is not the original gradation of the aggregate used in RA because the binder film on RA adds to the dimension of the aggregate. However, the original gradation of the recovered RA aggregate has been used for design purposes. Typical design software (i.e., spreadsheet programs) accounts for the differences in the batching material gradation and the "true" gradation of the RA material as well as for the bitumen contained in the RA material. Further discussions about the subject are presented considering the amount of binder available from RA materials, as well as being one of the main objectives to be answered in this thesis.

2.3.3. Degree of Blending and Degree of Binder Activation of the RA

Numerous research studies have been reported in the literature concerning methods of using RA and the performance of asphalt mixtures containing RA as presented in the previous sections. In order to be considered as a sustainable solution, high-content RA mixtures should meet at least the same requirements valid for conventional mixtures. However, RA is a complex material which is different from the traditional components used in asphalt mixtures. One aspect that shows a fundamental role in this process is the lack of fundamental understanding of some of the mechanisms involved in the RA mixing with the other components such as recycling agents, rejuvenators and binders. This section aims to provide a critical review of information, methodologies and terminologies to answer the question of how to determine the degree of blending between aged asphalt binders and additives as well as the degree of RA binder activation.

Firstly, in the literature, several definitions (Table 3) have been given to define the primary theme addressed in this section and often these led to confusion and overlapping of concepts.

Table 3: Binders blending literature definitions

Definition	Author reference
Amount of re-activated binder	(Stimilli *et al.*, 2015)
Binder transfer	(Zhang *et al.*, 2015)
Blending between the aged and the new bitumen	(Delfosse *et al.*, 2016)
Blending efficiency	(Bowers, Huang, Shu, *et al.*, 2014; Bowers, Moore, *et al.*, 2014; Ding *et al.*, 2016)
Blending of RA into HMA mixtures	(Huang *et al.*, 2005)
Blending ratio	(Delfosse *et al.*, 2016; Zhao, Huang, Shu, Moore, *et al.*, 2016)
Blending status	(Zhao, Huang, Shu and Woods, 2016)
Recycled binder mobilisation rate	(Zhao *et al.*, 2015)
Level/amount of binder blending	(Al-Qadi *et al.*, 2009)
Degree of partial blending	(Shirodkar *et al.*, 2011)
Degree of blending (DOB)	(Gaitan, 2012; Mogawer *et al.*, 2012, 2013; Navaro *et al.*, 2012; Booshehrian *et al.*, 2013; Rad, 2013; Shirodkar *et al.*, 2013; Coffey *et al.*, 2013; Rinaldini *et al.*, 2014; Kriz *et al.*, 2014; Norton *et al.*, 2014; Castorena *et al.*, 2016; Cavalli *et al.*, 2017)

Hence, this section intends first to provide clear terminologies associated with definitions of the general phenomenon occurring when RA is mixed with another materials. The goal is to provide a common ground for the literature review as well as including some of the definitions mentioned above and aims at explicitly identifying two components:

Degree of binder activation (DoBA): It is well known that when RA is incorporated within the manufacturing of mixtures, together with virgin bitumen and other additives, a portion of the aged binder surrounding the RA aggregates, becomes available and participates with the other components in providing the new mixture with the final mechanical properties. In this thesis it is considered that the interaction between the RA and the new components causes a certain "activation" of the aged binder and for simplifying practical implementation this is considered to be a property of the RA. So the DoBA can be defined as the proportion of RA binder that is activated in the RA and can be considered a unique property of the RA (Figure 9). DoBA is only influenced by the composition of the RA and the processing conditions associated with the use of the RA such as conditioning and mixing temperatures and times. Moreover, active binder is the amount of aged binder that does not need replacement with a new agent. This means that if an RA with 6% binder content is considered having a DoBA of 75%, then 4.5% of the RA binder can be considered active in the new design. Assuming the DoBA of the selected RA is crucial to obtain mixtures complying with specific design and performance standards. In fact, mixtures design methodologies naturally aim at estimating the optimum binder content of a given asphalt mixture, hence the risk is to over or under-dosing the bitumen for the new mixtures, which can be directly affected by the DoBA assumed.

Degree of blending (DOB): The difference between DoBA and DOB is that the activation, DoBA, can be influenced by processing conditions, additive and fresh binder, but it is a property of the RA while the DOB is a parameter of the final asphalt mixture and it is defined only in the presence of additional materials (e.g. bitumen, rejuvenator, additives.) (Figure 9). Regarding the DOB, usually, two significant processes are involved when binder blending is considered in asphalt mixtures with RA addition: achieved by mechanical mixing with the compatibility between the virgin and RA binders; and binder blending after initial contact, achieved predominantly by diffusion (Karlsson and Isacsson, 2006).

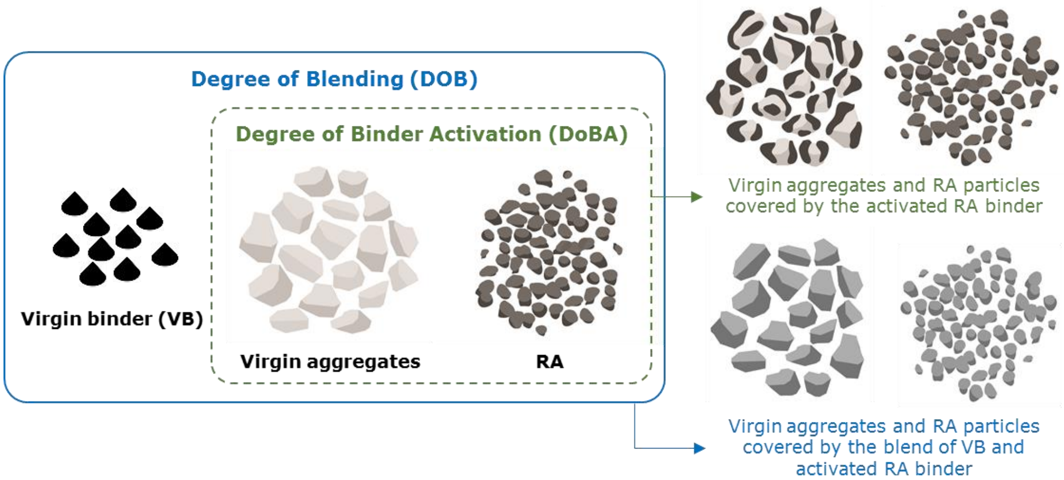


Figure 9: Basic representation of the DOB and DoBA

As a result, the critical question remains to be answered: How much binder is activated and available from RA within new asphalt mixtures during the manufacturing process? Moreover, how does this blend with fresh binders? There is a reason for why these questions are still un-answered. Assessing the DoBA of an RA and the DOB of a blend are multi-variables problems with several factors influencing the outcome. This section presents the results of a literature review of the investigations evaluating the extent of blending between RA and fresh binder and identifies the methodologies used so far to assess the DoBA and DOB.

2.3.3.1. Literature review on DOB and DoBA

Generally, the amount of RA binder that is mobilised is known as the DOB/DoBA, and there are two opposing theories associated with it, "full blending" and "black rock". Full blending assumes that 100% of the RA binder is reactivated and becomes part of the new mix whereas "black rock" states that 0% of the binder will be active and that the recycled aggregates are "black rocks".

Some important studies have been developed in this area of research to deepen the understanding of this phenomenon that occurs during the mixing process. Methodologies used by several researchers to determine both the DoBA and DOB can be grouped in different macro-areas, an overview of those have been identified and presented in three distinctive groups: Mechanical methods; Mechanistic approach; and, Chemical and visualisation methods. Some of the research in this area is explained in detail in order to highlight the importance of this phenomenon.

2.3.3.1.1. Mechanical methods

Huang *et al.* (2005) analysed a way to find out how much aged RA asphalt binder would be blended into virgin binder under normal mixing conditions. The RA was blended with coarse aggregates, RA in size smaller than #4 sieve and coarse aggregates all +#4 sieve particles in order that the aggregates could be distinguished. According to authors, the aggregates in the RA were limestone mostly, the bitumen content obtained was 6.8%, the bitumen film thickness was calculated being equal to 5.3 microns and the virgin binder utilised was a conventional PG 64-22. Huang et al. used three RA proportions (10%, 20%, and 30%), the blending was performed at 190°C mixed for 3 minutes. The binder content of RA reduced from 6.8% to 6.0%, which represented about 11% of binder loss due to mechanical mixing. These results indicated that aged binder tended to "stick" with the RA aggregate and a small portion (about 11%) of the RA binder was available to blend with virgin asphalt.

Shirodkar *et al.* (2011) developed a study to determine the degree of partial blending of reclaimed asphalt binder for HMA using 25% and 35% of RA. The authors used a gap gradation to separate the RA and the virgin aggregates at the #4 sieve, where the RA was sieved below the #8 (2.36 mm) sieve and the virgin aggregates were all above the #4 (4.75 mm) sieve. This gradation was created so that the degree of blending could be determined.

Figure 10 shows the coating study applied by these authors with the increase in weight of the virgin aggregates due to the coating by the RA binder after mixing. However they comment that the reduction in weight of the RA aggregates may be due to four things (a) loss of moisture content, (b) RA binder lost to bucket, (c) loss of fine particles of RAP during mixing, and (d) transfer of RA binder to virgin aggregates.

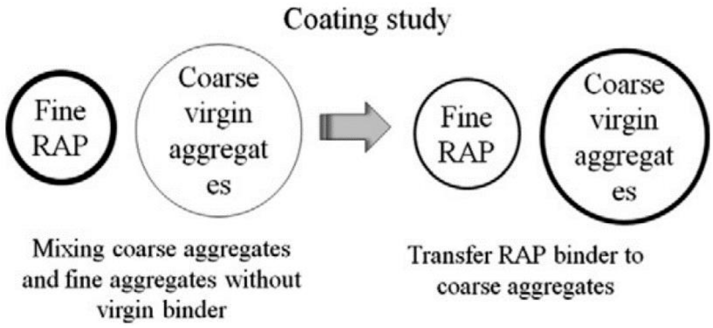


Figure 10: Schematic representation of coating study (Shirodkar et al., 2011)

This coating study without the virgin binder only provides an estimate of the partial blending because a portion of the RA binder will also coat the RA aggregates. This portion cannot be measured in this process and the impact of the presence of virgin binder on the degree of partial blending cannot be detected. The RA binder transfer for 25% and 35% RA-content averaged 24% and 15%, respectively. According to authors, these values of binder transfer seem much lower than what is expected and the lower binder transfer for higher RA content may be result of the RA binder sticking in RA materials.

The study used a design supplied by a plant where the optimum binder content was 4.8%. The approximate RA binder transfer from the above coating study was used to determine the amount of virgin binder content. For the binder properties, after mixing at 175°C, the virgin aggregates were separated from the RA aggregates. The binder from a separate mixtures was extracted and recovered. The RTFO G*/sin(δ) of the extracted binder was determined at selected temperatures for the mixture prepared with PG 70-28 and PG 58-28 binder. From the testing results, they found out that binder testing temperature did not affect the determination of the degree of blending.

All other tests were carried out at the same temperature. The $G^*/\sin(\delta)$ of RTFOT binder was selected mainly because the binder properties at high temperatures are usually more sensitive to blending than low-temperature test results. Equations 12 and 13 below were used to determine the results.

$$\text{Blending Ratio}=\frac{\left(\left|\left(\frac{G^*}{\sin(\delta)}\right)_{\text{blendbindervirginagg}}\right|-\left|\left(\frac{G^*}{\sin(\delta)}\right)_{\text{blendbindeRAagg}}\right|\right)}{\left(\left|\left(\frac{G^*}{\sin(\delta)}\right)_{\text{virginbinder}}\right|-\left|\left(\frac{G^*}{\sin(\delta)}\right)_{\text{RAvirginbinder0blend}}\right|\right)} \qquad \text{(Equation 12)}$$

$$\text{Degree of Blending }(\%) = 100 \times |1 - \text{Blending Ratio}| \qquad \text{(Equation 13)}$$

The authors concluded that the DOB for 25% RA by weight of aggregates in the case of PG 70-28 virgin binder was 70%. The degree of blending for 35% RA with PG 58-28 virgin binder was 96%. The values determined are higher than that determined by the coating study. Comparing results (blending and coating study), the influence of the presence of the virgin binder can be seen, resulting in a much higher degree of blending.

Huang *et al.* (2005) considered 10%, 20% and 30% RA-content in their coating study and concluded that it affected only a small movement of aged binder to the virgin aggregates, about 11%, regardless of the RA content. Similar results were obtained from the research conducted by (Bressi *et al.*, 2016) where significant mobilisation of RA binder was not observed, even by using significantly high-RA content (50% and 90%).

Another technique was applied by Huang *et al.* (2005) called "Staged Extraction and Recovery", to determine how much virgin binder was coating RA aggregates. Extraction was performed on four batches of staged extraction, representing four different layers of asphalt, where the fourth is close to the aggregate. Figure 11 presents a schematic flowchart for the staged extraction.

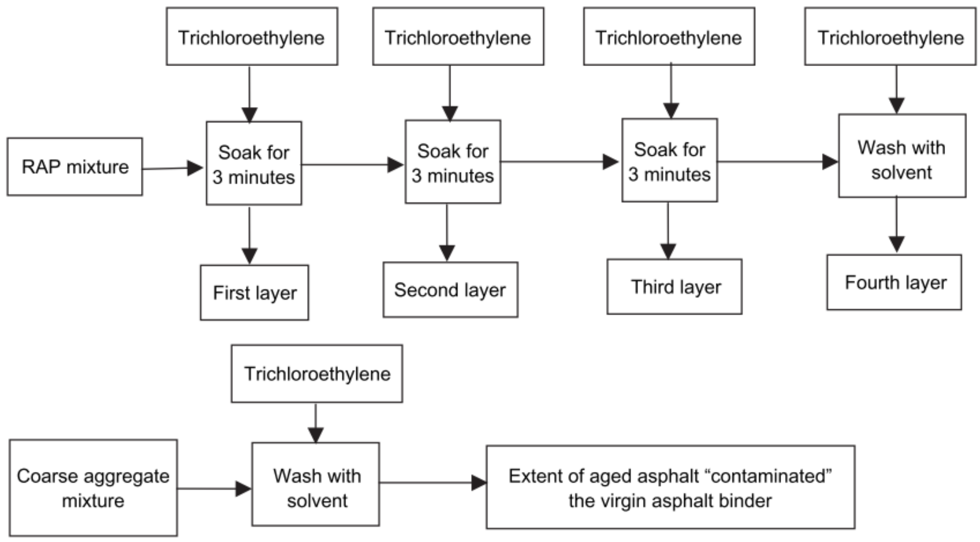


Figure 11: Flowchart for staged extraction from Huang *et al.* (2005)

In order to simulate the plant mixing, 20% RA was blended with virgin aggregates and PG 64-22 virgin binder (190°C and 3 minutes mixing). Virgin mixtures consisted of coarse aggregates and RA consisted of only fine particles separated afterwards. The rotational viscometer test was used to characterise the rheological properties of the binders at high temperature (135°C). The DSR was used to characterise binder properties at different temperatures. The viscosity and complex modulus increased going from the outside layers to the inside. In Layers 3 and 4 it was much stiffer than the binder in Layers 1 and 2.

The authors concluded that mechanical blending affects only a small portion of RA binder; the staged extraction can be used to analyse the binder from different layers around the aggregates; and, only a small proportion of aged RA binder participates in the re-mixing process, while the remainder forms a stiffer coating around RA aggregates.

Rinaldini *et al.* (2014) conducted rheological tests using the DSR on bitumen blends obtained using staged extraction process and constructed G* master curves. Based on research results, they confirmed that certain amounts of DOB appeared in the mixing phase. Liphardt *et al.* (2015) went a stage further from the determination of the DOB based on G* value. They used multiple stress creep recovery (MSCR) test to assess the DOB. The authors concluded that RA and virgin binder were not wholly blended, although the DOB was not quantified. Moreover, it was proven as an useful method for determination of the DOB, especially in the case of blending polymer modified and neat bitumen. Further, it was concluded that the increase of G* seems to be an indicator of the DOB trend confirming findings from other researchers (Huang *et al.*, 2005; Bowers, Moore, *et al.*, 2014; Liphardt *et al.*, 2015).

Bowers, Moore, *et al.* (2014) investigated the influence of mixing time, temperature and the inclusion of WMA additives to access the virgin and RA binder blending. A coarse virgin aggregate and a fine RA were blended with a virgin binder in defined mixing times from 30 seconds to 300 seconds and range of temperatures between 130°C and 160°C. Then, the aggregates were separated, and the binders were recovered from each case, the rheological properties were investigated using the DSR.

The virgin bitumen used was a PG 64-22 binder, RA from an unknown source, and a virgin aggregate. The mixtures created consisted of 65% RA and 35% virgin aggregate, 0.91% of the virgin binder was added by weight of aggregate to achieve the target binder content. A surfactant based WMA additive and a wax based WMA additive were added to the PG 64-22 virgin binder for their respective mixtures.

Figure 12 shows the master curve data (25°C) observing the different mixing times. It is noticeable that there is a significant gap between the virgin and RA binder curves. The authors mentioned that at 30, 60, and 105 s there is no visible difference between the coarse master curves, but when the time reaches 150 s there is an evident increase in the G* at lower frequencies. This may indicate an increase in the presence of RA binder in the binder recovered from the coarse aggregate.

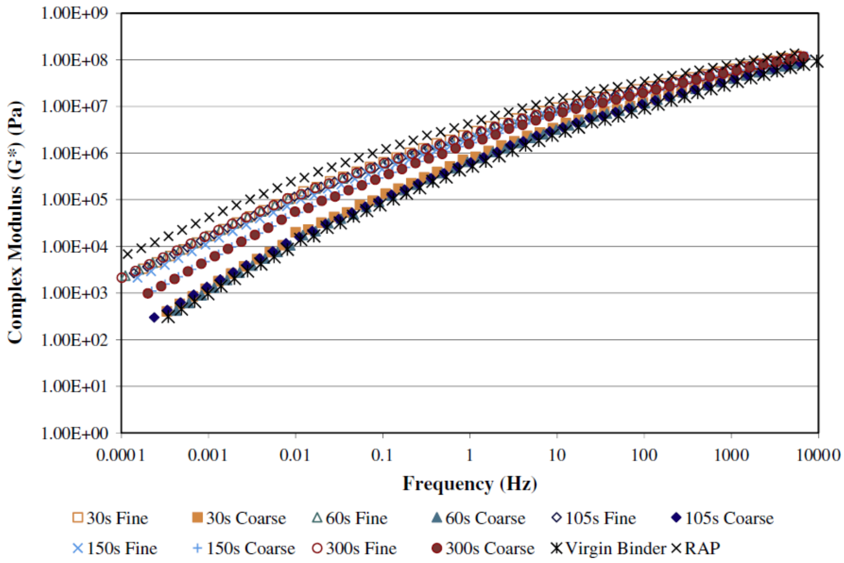


Figure 12: G* master curves changing mixing times (Bowers, Moore, *et al.*, 2014)

Figure 13 shows the master curve data observing the influence of mixing temperature. It can be seen that the coarse aggregate increases in complex modulus as the mixing temperature increases. Also, the fine aggregates master curves do not present any definite distinction; only that they were slightly softer than the RA binder which may indicate the presence of a virgin binder.

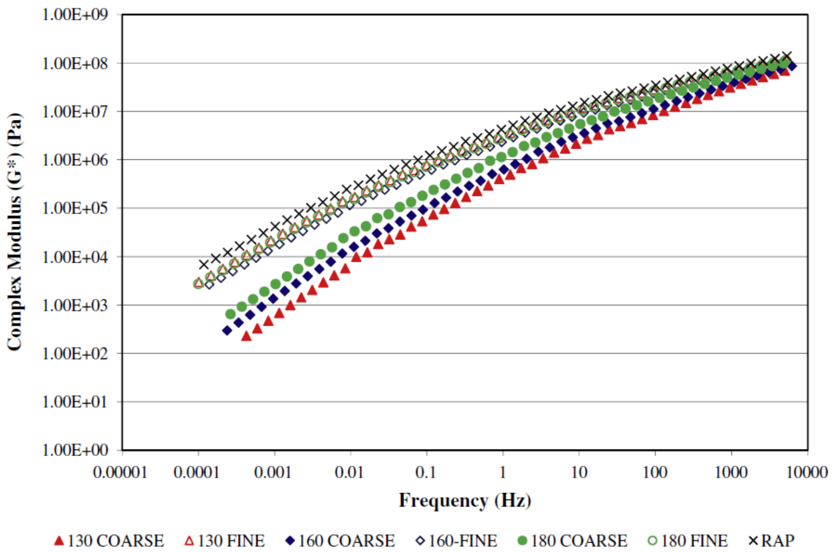


Figure 13: G* master curves changing temperatures (Bowers, Moore, *et al.*, 2014)

The evaluation of WMA additives yielded interesting results, where in both cases, the WMA additive increased the blend ratio. Figure 14 shows the master curves, the Surfactant based mixture had a blend ratio equivalent to that of the 160°C, 150 s mixture even though it was mixed for less time (105 s) and at a lower temperature (130°C). The researchers did not notice more significant progress in results when Wax based additive was used, indicating that further investigations are recommended by testing higher temperatures and mixing times.

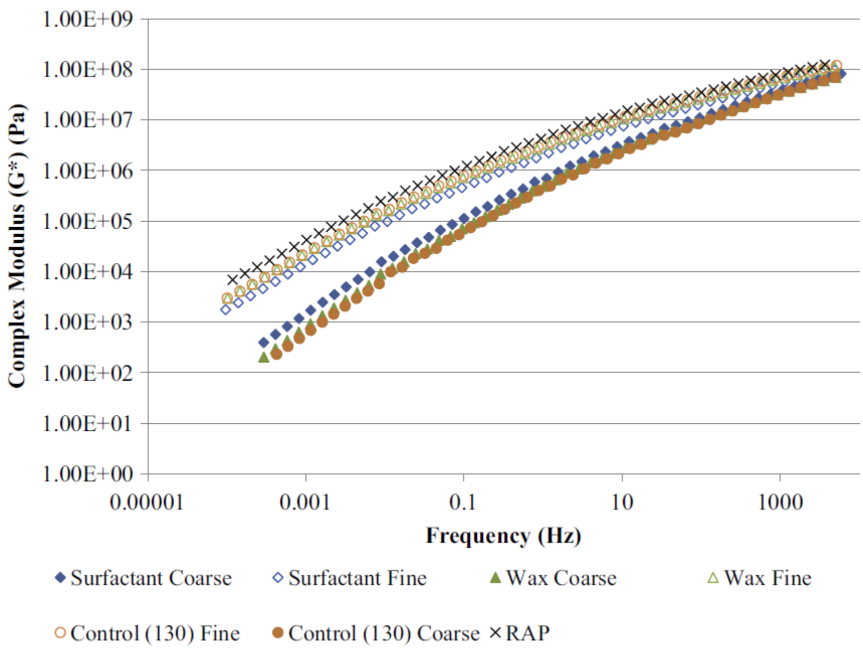


Figure 14: G* master curves with WMA additives (Bowers, Moore, *et al.*, 2014)

The authors found that there is a limit to which mixing time has an influence on binder blending. While mixing temperature may have a significant effect as well as the use of WMA additives. Increasing the mixing time beyond 150 seconds showed low to no influence. Mixing temperature presented a significant increase of the blending from 130°C to 180°C. The surfactant based WMA additive has a positive effect on blending. The researchers discovered that the wax based WMA additive improved workability of the mixtures at 130°C and seemed to reach the best aggregate coating.

Norton *et al.* (2014) carried out a performance-based experimental programme using low-temperature cracking, stiffness and moisture susceptibility tests on asphalt mixtures containing 25% and 35% RA. First, they assumed different levels of the DOB, from 70% up to 100% DOB. They then calculated the DOB based on volumetric properties: 67% DOB in mixture with 25% RA, while the mixture with 35% RA was assumed as 100% DOB as it was not possible to calculate DOB due to variability issues. The conclusion was that 100% of the DOB might have an adverse effect on mixture performance, particularly when more than 25% RA is used. Performance-based parameters have been used in other studies (McDaniel *et al.*, 2000; Al-Qadi *et al.*, 2009) to compare the mechanical performance parameters for bituminous mixtures containing RA and conventional mixtures.

Stimilli *et al.* (2015) propose an analytical method combining performance-based equivalence principle and specific surface area approach, where an assumption is made: the DoBA is proportional to the re-activated bitumen film thickness. Four RA mixtures were prepared: reference mixture with 25% of unfractionated RA and three mixtures with 40% RA (one with coarse RA, one with fine RA and one with combined fractions). Results showed that mixtures with 40% fine RA and 40% of combined fractions had almost the same DoBA, around 70%. Furthermore, it was concluded that the proposed methodology overestimates the real amount of activated binder in the mixture with a high amount of fine RA particles. This was, according to authors, because a certain amount of RA particles possesses a lower surface area than the one calculated from the original RA aggregates obtained after binder extraction. Performance-based equivalence principle was based on the assumption that the effective binder content in virgin asphalt mixture and the RA mixture is the same if the mechanical performance of both mixtures is comparable.

2.3.3.1.2. Mechanistic approach

This methodology aims to predict the mixtures performance (Dynamic modulus - E*) considering the performance of the asphalt binder (G*).

Mogawer *et al.* (2013) and Booshehrian *et al.* (2013) investigated the DOB by comparing the bitumen and mixture performances. Firstly, asphalt mixtures are tested to obtain the E*, while binders are extracted and recovered from these mixtures to measure the G* in the DSR. The Hirsch Model was then applied with the obtained data, together with voids in mineral aggregate and voids filled with bitumen of the asphalt mixtures to estimate the E*. The E* was then compared with the measured E*, where the high correlation of the data indicates high DOB. In the same research from Mogawer *et al.* (2012), the influence of mixing, storage time and RA preheating on the DOB were determined; it was concluded that almost all mixtures presented satisfactory DOB. Similar investigation was carried out years earlier by Al-Qadi *et al.* (2009), according to the authors the results were affected by high variability on mixtures, with the conclusion that the Hirsch model may not be appropriate to back-calculate E* from HMA with RA and the DOB could not be accurately determined by using this method.

Gaitan *et al.* (2013) carried out a procedure comparing WMA mixtures with HMA, using the specific surface area approach. The Bailey's method is used to estimate the surface area of fine RA aggregates to determine the proportion of RA and virgin binder that would coat the fine RA aggregates under zero blending condition. The determined amount of virgin binder is blended with the RA binder and exposed to short-term ageing. Two amounts of RA were used in the study, and it was concluded that the DOB is higher in WMA mixtures than the RA mixture (82-85% comparing to 59%, respectively). Also, it was observed that modified bitumen, as well as mixing time, helped in increasing the DOB, while conditioning time and mixing temperature did not affect it.

Zhang *et al.* (2015) conducted a coating study using different RA-contents (10%, 30% and 50% RA), different virgin aggregate temperatures (160, 180 and 190°C) and RA moisture content (0%, 3% and 5%) to investigate the DoBA. The complete procedure was simulated using the discrete element method (DEM). Simulation results confirmed laboratory results that the DoBA was dependent on RA content, mixing temperature, mixing time and moisture content. Increasing RA and the moisture content resulted in the DoBA decreasing, while it increased as the virgin aggregate temperature increases. Regarding the amount of bitumen from the virgin aggregate, DEM results showed higher values than laboratory results, possibly due to limitations of the method (single-sized RA particles were used). In summary, DEM has shown potential to evaluate the effects of the RA content and virgin aggregate temperature on the DoBA, but qualitatively, not quantitatively.

Delfosse *et al.* (2016) conducted research using an extraction leaching method to recover binders from recycled mixtures, measuring G* values of the obtained binders to apply a mathematical model to calculate E* and compare with measured E* value from produced mixtures. Calculated values of E* were nearly the same as measured E* when a high level of DOB was present. However, in cases with an inferior DOB, authors suggested that improvement of models are necessary.

2.3.3.1.3. Chemical & Visualisation methods

Chemical methods have been used mostly with Gel Permeation Chromatography (GPC) and Fourier Transform Infrared Spectroscopy (FTIR). The GPC is used to separate molecules of a solution into various sizes. Typically, it is used to determine the relative molecular weight of polymer samples and the distribution of molecular weights. Also, it can be used for studying the ageing mechanism of bituminous binders. This procedure is capable of differentiating aged binder from virgin binder because aged binder has a higher portion of large molecules than the virgin binder. The FTIR is a measurement technique that can be used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects high-spectral-resolution data over a more extensive spectral range and it is possible to determine functional groups.

Visualisation methods at various scales have been used to investigate the nature of the DOB, although they have perspectives for determining DOB as well. For this purpose, methods and equipment such as optical and electron microscopy, atomic force microscopy, nanoindentation, computed tomography (nano and micro level), have been used.

Bowers, Huang and Shu (2014) prepared an artificial RA binder by ageing a virgin binder through RTFOT followed by double PAV ageing. Thus, 9.5 mm aggregate was mixed with a virgin binder and artificial RA binder, then the staged extraction and recovery procedure were followed. The binder blends were then tested with FTIR to compare the ratio of the carbonyls (C=O) and the saturated C-C vibration to evaluate oxidation due to an increase in the carbonyl, as an indicator of the oxidation of the bitumen. From the results the ratio is higher as the binder layer is closer to the aggregate, concluding that the DOB was not entirely uniform.

Zhao, Huang, Shu, Moore, *et al.* (2016) also performed a similar study on the binders obtained after extraction and recovery from coarse virgin aggregate and fine RA, which had been mixed with a virgin binder. Within this research the DOB was not determined quantitatively, but it was concluded that binder film coating the virgin aggregate was blended better with virgin bitumen than binder film around RA aggregate. The authors confirmed that binder blend re-coated both virgin and RA aggregates, while un-mobilised binder was still attached to the RA.

Ding *et al.* (2016) tested three plant-produced recycled mixtures (WMA, HMA and HMA + rejuvenator) to investigate the DOB. FTIR procedure was applied to the binders extracted from different sizes of aggregate particles which had been taken from each mixture. The precise amount of the DOB could not be estimated for each case, but authors found it possible to compare them using ageing indexes. Research results showed that WMA had the highest DOB and that rejuvenators slightly improved the DOB of HMA.

A procedure based on a progressive bitumen recovering was performed by Delfosse *et al.* (2016) and called the "leaching blending test", where through FTIR test analysis of the leachates, a Carboxyl index was determined. In the methodology, the DOB was qualified by the authors as the ratio between last and first oxidation level. When the ratio is below 1.3, a good level of the DOB is defined. However, when DOB is above 1.3, it is estimated as a poor DOB. Testing results showed that HMA containing 20% and 35% RA, with PMB and neat bitumen respectively, had a high level of blending. Figure 15 shows two opposite results found in the author's investigation, where their work on DOB is called Blending Ratio (B.R.).

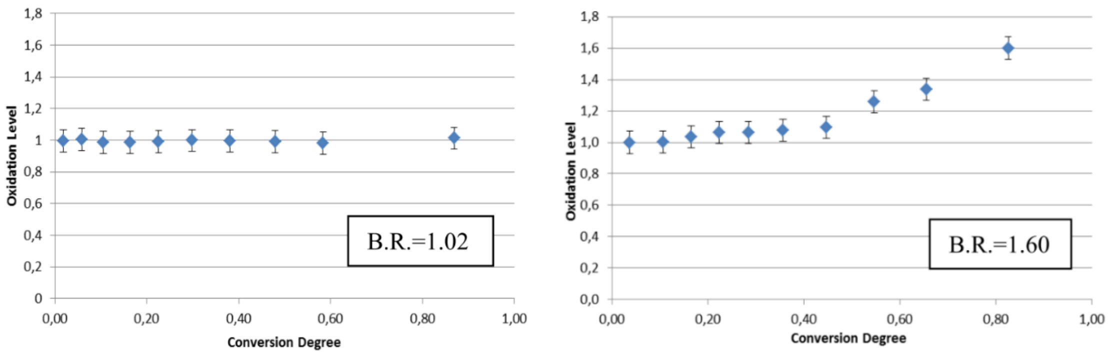


Figure 15: (a) Good and (b) Poor DOB obtained by Delfosse *et al.* (2016)

Scanning Electron Microcopy (SEM) was used by Al-Qadi *et al.* (2009) to determine the bitumen film thickness on aggregate particles and to investigate if the virgin and RA binder could be exclusively identified. The film thickness was found to be expressively smaller than the theoretically calculated values. This method was identified as not appropriate to quantify the DOB, but it may help in observation of the binder blend homogeneity and it may be used as an additional tool to investigate the DOB level.

Navaro *et al.* (2012) attempted to evaluate the DOB through homogeneity of binder blend under different mixing temperatures and times. The image analysis protocol was conducted on images taken under white and ultraviolet lights. The main conclusion from microscopic observation was that the manufacturing temperature is intimately connected with mixing time and that both parameters have a very high influence on the DOB.

Nahar *et al.* (2013) observed the presence of blending zone at the interface of the two binders of different grades by using Atomic Force Microscopy (AFM) images, as a visualisation method, where the blending zone was directly detected. It was identified that at the interface of RA and virgin binder, the DOB was 100%, but just in a transition area. Moreover, the range of the blending zone (see "d", Figure 16), according to the authors, is possibly dependant on parameters such as temperature, binder type and contact time.

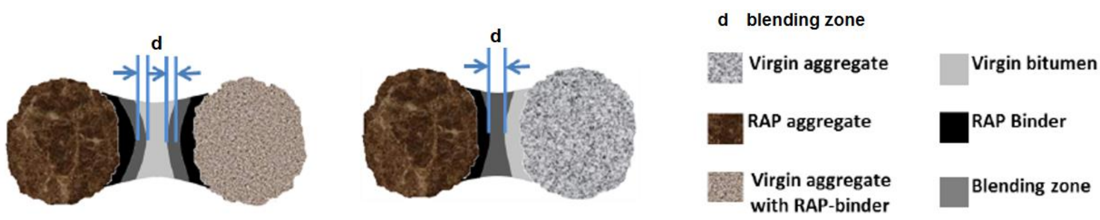


Figure 16: Prospects for the development of a blending zone between the RA and virgin aggregate, adapted from Nahar *et al.* (2013)

Rinaldini *et al.* (2014) concluded that using X-ray Computed Tomography (X-ray CT) allows the virgin and RA materials grouped in homogenous but distinct clusters to be observed. From the X-ray CT results the DOB was found to be location dependent. Mohajeri (2015) also used Nano-tomography scanning images to evaluate the DOB, but found it impossible to distinguish two binders in the mixture with RA, though it was possible to notice the interface between bitumen and aggregate as well as to determine the binder film thickness. Although the DOB cannot be estimated directly by using these methods, they can be applied as additional methods for other techniques.

Energy dispersive X-ray spectroscopy (EDS) and SEM have also been used by Castorena *et al.* (2016) to analyse the DOB in the mixtures with coarse RA particles and pre-processed RA. Titanium dioxide was used as a tracer to recognise whether the blending of RA and virgin binder occurred. As a result, it was concluded that mixtures containing pre-processed RA lowering the formation of RA clusters and as a consequence, this provided the higher level of DOB. Bressi *et al.* (2015) reported similar findings on their investigation through RA clustering formation.

Cavalli *et al.* (2016) used imaging techniques, Environmental Scanning Electron Microscopy (ESEM) and X-ray CT, to investigate asphalt mixtures with high-RA content. It was concluded that the RA binder thickness tends to decrease by increasing the mixing temperature. Moreover, it was observed that increased local curvature of the aggregates might influence the RA binder film thickness and the binder activation. Results regarding mixing temperature and micro geometrical inhomogeneity were confirmed by the same authors in Cavalli *et al.* (2017).

2.3.3.2. DOB and DoBA: Conclusions

As it can be seen, many parameters influence both DoBA and DOB. Since the most of these parameters are connected, some of them are explained separately as follows:

- **Mixing temperature**: if the mixing temperature is high enough, the RA binder becomes fluid ensuring a higher level of both DoBA and DOB;

- **Mixing time**: if mixing time is prolonged, the virgin aggregate has more time to detach the aged binder from RA particles and to make it available for mixing with virgin bitumen. With prolonged mixing time without addition of the virgin binder, the DoBA might be higher, as well as the DOB after the addition of the virgin bitumen;

- **RA content**: if the RA content is high, above 50%, the aged binder surrounding RA particles behave as an absorber, while a lower amount of the virgin aggregate does not has enough energy to detach the aged binder. This may cause lower values of both parameters;

- **Virgin aggregate shape**: when virgin aggregate grains present a lot of sharp edges, it might be easier to release the aged binder. It can therefore be concluded that if round-shaped grains are mixed with the RA, the DoBA/DOB would be affected;

- **RA and virgin binder grade (stiffness)**: increasing the RA binder stiffness (more aged bitumen) results in higher difficulty to increase the DoBA, however, if the virgin binder grade is lower the DOB should be higher;

- **The binder film thickness**: having a thick aged binder film should result in higher DoBA and DOB because more binder is available to be detached, according to some methodologies presented;

- **Binder modification**: there are many different ways of binder modification such as the use of recycling agents, polymers and anti-stripping agents. Some of these products can be mixed with the RA or added to the virgin binder with the purpose to soften or reactivate the aged binder improving the DoBA/DOB;

- **RA fraction size**: due to an increase of the specific surface area with a reduction in the size of RA particles, the amount of aged binder is relatively higher, implying more binder available to increase the DoBA/DOB.

This section aimed to provide a review of information and methodologies to answer the question of how to determine the DoBA and the DOB. In addition to the above parameters, another variables that are possible to have an impact in the DoBA and the DOB were found in the literature:

- RA binder content;
- RA variability, related to stockpiles management and the material processing;
- RA moisture content;
- RA conditioning temperature and time;

- Mixtures discharge temperature (regarding asphalt plant production);
- Virgin aggregates absorption;
- Grading curves and amount of filler;
- Micro geometrical inhomogeneity of RA aggregate;
- Surface texture of the RA and virgin aggregates.

According to the research presented in this section, is evident that the full blending (100%) between a virgin binder and RA binder does not occur, and its properties during mixing procedures change according to various factors such as temperature, mixing time and types of materials. In this sense, it becomes essential to study the degree of partial blending between RA and virgin materials, or even the RA binder released (DoBA), to be able to maximise the incorporation of recycled material in new asphalt mixtures.

Considering the literature review presented, together with the methodology tailored for the research, some of the variables that can possibly affect the DoBA are in some way involved in this thesis: mixing time, mixing temperature, RA conditioning temperature and time, RA binder grade, RA binder modification, RA binder content, virgin aggregates absorption and grading curves.

2.3.4. Additives and rejuvenators to increase the amount of RA

Reclaimed asphalt binder properties depend on the composition of the original binder and ageing during service. If the aggregates are of good quality, then the main obstacle for increasing the RA dosage is the aged RA binder. According to many researchers (Shen and Ohne, 2002; Karlsson and Isacsson, 2006; Al-Qadi *et al.*, 2007; Shirodkar *et al.*, 2011; Zaumanis, Mallick and Frank, 2014b, 2014a) the stiff binder has low workability and may cause fatigue and thermal cracking. The increase in RA proportion in pavements increases the potential of such cracking which is one of the main reasons for government agencies to set a limit on the maximum allowed RA content. Other reasons are the unknown degree of blending that occurs between virgin and RA binders and the active contribution of the RA binder towards the total binder content of the mixture (Item 2.3.3).

For this purpose, when RA content is higher than a certain percentage (limitations depend on RA properties and local specifications) or when the RA contains a particularly hard aged binder, it seems to be necessary to introduce another component in the mixture (Shen and Ohne, 2002). This component will be responsible for restoring some of the properties that the RA had before its first service life (Karlsson and Isacsson, 2006; Tran *et al.*, 2012; Zaumanis *et al.*, 2013).

Successful use of rejuvenators should reverse the RA binder ageing process, restore the properties of asphalt binder for another service period and make the RA binder efficiently "available" to the mix. Hence allow a significant increase in the amount of RA that could be used in HMA (Zaumanis, Mallick and Frank, 2014b). These products have the potential to do so by restoring the rheological and chemical components of aged RA binder. Rejuvenators are sometimes also referred to as softening additives or recycling agents but due to a lack of industry consensus, these products are usually simply called ''rejuvenators'' (Karlsson and Isacsson, 2006; Zaumanis, Mallick, Poulikakos, *et al.*, 2014).

Zaumanis, Mallick, Poulikakos, *et al.* (2014) established the expected short and long-term performance of rejuvenated binder as follows:

Short-term: Rejuvenators should allow the production of high RA content mixtures by rapidly diffusing into the RA binder and mobilising the aged bitumen to produce uniformly coated mixtures. Should soften the binder to produce a workable mixture that can be smoothly paved and compacted to the required density without the hazard of producing harmful emissions. A major part of diffusion process should be completed before the traffic is allowed to avoid reduction of friction and increased susceptibility to rutting.

Long-term: Rejuvenators should reconstitute chemical and physical properties of the aged binder and maintain stability for another service period. The binder rheology has to be altered to reduce fatigue and low-temperature cracking potential without over softening the binder to cause rutting problems. Sufficient adhesion and cohesion have to be provided in the mixture to prevent moisture damage and ravelling.

During the years, various materials with the purpose of altering properties of old binders at asphalt recycling have been proposed. A distinction is made between softening agents and rejuvenating agents, where softening agents plainly are aimed at lowering the viscosity of aged bitumen, whereas rejuvenating agents also are added to restore physical and chemical properties of the old binder (Karlsson and Isacsson, 2006).

Concerns about recycling agents are mostly associated with their ability to diffuse into the old bitumen. The diffusion occurs most rapidly at elevated temperatures during mixing, storage and compaction, and it can continue during the service life. According to Zaumanis, Mallick and Frank (2014b), incomplete diffusion can cause pavement distresses:

- If the diffusion has not been finalised before the traffic is released, the external layer of binder film will have an increased dosage of recycling agent, and this soft coating might cause premature plastic deformations in the pavement life;
- If the recycling agent does not appropriately diffuse into the RA binder, part of the film will remain as "black rock". This can present effectively lower active binder content, increasing the risk of cracking.

Carpenter and Wolosick (1980) assumed that the diffusion of rejuvenators into the old binder could be described in steps as follows: The rejuvenator forms a low viscosity layer surrounding the bitumen-coated aggregate; Starts to penetrate into the old binder layer, decreasing the amount of raw rejuvenator surrounding the aggregate and softening the aged binder; When no raw rejuvenator remains, the penetration of the rejuvenator continues decreasing the viscosity of the inner layer and gradually increasing the viscosity of the outer layer; and, after a certain time, equilibrium is approached over the majority of the recycled binder film. Karlsson and Isacsson (2006) conducted a literature review and concluded that this diffusion phenomenon does not detain from the creation of homogeneous recycled binder films, even though diffusion may be time-consuming at low temperatures.

Studies using rejuvenators in high-RA content recycling has been carried out over the years. Shen and Ohne (2002) performed a study to determine the rejuvenator content based on performance-related properties of binders using the SHRP critical temperatures, an oil type of rejuvenator available commercially in the USA was used. They found linear correlations between critical temperatures (complex modulus G* and sin δ parameters) and rejuvenator dosage, concluding that linear blending charts could be used to obtain the rejuvenator content. The addition of the rejuvenator decreased the high critical temperature and improved both intermediate and low temperatures, meaning that the maximum rejuvenator content has to be controlled.

Shen *et al.* (2007) continued the previous work mentioned above by using the optimum rejuvenator dosage that was found to manufacture mixtures with RA content up to 48%. The obtained results showed that rejuvenators improve mixture performance in comparison to virgin mixtures (with soft bitumen).

Tran *et al.* (2012) conducted an investigation with 50% RA mixtures and recycling oils, concluding that rejuvenating agents improved cracking resistance of mixture without adversely affecting rutting resistance and moisture damage.

Zaumanis, Mallick and Frank (2014a) studied the dosage using penetration and binder film thickness as the design parameters, correlating the results according to Performance Grade critical temperatures. Zaumanis, Mallick, Poulikakos, *et al.*

(2014) studied the performance of six rejuvenators (waste vegetable oil, waste vegetable grease, organic oil, distilled tall oil, aromatic extract and waste engine oil). Their results showed that none of the rejuvenators used decreased the high critical temperature of the recovered binders and there is no danger of increased rutting susceptibility, and all the binders met the Superpave fatigue resistance requirements. All recycling agents were able to improve low critical temperatures by around 10^0C and reduced binder viscosity allowing the mixture manufacture temperature to decrease by 15-25^0C. In a sequence of these studies, these six rejuvenators were analysed in 100% RA mixtures. The results showed that all RA mixtures exhibited good rutting resistance and improved thermal cracking resistance in comparison to non-rejuvenated RA mixtures, with the results also being compared to a virgin mixture. They concluded that the application of organic products as recycling agents require much lower dosage than petroleum ones and can obtain the same rejuvenating effect.

Ongel and Hugener (2015) also studied the use of rejuvenators in 100% RA binders, comparing the ageing effect on these recycled binders with a virgin bitumen through rheological tests as well as penetration and softening point. The rejuvenators were two resins from cashew nut shells and an oil additive. They concluded that this practice is possible and that in fact, the ageing of RA binders with rejuvenators and virgin bitumen presented no significant difference. However, it was shown that the ageing of the virgin binder is slower than that of the rejuvenated bitumen.

Ali *et al.* (2016) investigated the ability of five selected rejuvenators (naphthenic oil, a paraffinic oil, an aromatic extracts, a tall oil and an oleic acid) to restore low and high temperature through performance grades of aged binders. The study applied tests on asphalt mixtures containing 25 and 45% of RA on mixtures prepared with a polymer modified binder (PMB) as well as a control mixture for reference. The lab produced mixtures were aged for 2 and 6 hours for further binder recovery to test the binders in the DSR and BBR. The rejuvenators were found to improve the fatigue resistance without significantly influencing rutting performance, as well as the rejuvenation efficiency not being affected by ageing or the amount of RA.

Borghi *et al.* (2017) used the procedure based on rejuvenators' dosage using the European methods of penetration and softening point. The additive used as a rejuvenator was derived from pine trees with dosage investigated on two sources of RA. The author's results showed that physical and rheological properties could be completely restored by the addition of the proper dose of rejuvenator, while the chemical composition required further studies. Rejuvenated binders exhibit good long-term performance especially at high and low temperatures, but also showed significant resistance against fatigue at intermediate temperatures. Similar work was carried out by Arámbula-Mercado *et al.* (2018), using a method to estimate the optimum recycling dosage of the rejuvenators through the restoration of the performance grade (PG) of the blended binders. The authors recommend the high-temperature PG method to determine the optimum dosage of the recycling agents.

Jiménez del Barco Carrión *et al.* (2017) studied two bio-materials and their rejuvenating effect on RA binders, compared to neat and PMB virgin binders. The author's case simulated 50% RA in the mixture (only for design purposes), with the rejuvenating effect been assessed through Apparent Molecular Weight Distribution and other rheological parameters such as complex modulus, R-value and crossover frequencies. Results revealed the ability of bio-materials to restore RA bitumen, showing their capability to be also used as fresh binders in high-RA content mixtures. Cavalli *et al.* (2018) have also investigated bio-based rejuvenators and their restoring effect on RA binders through rheological, mechanical and chemical tests. The authors propose an ageing index to show that the rejuvenators are affected differently by ageing. The work demonstrated that ageing could affect the chemical and rheological properties, where DSR results show that the additives enhance the mechanical properties of RA binders mostly in their unaged state. Furthermore, according to the authors, the mechanical changes were not caused by chemical changes but due to a rearrangement at higher molecular scale such as polar and nonpolar components. In summary, the authors conclude that the effect of ageing is essential to identify how the rejuvenators can affect the chemical and mechanical properties of the RA binders.

Nabizadeh *et al.* (2017) investigated the effects of three rejuvenators (petroleum, green and agriculture tech bases) on the mechanical characteristics of asphalt and fine aggregate matrix mixtures containing 65% RA content. The experimental programme considered tests to evaluate stiffness, cracking, permanent deformation and damage-induced performance of those mixtures. In summary, the rejuvenators increased the ductility of the RA mixes and improved the cracking resistance; in comparison with second type of mix, authors found a positive correlation in this investigation, implying that the fine aggregate matrix could provide information for predicting asphalt mixtures.

2.4. Characterisation of asphalt binders and mixtures for engineering purposes

2.4.1. Binders rheology

Rheology, the study of the flow of matter, is a fundamental science that is concerned with the study of the internal response of real materials to stresses. It also includes the study of materials that exhibit both solid and liquid characteristics. Bitumen rheology can be defined as the fundamental measurements associated with the flow and deformation characteristics of bitumen.

A proper specification should address the predominant failure modes of asphalt pavements. In this sense, the bitumen behaviour can be discussed in three regions of differing behaviour: Low-temperature linear elastic; high temperature viscous, and; intermediate temperature viscoelastic region (Southern, 2015; Taylor and Airey, 2015).

2.4.1.1. Viscoelastic behaviour of bitumen

Bitumens are viscoelastic materials, so their behaviour is partially elastic and partially viscous. The strain response under load application depends on the loading time and the temperature, and when it ceases, bitumen will recover only some of that deformation. At low temperatures, the elastic properties dominate, while at high temperatures the bitumen behaves like a liquid. At normal pavement temperatures, the bitumen has properties that are in the viscoelastic region, where both elastic and viscous behaviour is presented (Airey, 1997). This behaviour can be seen in the example in Figure 17, constant static stress is applied during a certain period (Load on) and then removed (Load off). The elastic component of bitumen behaviour is responsible for the instantaneous deformation and immediate recovery. On the other hand, the viscous component is responsible for the creep, delayed recovery and permanent deformation.

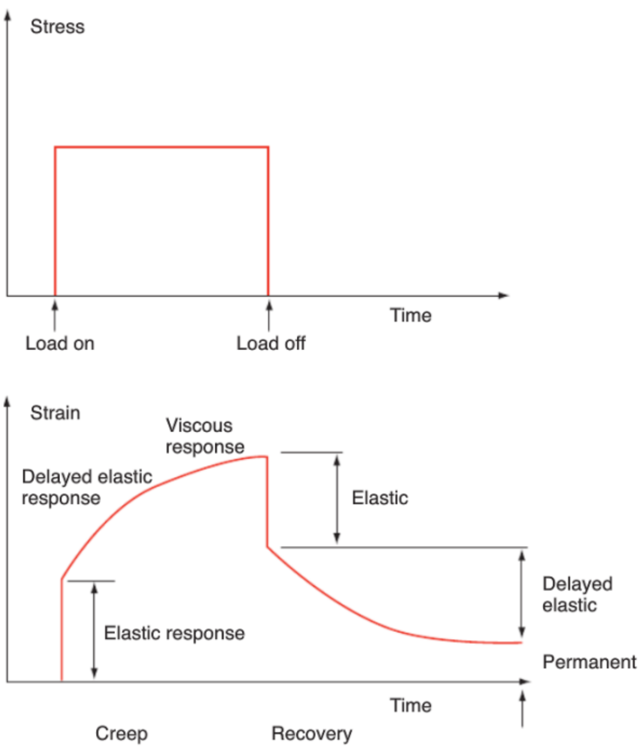


Figure 17: Stress-strain response in a bitumen test (Taylor and Airey, 2015)

Bitumen response presented above is associated with linear viscoelastic (LVE) behaviour. LVE behaviour implies that for any applied load, stress and strain are proportional. Non-linear response is not easy to study and characterise in both modelling and experimental studies. For this reason, for engineering purposes, bitumen is usually studied within the limits of LVE region, which implies that its behaviour only depends on loading time and temperature (Airey, 1997).

2.4.1.2. Dynamic Mechanical Analysis (DMA) and Dynamic Shear Rheometer

Viscoelastic materials have high mechanical damping, and mechanical vibrations do not build up easily at natural frequencies and high temperatures. The rheological properties of unmodified bitumen vary with the applied load rate and temperature, at temperatures below 60°C, and vary with temperature above 60°C (Airey, 1997). Therefore, the materials need to be characterised over a wide range of temperatures and loading times to predict their performance, and it is standard practice to use oscillatory type testing for doing DMA to investigate the rheology of a viscoelastic material. Regarding DMA, a sinusoidal strain or stress controlled load, within the linear viscoelastic range, is applied to a sample of bitumen, in the dynamic shear rheometer (DSR), sandwiched between two parallel discs with a loading frequency, ω (rad/sec).

The ratio of the resulting stress to the applied strain at any time is called the complex shear modulus, G*, defined by Equation 14:

$$G_t = \frac{\sigma_0}{\gamma_0} = \left(\frac{\sigma_0}{\gamma_0}\right)\cos\delta + i\left(\frac{\sigma_0}{\gamma_0}\right)\sin\delta = G' + iG'' \qquad \text{(Equation 14)}$$

The term $\sigma o/\gamma o$ (the ratio of the peak stress to the peak strain) is called the norm of the complex modulus, |G*|, Pa. The G′ is the storage modulus, and G″ is the loss modulus.

The magnitude of the |G*| can be calculated as the square root of the sum of the squares of the storage modulus and loss modulus as expressed below in the Equation 15:

$$|G^*| = \sqrt{(G')^2 + (G'')^2} \quad \text{(Equation 15)}$$

The ratio of the viscous component of the complex modulus to the elastic component of the complex modulus is known as the tangent of the phase angle (δ) and also as the loss tangent (Equation 16):

$$\tan\delta = \frac{G''}{G'} \text{ ; thus } \quad \delta = \tan^{-1}\frac{G''}{G'} \quad \text{(Equation 16)}$$

According to the Asphalt Institute (1994), the dynamic viscoelastic response of the materials described above must be within the linear range during the DSR testing so that the stiffness of materials is not influenced by the magnitude of the applied strain or load, but is only influenced by temperature and loading time.

According to the Asphalt Institute (1994), since the bitumen behaviour depends on both loading time and temperature, the ideal test should include both factors. The testing equipment with this capability is generically known as dynamic rheometers, dynamic shear rheometers or oscillatory shear rheometers. In such tests, described in the European Standard EN 14770 (2012), a sample of bitumen, which is sandwiched between two parallel discs or plates (one that is fixed and one that oscillates – Figure 18), is subjected to alternating shear stresses and strains. As the plate oscillates, the centreline of the plate at point A moves to point B. From point B, the plate centreline moves back and passes point A to point C. From point C the plate moves back to point A, completing the cycle. The speed of oscillation is the frequency.

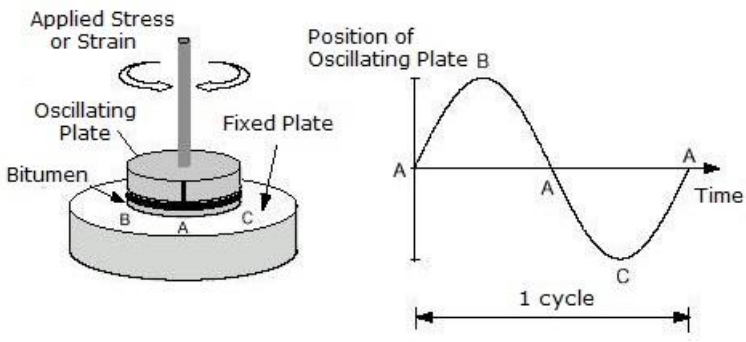


Figure 18: DSR operation, adapted from Asphalt Institute (1994)

Figure 19a shows the behaviour of two bitumens, represented by vector arrows. When these bitumens are loaded, part of their deformation is elastic and part is viscous. In this example, both bitumens have the same G*, but Bitumen 2 is more elastic than Bitumen 1, because of its smaller phase angle δ. Because Bitumen 2 has a more significant elastic component, it will recover more deformation from an applied load. This example shows that G* alone cannot describe bitumen behaviour, δ is also needed (Asphalt Institute, 1994).

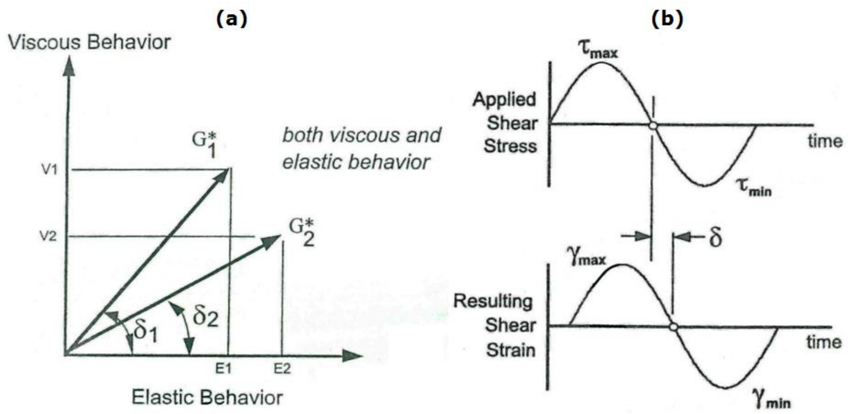


Figure 19: Viscoelastic behaviour – (a) G* and δ; (b) Stress/Strain response, adapted from (Asphalt Institute, 1994)

In summary, the G* is the ratio of total shear stress (τ_{max} - τ_{min}) to the total shear strain (γ_{max} - γ_{min}). The time lag between the applied stress and the resulting strain, or vice-versa, is related to the δ, as shown by Figure 19b. The equations used by rheometer's software to calculate the τ and γ are presented below (Equations 17 and 18):

$$\tau = \frac{2T}{\pi r^3} \quad \text{(Equation 17)}$$

$$\gamma = \frac{\theta r}{h} \quad \text{(Equation 18)}$$

Where τ is the stress in a distance r from the centre of the sample, γ is the strain in the r distance, T is the torque (N.m), θ is the rotation angle (rad), r is the radius (m) and h is the height (m) of the sample.

The Superpave specifications use only G* and δ results, although the DSR is capable of providing more information for analysis. With the results, two analysis can be made: Permanent deformation which is governed by limiting G*/sinδ at the test temperature to values greater than 1.0 kPa for original bitumens and 2.2 kPa after short-term ageing, and; Fatigue cracking which is managed by limiting G*.sinδ after long-term ageing to values less than 5.0 MPa at the test temperature. The tests should be performed at a frequency of 1.59 Hz for these specifications (Asphalt Institute, 1994).

2.4.1.3. Bending Beam Rheometer (BBR)

Evaluating the low-temperature characteristics of asphalt is crucial to perceive the susceptibility of such pavement for sustaining the low temperature cracking and thermal fatigue cracking. Bitumens become stiffer as the temperature is reduced. While this would lead to higher mixture stiffness, which would ordinarily be a decisive factor in respect to load-bearing capacity, the ability of the binder to dissipate stress decreases as the temperature reaches the glass transition temperature (Southern, 2015). The BBR is the most widely used test for determining the stiffness of bitumen at low temperatures (down to -36°C) and is described in the European Standard EN 14771 (2012). The outputs of the test are the flexural creep stiffness (S, in MPa) and m-value of the binder.

During the test, a beam is subjected to constant stress using a loaded piston (100g) in a three-point bending machine. By knowing the applied load on the beam and measuring the deflection (vertical displacement) during the test, the stiffness can be determined. Bitumens which have low stiffness S will not crack in cold climates. Likewise, bitumens having higher m-values are more efficient to dissipate the stresses formed during its contraction when the temperature drops abruptly, by minimising the formation of cracks. Therefore, the S parameters relate to the formation of thermal cracks, due to low temperatures. The S is calculated by the following Equation 19. The m-value is the absolute slope of the curve of the logarithm of the stiffness versus the logarithm of time.

$$S = \frac{pL^3}{4\delta(t)bh^3} \qquad \text{(Equation 19)}$$

Where *L* and *h* are the beam length and heigh, *p* is the applied constant load, *b* is the width and δ(*t*) is the diplacement at time *t*.

According to the Superpave specification, the S of the bitumen must be less than 300 MPa, and the m-value should be higher than 0.300, both at a load time of 60 seconds (AASHTO MP-1, 1998). The lower the S, higher the resistance to thermal-cracking, therefore is limited to a maximum value for S. As m-value decreases, the tendency is to relieve thermal stresses in the asphalt mixture, therefore is limited to a minimum value required to m.

2.4.2. Binders performance-related properties

2.4.2.1. Rutting

Rutting in asphalt mixtures appears due to plastic deformations taking place in the material because of the repetitive loading of traffic. Therefore, its assessment has to be done under dynamic cyclic loading. Accumulation of permanent deformation in a flexible pavement occurs at high in-service temperatures and/or under slow-moving loads. According to Anderson *et al.* (1991), it is well known that the viscous component of the bitumen leads the rheological response at high temperatures and extended loading time and it is, therefore, exclusively responsible for the non-recoverable deformation.

There are different rutting tests to assess the rutting resistance of asphalt mixtures and binders. The Superpave $|G^*|/\sin\delta$ rutting parameter is widely used and obtained at 1.59Hz which also defines the high-critical temperature for binders (Anderson *et al.*, 1994). Although the Superpave parameter has been used for years to investigate the rutting potential of binders, researchers have found that exist a poor correlation between this parameter and the rutting performance of asphalt mixtures, especially for modified binders (Dongre and D'Angelo, 2003).

In this regard, new tests have been developed to relate the rutting behaviour of binders and asphalt mixtures better. The Multiple Stress Creep Recovery (MSCR) test, which is based on binder creep and recovery characterisation, was firstly developed by the NCHRP 9-10 research program (Bahia *et al.*, 2001) and then improved by Dongre and D'Angelo (2003). This test assesses the bitumen capability to recover deformation, and therefore, their potential resistance to rutting in the asphalt mixture. The validity of the test to characterise binders at high-temperatures has been ascertained by many researchers, and it does better than Superpave parameter in rating binders to their mixtures (D Angelo *et al.*, 2006; D'Angelo, 2009; Tabatabaee and Tabatabaee, 2010; Wasage *et al.*, 2011; Gibson *et al.*, 2012; Zoorob *et al.*, 2012).

The MSCR test consists of applying repeated creep recovery of shear stress for a period of 1s and then removing the stress for 9s to allow the material to recover, this is repeated for 10 cycles using different stress levels, a typical one cycle of creep-recovery is shown in Figure 20, and the typical MSCR curve after 10 consecutive cycles in Figure 21. The test is usually performed on RTFO aged samples to simulate the ageing during mixing and lay-down. The test is conducted on samples between two parallel plates of 25mm diameter using the DSR equipment and described in detail in the EN 16659 (2015) or AASHTO M 332 (2014) standards. In the new test protocol, two levels of shear stress are used 0.1kPa and 3.2kPa, 10 repeated cycles of 10s at each stress level with no time lag between cycles.

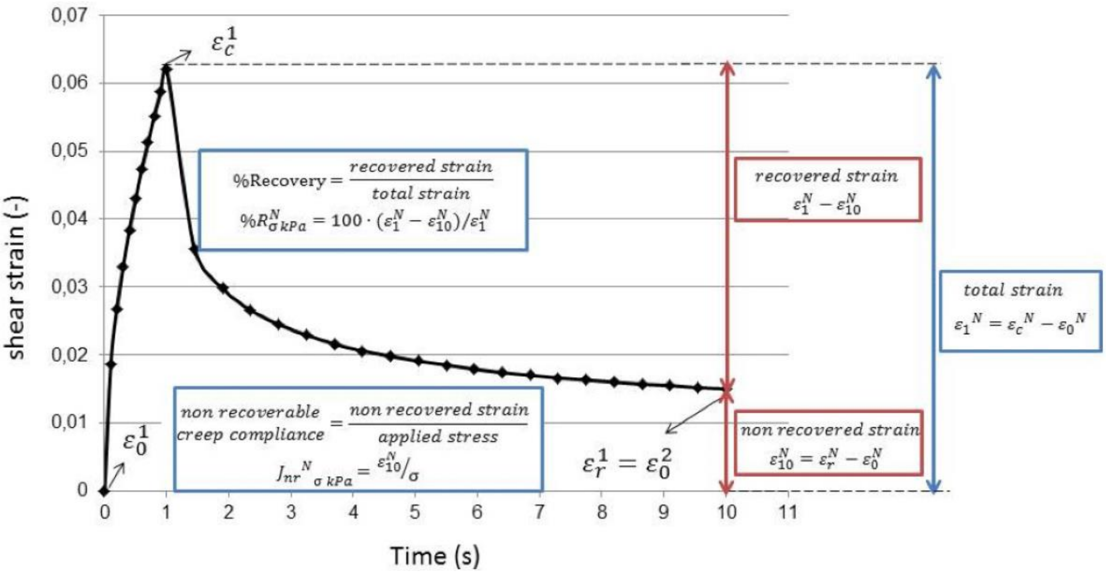


Figure 20: Typical creep-recovery cycle (EN 16659, 2015)

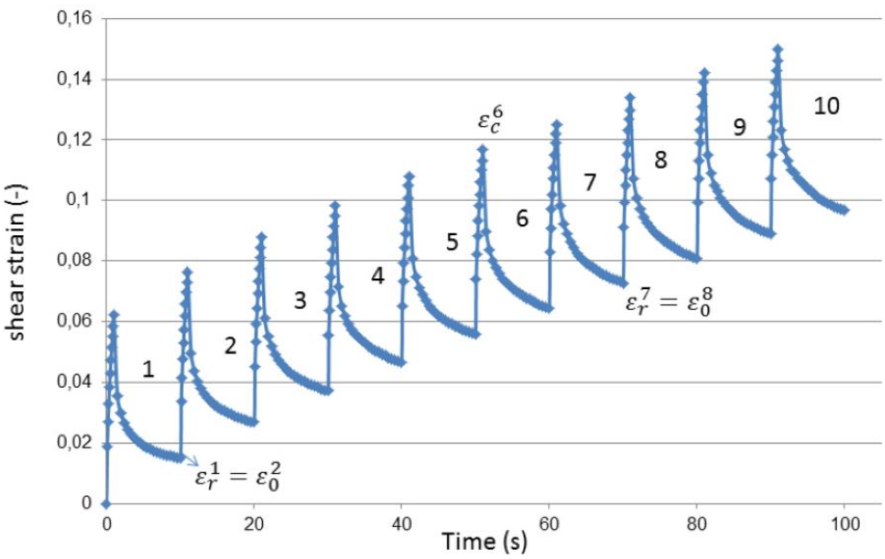


Figure 21: Typical MSCR curve after 10 consecutive cycles (EN 16659, 2015)

Two essential parameters are obtained from MSCR: the percent recovery (%) and the non-recoverable Creep Compliance (Jnr), and they are defined in Equations 20 and Equation 21, respectively:

$$\%R_{\tau}=\frac{1}{10}\sum_{N=1}^{10}\left(\%R_{\tau}^{N}\right)=\frac{1}{10}\sum_{N=1}^{10}\frac{100*\left(\varepsilon_{1}^{N}-\varepsilon_{10}^{N}\right)}{\varepsilon_{1}^{N}} \qquad \text{(Equation 20)}$$

Where,

$\%R_\tau$ = average recovery of the 10 cycles tested at 0.1kPa or 3.2kPa;
$\%R_\tau^N$ = percent recovery for each cycle N tested at 0.1kPa or 3.2kPa;
ε_1^N= is the strain after 1 second (creep) at cycle N;
ε_{10}^N = is the strain after 10 seconds (recovery) at cycle N.

$$J_{nr,\tau}=\frac{1}{10}\sum_{N=1}^{10}\left(J_{nr,\tau}^{N}\right)=\frac{1}{10}\sum_{N=1}^{10}\left(\frac{\varepsilon_{10}^{N}}{\tau}\right) \qquad \text{(Equation 21)}$$

Where,

$J_{nr,\tau}$ = average non-recoverable compliance at the stress level 0.1kPa or 3.2kPa;
$J_{nr,\tau}^N$ = non-recoverable compliance at cycle N;
τ = test stress level, 0.1 kPa or 3.2 kPa;
ε_{10}^N = is the strain after 10 seconds (recovery) at cycle N.

2.4.2.2. Fatigue

The bitumen behaves distinctively under different temperatures and loading periods. It is purely viscous at high temperatures and under slow-moving loads; it is also elastic and brittle at low temperatures and high rapid loads. However, within intermediate in-service pavement temperatures (10 to 35 °C), where the pavement is subjected to a considerable portion of its repetitive traffic loads, within those temperatures, the primary mode of distress is the fatigue cracking. The asphalt pavement is moderately stiffer and more elastic at these temperatures, and the excessive repetitive stress loads are dissipated through crack initiation and ultimately propagation (Subhy, 2016). The fatigue resistance of asphalt mixtures is significantly related to the properties of their bituminous binders. Thus, characterising the fatigue resistance of binders and improving this property using modification have been a topic of intensive studies for many years.

The SHRP fatigue parameter ($G^*\cdot\sin\delta$) (Asphalt Institute, 1994) is widely used to characterise and control the fatigue property of binders within intermediate temperatures. However, many studies have suggested that the current SHRP fatigue parameter does not reflect the binder contribution related to the pavement performance (Chen and Tsai, 1999; Bahia *et al.*, 2001; Planche *et al.*, 2004; Tsai and Monismith, 2005; Zhou *et al.*, 2012).

Consequently, different approaches have been investigated to develop a more fundamental and related performance-based characterisation, such as the Linear Amplitude Sweep (LAS) test. The LAS test has been recently proposed to replace the Superpave parameter $G^*\cdot\sin\delta$. (Johnson and Bahia, 2010; Hintz *et al.*, 2011). The test is performance related and can be conducted in the very short period; however, the fatigue life equation is derived from complex mathematical formulations and statistical fitting (Johnson and Bahia, 2010; Hintz *et al.*, 2011; Zaumanis *et al.*, 2013; Jia *et al.*, 2014).

The test data is analysed following the standard procedure described in AASHTO TP101 (2014) for the viscoelastic continuum damage (VECD) analysis, and three parameters are taken into account to evaluate the fatigue resistance:

- **Damage at failure (D_f)**: the level of damage reached before failure. Materials with longer fatigue life can accumulate more damage before failure;

- **Parameter alfa (α)**: an indicator that reflects the slope of the fatigue life and the applied strain in a logarithmic scale. Higher values represent binders with less fatigue resistance;

- **Fatigue - N_f (traffic volume indicator):** the number of the equivalent single axes loads (ESALs) that can be supported by the pavement before the failure.

For the VECD analysis, firstly the α should be determined. The G* and the δ are converted to storage modulus G' using the Equation 22:

$$\log G'(\omega)=m(\log \omega)+b \quad \text{(Equation 22)}$$

Where, the modulus $|G^*|(\omega)$ and the phase angle $\delta(\omega)$ are converted into the storage modulus $G'(\omega)$ for each frequency. The value obtained for m is recorded, and the value of α is obtained by performing the following Equation 23:

$$\alpha = 1/m \quad \text{(Equation 23)}$$

Then the damage accumulation is calculates using the date from the amplitude sweep test. The Equation 24 is applied:

$$D(t)=\sum_{i=1}^{N}[\pi\gamma_0^2(C_{i-1}-C_i)]^{\frac{\alpha}{1+\alpha}}(t_i-t_{i-1})^{\frac{\alpha}{1+\alpha}} \quad \text{(Equation 24)}$$

Where,

$C(t)=|G^*|_{(t)}/|G^*|_{(initial)}$: G* at time t, divided by the initial "undamaged" value of G*;
γ_0 = applied strain for a given data point, expressed in %;
G* = is the complex shear modulus, expressed in MPa;
α = value calculated in Equation X;
t = the time, expressed in seconds.

The value of D(t) at failure, D_f, is defined as the level of damage accumulated before failure and it is an indicator proposed by the standard, the present parameter is calculated according to the Equation 25:

$$D_f = \left(\frac{C_0-C_{PeakStress}}{C_1}\right)^{\frac{1}{C_2}} \quad \text{(Equation 25)}$$

Where C_0 is equal to 1; C_1 and C_2 are curve-fit coefficients.

Finally, the binder fatigue performance parameter (N_f) can be calculated (Equation 26), this parameter provides the number of ESALs that the pavement can withstand depending on the applied strain:

$$N_f = A(\gamma_{max})^{-B} \qquad \text{(Equation 26)}$$

Where, γ_{max} is the maximum strain expected for a pavement, A and B are coefficients determined for each binder.

2.4.2.3. Cracking parameters

It is well known that the binder rheology has an impact on asphalt cracking resistance. The SHRP developed a measure of bitumen rheology ($G^*\sin\delta$) as a criterion for fatigue performance of asphalt mixtures at an intermediate temperature. The maximum value of 5000 kPa is considered for asphalt binders subjected to long-term laboratory ageing. However, it is accepted that $G^*.\sin(\delta)$ is not able to efficiently represent the fatigue cracking behaviour of those binders (Rowe *et al.*, 2014).

Sui *et al.* (2010) conducted frequency and temperature sweep tests in the DSR, using 4mm plates, and developed the G* and phase angle master curves to describe the stiffness and relaxation capability of the bitumens, respectively. Researchers have suggested that the use of Black Space can also provide adequate information about the ability of materials to resist cracking and also to capture the ageing impacts on bituminous materials (King *et al.*, 2012; Mensching *et al.*, 2015, 2017). The change in G* and phase angle in Black Space was, therefore, suggested to serve as an adequate indicator to evaluate the resistance of bituminous materials to age-related cracking (Glover *et al.*, 2005; Anderson *et al.*, 2011; Makowska *et al.*, 2017).

From this type of investigation in the Black Space, researchers developed two useful parameters from the results of DSR testing that are the Rheological Index, also known as R-value, and the Glover–Rowe (G–R) parameter (Glover *et al.*, 2005; Rowe *et al.*, 2014).

2.4.2.3.1. Rheological Index and Crossover frequency

The ageing process of the binder is due to oxidative reactions which bring remarkable changes in both chemical and physical properties. While the changes in chemical properties can be analysed with the FTIR (Fourier-transform infrared spectroscopy) or with the SARA (Saturate, Aromatic, Resin and Asphaltenes) content, the changes in physical properties can be investigated observing at the rheology. The alterations in rheology can be evaluated by using the Rheological Index and the crossover frequency (Christensen and Anderson, 1992; Rowe *et al.*, 2014, 2016).

The Rheological Index (R-value) is a useful indicator of the rheology type, observed through G* master curves. It is defined as the difference between the glassy modulus and the log G* at the crossover frequency (ω) – the frequency where the phase angle is 45° (G'=G") (Anderson *et al.*, 1994; Booshehrian *et al.*, 2013; Jacques *et al.*, 2016). The Equation 27 is used to calculate the R-value, with the glassy modulus (G_g) assumed equal to 1 GPa.

$$R = \frac{\log(2)\times\left(\frac{G^*(\omega)}{G_g}\right)}{\log\left(1-\frac{\delta(\omega)}{90}\right)} \qquad \text{(Equation 27)}$$

The R-value is related to the shape of the master curve; it decreases if the master curve becomes flattered and this can be related to ageing or recycled binders. Whereas, the crossover frequency decreases with the increase of ageing or flattered master curves (Booshehrian *et al.*, 2013; Jacques *et al.*, 2016). Based on this concept, Rowe *et al.* (2016) have suggested that crossover frequency versus R-value can show the relative ageing of bitumens (Figure 22). This approach is used to simplify and help to quickly assess the effect of ageing and different additives such as rejuvenators in the rheology of binders (Rowe *et al.*, 2016; Rahbar-Rastegar *et al.*, 2017).

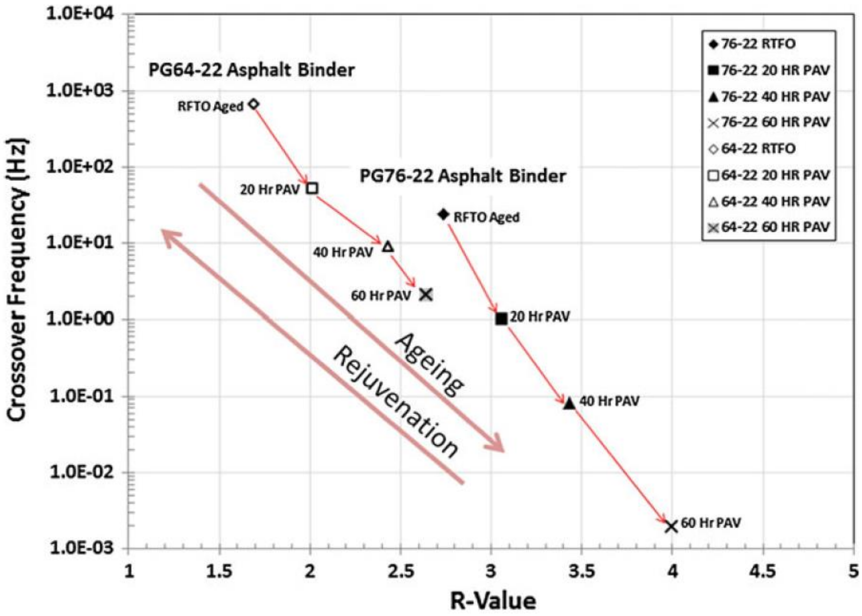


Figure 22: Changes in the R-value and Crossover frequency during ageing (Rowe *et al.*, 2016)

2.4.2.3.2. Glover-Rowe

The G–R parameter can be used to assess the cracking resistance of an asphalt binder. The beginning of this approach was initially put forth by Glover *et al.* (2005). They suggested a correlation between a new DSR function with ductility using a temperature–frequency combination of 15°C and 1 rad/s. Subsequent work established a unique relationship between the ductility test (1 cm/min, 15°C) and the viscoelastic properties of binders at a frequency of 0.005 rad/s and 15°C (Mensching et al., 2015; Anderson et al., 2011). Based on this relationship, Anderson *et al.* (2011) and King *et al.* (2012) generated a criterion for damage curves in the Black Space using the following Equation 28:

$$G - R = \frac{G^* \,(\cos\delta)^2}{\sin\delta} \qquad \text{(Equation 28)}$$

The above equation is known as the G-R parameter and it is computed from the G* and δ of the binder at a frequency of 0.005 rad/s and 15°C. The characterisation of cracking potential by the G-R parameter is represented in Figure 23.

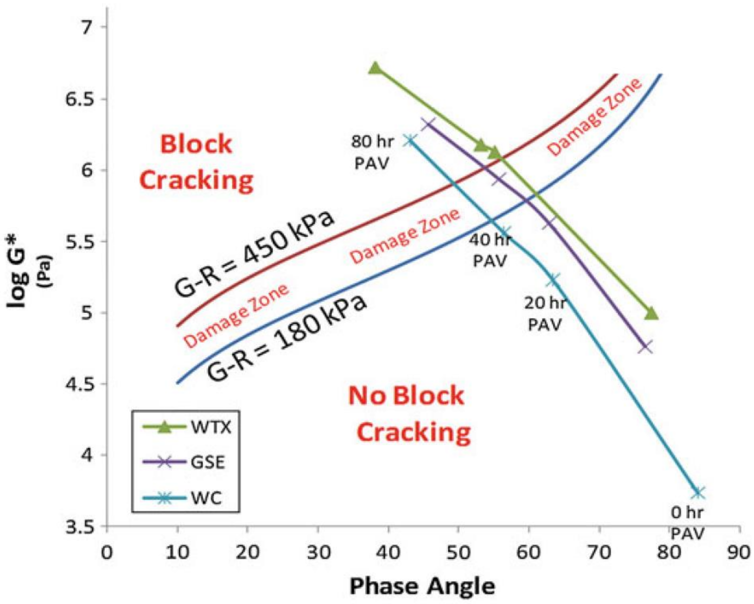


Figure 23: Characterisation of cracking potential by the G-R parameter (Rowe *et al.*, 2016)

The two curves (the blue and red curves) shown in Figure 23 represent the G-R parameter at values of 180 kPa and 450 kPa, respectively. These values are deemed to control the onset of cracking; a value of 180 kPa and higher is associated with the onset of cracking and is linked to a ductility of 5 cm, while a value of 450 kPa and higher is associated with significant cracking and is linked to a ductility of 3 cm (Makowska *et al.*, 2017).

2.4.2.4. Thermal Cracking

Thermal cracking in asphalt mixtures is related to the relaxation capability of binders. The cracking occurs at low temperatures and is predominantly due to the tensile stress and the temperature cycles. Temperature cycles tend to cause multiple stresses in the pavement: the friction between the top layers and the base layer demanding to resist these stresses, develop micro-cracks. These micro-cracks then spread towards the surface and show itself mostly as a transversal cracking on the surface course. This phenomenon is usually studied at temperatures lower than 0ºC to subject the materials to extreme conditions to test their relaxation capacity.

In order to characterise thermal cracking resistance of binders at low temperature, the flexural creep stiffness (S, in MPa) and relaxation capacity (m-value) were obtained using the BBR. When analysing the test results, if the creep stiffness is too high, the bitumen will behave in a brittle manner and is more likely to be susceptible to cracking. To prevent this cracking, the cracking potential can be analysed according to Anderson *et al.* (2011) using the differential between both critical temperatures (Equation 29) determined using the Stiffness and m-value from BBR results:

$$\Delta T_{cr} = [T_{cr(Stiffness)} - T_{cr(m-value)}] \qquad \text{(Equation 29)}$$

Where,

$T_{cr(Stiffness)}$ = critical temperature determined by Stiffness from BBR test;
$T_{cr(m\text{-}value)}$ = critical temperature determined by m-value from BBR test.

According to Equation 29, if ΔTcr is higher than 0, the low critical temperature of the bitumen is controlled by the stiffness, while if ΔTcr is lower than 0, the critical temperature is leaded by the m-value. Researchers have found that ΔTcr and G-R parameter are related and consequently, the cracking warning and limit of the G-R parameter could be extrapolated to find the same type of limits to use ΔTcr as an indicator of potential cracking. A crack warning value of −2.5°C was suggested by Anderson *et al.* (2011), and a cracking limit value of −5°C was suggested by Rowe (2011). Moreover, Anderson (2016) mentions that ΔTcr can be related with the ageing of binders as well as with the rejuvenation effect from additives. In this regard, as a bitumen ages, ΔTcr becomes more negative and indicates a reduction on its relaxation capacity.

2.4.3. Mixtures mechanical properties

The pavements are multilayer structures, the wearing course being the layer that is intended to receive the load of the vehicles and more directly the climactic action. Therefore, this layer should be as much waterproof as possible and resistant to moving tire-pavement contact efforts, which are varied depending on the load and speed of vehicles. The technical and quality requirements of an asphalt pavement must be met with an appropriate design of the pavement structure and with the mixture design as well as compatible with the other layers chosen. This dosage involves the appropriate choice of materials provided to withstand the anticipated traffic and climate demands (Bernucci *et al.*, 2008).

According to Read and Whiteoak (2003), in order to meet the rigours imposed on the roads by modern traffic conditions, asphalt pavement layers must:

- be able to resist permanent deformation and fatigue cracking;
- be workable during laying, enabling the material to be satisfactorily compacted with the available equipment;
- be impermeable, to protect the lower layers from water;
- be durable, resisting abrasion by traffic and the effects of air and water;
- contribute to the strength of the structure;
- be easily maintained;
- be cost-effective.

The asphalt mixture design is, therefore, essential to define an optimum material blend that meets the requirements and specifications of pavement construction. The behaviour of asphalt mixtures under the main distresses affecting them are determined by their linear and non-linear viscoelastic properties. Therefore, a full characterisation of asphalt mixtures should consider all these properties and distresses. To characterise asphalt mixtures, laboratory tests are designed to simulate the traffic loading and climate conditions in the pavement to assess its response under traffic and climatic conditions. In this sense, the following sections describe the mechanical tests applied in the mixtures to investigate performance-related properties.

2.4.3.1. Stiffness Modulus

In the UK, much work has been done to develop an economical and practical means of measuring the structural and performance-related properties of mixtures. The stiffness modulus is now commonly recognised as a significant indicator for asphalt materials, and it is considered an input property to determine the required layer thickness in mechanistic pavement design. The stiffness of bituminous materials reflects the capability of a material to spread the traffic loading over an area in a pavement, where for example the stiffer materials can spread the traffic loading over a wider area than softer materials.

It can be used as a measure of the load-spreading ability of bituminous paving layers, and it is strongly related to the levels of traffic-induced tensile strain at the bottom of the base which is regarded to be responsible for fatigue. Also, it controls the levels of stresses and strains in the subgrade that can lead to structural deformation. Therefore, there is growing interest in developing a practical means of measuring the stiffness.

For many years the measuring of stiffness modulus of bituminous materials was conducted with sophisticated tests such as 3-point bending tests and bending or push-pull tests. These tests are capable of measuring the complex modulus of an asphalt beam through frequency and temperature sweep tests. However, these methods are expensive and time-consuming. In order to simplify the means of measuring stiffness modulus, a form of the indirect tensile test, the so-called Indirect Tensile Stiffness Modulus (ITSM) test – nowadays called Indirect tension to cylindrical specimens (IT-CY), became available in the late 1980’s (Cooper and Brown, 1989; Brown and Cooper, 1993). The IT-CY test is undertaken using the

Nottingham Asphalt Tester (NAT) (Figure 24), developed at the University of Nottingham in response to the need for rapid, economical test methods to measure the essential mechanical properties of asphalt mixtures under repeated loading conditions.

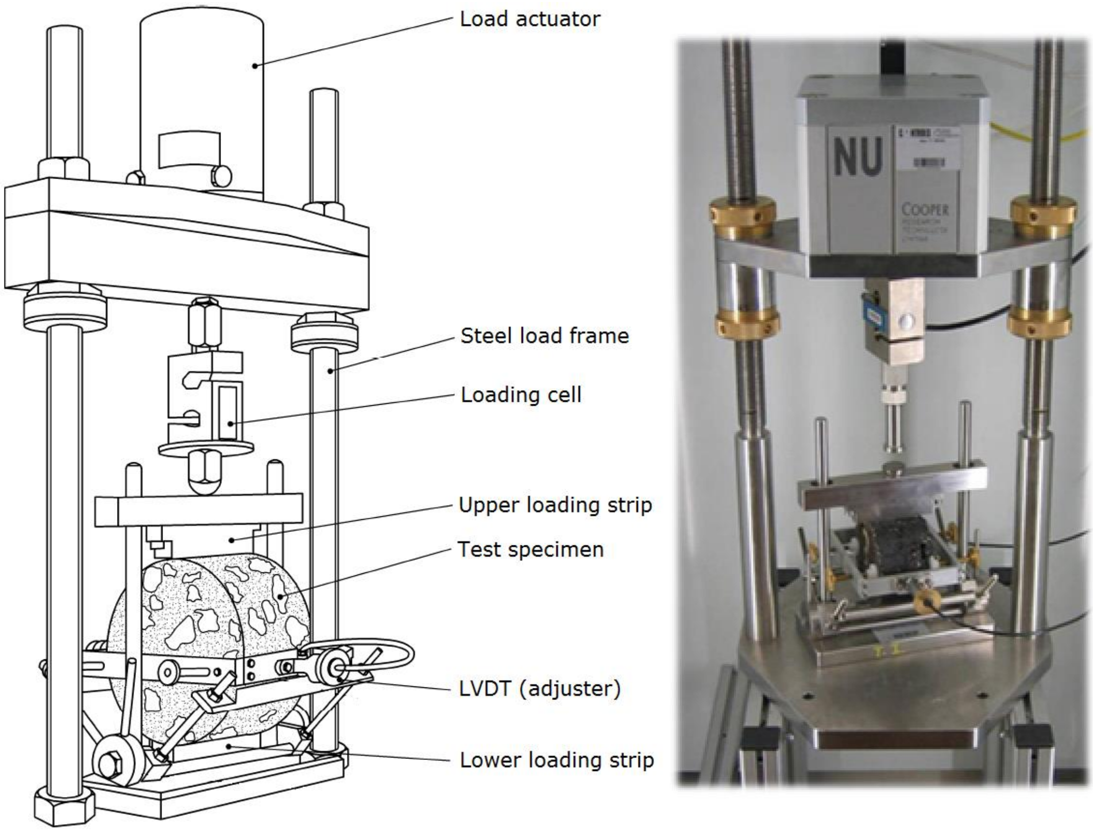


Figure 24: IT-CY Test equipment and NAT (adapted from EN 12697-26, 2012)

The NAT was designed to carry out tests on either 100mm or 150mm diameter specimens which could be produced either by moulding (compaction) in the laboratory or by coring from the pavement. The NAT comprises a pneumatic loading system, rigid test frame manufactured from stainless steel and a series of sub-systems (loading modules) that can be used to perform indirect tensile and direct compression tests (Brown and Cooper, 1993; Brown *et al.*, 1994). Data acquisition and control are computerised, and the entire system is located in a temperature-controlled unit.

The IT-CY method is described in the European Standard EN 12697-26 (2012). To avoid causing damage to the specimen during the test, the target horizontal deformation (5 microns) and the target load pulse rise-time (124 ms) should be selected for samples with 100mm in diameter. Ten (10) conditioning pulses are applied to make any minor adjustments to the magnitude of the vertical force needed to generate the horizontal deformation required and, to seat the loading strips correctly on the specimen. After that, five load pulses are applied, causing an indirect deformation on the horizontal diameter to measure the strain using two linear variable differential transformers (LVDTs). The test is then repeated after rotating the specimen through 90° and the mean stiffness from the two tests recorded as the stiffness modulus of the asphalt mixture specimen. The stiffness modulus can be calculated using the following Equation 29:

$$S = \frac{F \times (\nu + 0.27)}{z \times h} \qquad \text{(Equation 29)}$$

Where,

S = measured stiffness modulus, in mega pascals (MPa);
F = peak value of the applied vertical load, in Newton’s (N);
z = amplitude of the horizontal deformation, in millimetres (mm);
h = mean thickness of the specimen, in millimetres (mm);
ν = Poisson's ratio.

2.4.3.2. Rutting resistance

Permanent deformation is one of the most common distresses in asphalt pavements, which can be attributed to the surface or sublayers, or to a combination of effects. Rutting is defined as longitudinal depressions in the wheel tracks caused by the plastic deformation of the asphalt concrete and the granular layers/subgrade under the action of axle loads. These problems can be avoided by a selection of materials, adequate compaction and good structural design to limit the acting stresses to the permissible and safe levels (Hawks *et al.*, 1993; Bernucci *et al.*, 2008; Papaginnakis and Masad, 2008).

The susceptibility of asphaltic materials to rutting can also be evaluated in the NAT machine using the method for determining resistance to permanent deformation of bituminous mixtures subject to unconfined dynamic loading - Repeated Load Axial Test (RLAT). The RLAT is the most used test in the UK to characterise the permanent deformation behaviour of bituminous mixtures. The test was developed to investigate the behaviour of asphalt material under more realistic loading conditions (after the static load testing method), the load is repeatedly applied for a selected loading time with a rest period implemented after each loading pulse. The loading can be carried out in different waveforms such as sinusoidal, square, triangular, and trapezoidal. Authors found that the sinusoidal shape of loading is most simulative of field loading (Brown, 1976; Loulizi *et al.*, 2002).

The test is conducted according to the British Standard BS DD 226 (1996), Figure 25 shows the RLAT test in the NAT machine.

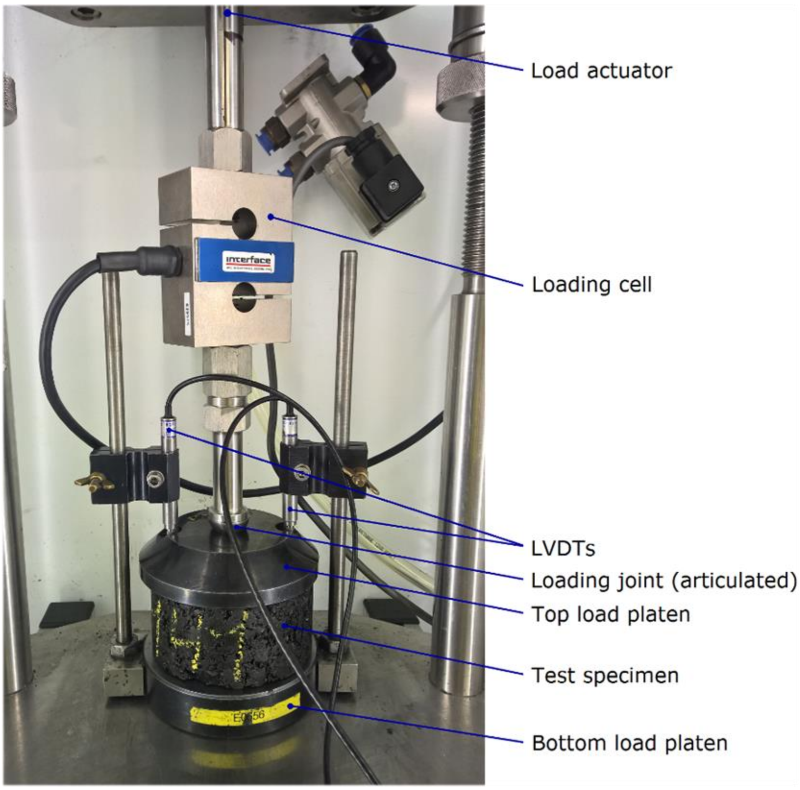


Figure 25: RLAT test in the NAT machine

A load pulse consisting of a square wave with a frequency of 0.5 Hz (1s loading followed by 1s rest period), is applied by an actuator. The load is applied vertically to the specimen, the resultant strain during the cycling load is measured along the same axis as the applied stress; the accumulated permanent strain is monitored by two LVDTs fixed on top of the upper platen.

The typical results obtained from the RLAT test for different mixtures are represented in Figure 26, where permanent axial strain is plotted against load cycles.

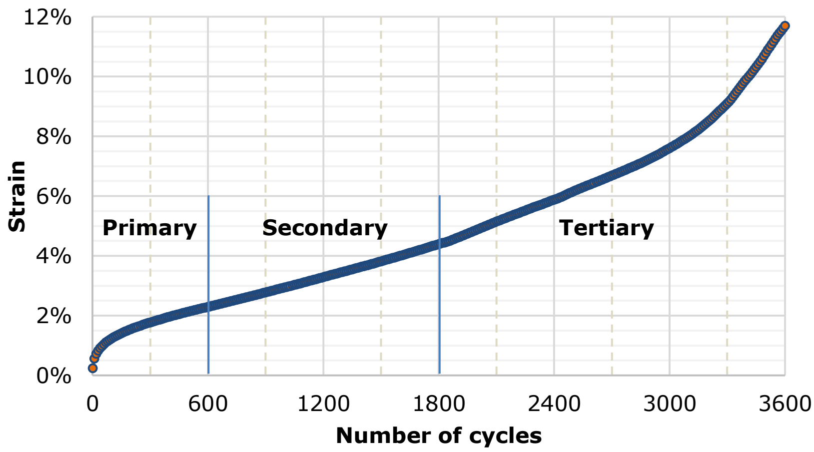


Figure 26: The three Stages of RLAT Test

This kind of plot usually gives three distinctive phases of the material. In the first or primary phase, the accumulation of vertical strain increases rapidly due to a combination of adjustment of loading platens and material densification. However, the permanent deformation rate gradually decreases. The secondary phase takes place when the strain rate reaches approximately a steady state response, and the strain increases approximately in proportion to the load cycles. The tertiary phase starts when the rate of deformation increases is rapidly indicating specimen failure.

In order to compare the permanent deformation performance of all the asphalt mixtures, the RLAT parameters, in terms of ultimate percentage strain and microstrain per cycle – the slope of the steady state phase, are calculated.

According to Airey (2004), of the two parameters, the mean strain rate can be considered to be a more reliable measure of the rutting performance of the mixtures as this parameter is independent of the initial strain experienced during the RLAT test. The methodology can me summarised as follow:

The strain rate (ε) is calculated for each cycle by using the following Equation 30:

$$\varepsilon = \frac{\varepsilon_2 - \varepsilon_1}{t_2 - t_1} \qquad \text{(Equation 30)}$$

Where, t_2 *and* t_1 are the time in seconds (number of cycle) corresponding to the ε_2 and ε_1. The slope of the steady state phase is determined from a segment including the secondary and tertiary stages, e.g. from 1000 to 3000 pulses as follows Equation 31:

$$\mu\varepsilon/cycle = \frac{\varepsilon_{3000} - \varepsilon_{1000}}{1000} \times 10^{-6} \qquad \text{(Equation 31)}$$

Where, ε_{3000} in the accumulated strain at 3000 pulses and ε_{1000} at 1000 pulses.

2.4.3.3. Fatigue resistance

The fatigue phenomenon is defined as the process of permanent, progressive and localised structural change occurring at a point in the material subject to stresses of varying amplitudes that produce the cracks leading to the totality of the fault after a given number of cycles (ASTM E 206-72, 1979). In pavements, according to Read (1996), it consists of two main stages, crack initiation and crack propagation, and is caused by tensile strains generated in the pavement by traffic loading, temperature variations and construction practices. According to Bernucci *et al.* (2008) fatigue occurs through mechanical and thermal actions that do not seem to be critical in themselves compared to the resistance under monotonic loading, but in fact they are decisive for the life cycle of the material.

Regarding the fatigue tests on asphalt mixtures, amongst the most common tests, Two-point bending, Three-point bending, Four-point bending tests and Indirect Tensile Fatigue Test are performed in Europe according to the standard EN 12697-24 (2012). There exist different fatigue tests under different loading modes. The primary distinction is made between strain-controlled and stress-controlled tests: Strain-controlled tests apply a constant cyclic strain and record decreasing the stress, while stress-controlled tests apply constant cyclic stress and measure the increasing strain. In the stress-controlled mode, once the crack appears its propagation is fast due to the stress concentration.

The Indirect Tensile Fatigue Test (ITFT) is the most commonly used fatigue test in the UK and is performed according to the Standard BS DD ABF (2003). The test is extensively used because of several reasons such as the simplicity, can be performed on field cores - allowing the correlation with lab-produced specimens and the high repeatability. The standard BS DD ABF (2003) characterises the mixtures under repeated load applications with a stress-controlled mode using the NAT machine (Figure 27).

Figure 27: ITFT set up

During the test, a haversine load signal is applied to the vertical diametric plane which causes tensile stress along the vertical diametric plane perpendicular to the load direction. The test is performed on the specimens at controlled temperature and different stress levels at a frequency of 40 ± 1 pulses per minute. The stress levels are selected to obtain fatigue laws from a range of approximately 1,000 to 100,000 cycles to failure. The failure condition is determined as the total number of load applications that cause a complete fracture on the specimen or a vertical deformation larger than 9 mm.

The maximum tensile strain generated at the centre of the specimen is defined as:

$$\varepsilon_{x\,max} = \frac{\sigma_{x\,max}(1+3v)}{S_m} \times 1000 \qquad \text{(Equation 32)}$$

Where,

ε_{max} = maximum tensile horizontal strain at the centre of the specimen in microstrain;
σ_{max} = maximum tensile stress at the centre of the specimen in kPa;
v = Poisson's ratio;
S_m = indirect tensile stiffness modulus at σ_{max} in MPa.

The maximum tensile stress at the centre of the specimen is defined with the Equation 33:

$$\sigma_{x\,max} = \frac{2P}{\pi d t} \qquad \text{(Equation 33)}$$

Where,

σ_{max} = maximum tensile stress at the centre of the specimen in kPa;
P = maximum vertical load at the centre of the specimen in kN;
d = the specimen diameter in m;
t = the specimen thickness in m.

Linear regression analysis of the ITFT results are used to determine fatigue laws for the asphalt mixtures using the following relationship (Equation 34):

$$N_f = a\ (\varepsilon_0)^{-b} \qquad \text{(Equation 34)}$$

Where,

Nf = fatigue life;
ε = initial tensile strain (microstrain);
a and b = experimentally determined coefficients.

2.4.3.4. Water sensitivity

Moisture damage significantly influences the durability of bituminous mixtures. The assessment of damage to asphalt mixtures caused by water is of great importance as it affects the performance and service life of the pavements (Bernucci *et al.*, 2008). In fact, if a pavement is not able to properly drain, the presence of water can induce a loss of the adhesive bonding between aggregates and binder and a loss of cohesive strength of the bitumen (Kandhal, 1994; Terrel and Al-Swailmi, 1994).

A reduction of cohesion results in a decrease of the strength and stiffness of the mixture and thus a reduction of the pavements ability to support traffic-induced stresses and strains. Failure of the bond between the bitumen and aggregate, the stripping, also affects the pavement support. Both mechanisms of water damage result in a fragile pavement layer and one which is prone to deform under traffic loading. Also, stripping can result in the loss of material and ultimately the total deterioration of the asphalt mixture (Airey and Choi, 2002).

Although it is recognised that it is difficult to associate laboratory test results with the performance of field mixtures, there are several tests to identify the potential for moisture damage in mixtures. They can be classified into two categories: (i) made in non-compacted mixtures and (ii) made in compacted mixtures (Solaimanian *et al.*, 2003). Since the tests for compacted samples are the most representative of real conditions and the complex problems related to adhesiveness, some existing moisture damage evaluation are: Moisture vapor susceptibility, Immersion-compression test, Marshall immersion, Freeze-thaw pedestal test, Original Lottman indirect tension, Modified Lottman indirect tension, Hamburg wheel tracking (Airey and Choi, 2002; Solaimanian *et al.*, 2003).

The water sensitivity of asphalt mixtures is usually investigated by the change in a mechanical property after immersion in water. The indirect tensile stiffness (IT-CY) and/or indirect tensile strength (ITS) are the mechanical properties that are traditionally measured for this purpose (Airey and Choi, 2002). The European Standard 12697-12 (2018) describes the ITS method and the Interim Guideline Document for the Assessment and Certification of Thin Surfacing Systems for Highways (BBA, 2013) is well known in the UK using the IT-CY test.

3. TESTING METHODS AND EXPERIMENTAL PROGRAMME

3.1. RA Degree of Binder Activation Study

According to the research shown in the previous sections, it is evident that full blending (100%) between a virgin binder and RA binder does not occur, and the properties during the mixing procedures change according to various factors such as temperature, mixing time and types of materials. In this sense, in order to maximise and optimise the incorporation of recycled material in new asphalt mixtures, it becomes fundamental to have a plausible assumption of the DoBA of a selected RA for a specific asphalt manufacturing process. In this regard, it is, therefore, necessary to assess the amount of binder released by the selected RA during the manufacturing process of the final asphalt mixture.

The approach taken was inspired by research carried out by the group at the University of Stellenbosch in South Africa, who developed a procedure called the Cohesion Test (Campher, 2012) that tested 100% RA samples by Indirect Tensile Strength Test (ITS Test) and according to the results classified the material as inactive, semi-active or active related to the degree of ageing of the RA binder. These tests were performed on different RA sources by compacting Marshall Specimens with 100% RA after mixing the material at different temperatures (previously conditioned in an oven) and then testing the samples by ITS. This test was also introduced in the framework of the RILEM TC 237-SIB on Cohesion Test for Recycled Asphalt (Tebaldi *et al.*, 2018) which tried to assess whether ITS values of samples prepared with the cohesion tests could be identified, so to explore the potential to characterise RA with an easy-to-perform test that did not require binder recovery.

In fact, one of the reasons for moving forward with this method is that during the mixture design phase in the lab, it is relatively simple to recover binders and have all the information about the RA binder, but during construction in the field, the time available for quality control does not allow all necessary tests that are possible to carry out in the lab to be performed. Furthermore, it must be underlined that bitumen recovery is an operation that was adapted for the use of RA in new mixtures because it was already carried out in asphalt laboratories to allow practitioners to perform a new mixture design by carrying out well-known material characterising such as testing recovered binders and aggregates. However, it is believed that RA is a composite material that due to its nature will possibly behave in far more complex manner than those well-known so far. In other words, characterisation of RA should possibly be performed on the RA itself directly, and this material should be considered, in new mixture design, as a component on its own that can interact with the other components, such as binder and grading curves, and therefore design must be adapted to account for this fact.

3.1.1. RA-DoBA Labelling: Introduction

On this basis, the present investigation tried coupling the potential of the cohesion test to be a wide-spread method to characterise RA with the need of having a label for the DoBA of RA at different processing temperatures and time. The principle behind this investigation is simple: if a selected RA has latent binding properties, which can be seen in aggregates surrounded by "active" bitumen, the total energy, pre-peak energy, post-peak energy, flexibility and relative tensile strength of a sample of 100% of this RA mixture should be higher than those of a mixture of 100% of RA surrounded by "inactive" bitumen (Harvey, 2010; Choudhary *et al.*, 2012; Jitsangiam *et al.*, 2012; Katman *et al.*, 2012; Jaya and Asif, 2015). This should vary and possibly be more evident if RA is processed at higher temperatures and longer times.

In order to verify this assumption, an experimental programme with the use of an artificially aged control RA could provide parameters and indexes by which a practical methodology to label RA based on its DoBA could be derived. Figure 28 shows the experimental programme crafted around this idea which aims at comparing the binding properties of a 100% RA mixture with a control manufactured at the same temperature, with the same white grading curve of the selected RA and with a binder artificially aged to have similar properties to that recovered from the selected RA. Regarding DoBA, this will allow an uncertain scenario (100% RA) to be compared with a "full activation" scenario (control). The two designed mixture were therefore manufactured, compacted and tested with the Indirect Tensile Test (ITT) in identical conditions so that indexes can be created on the bases of the resulting ITT.

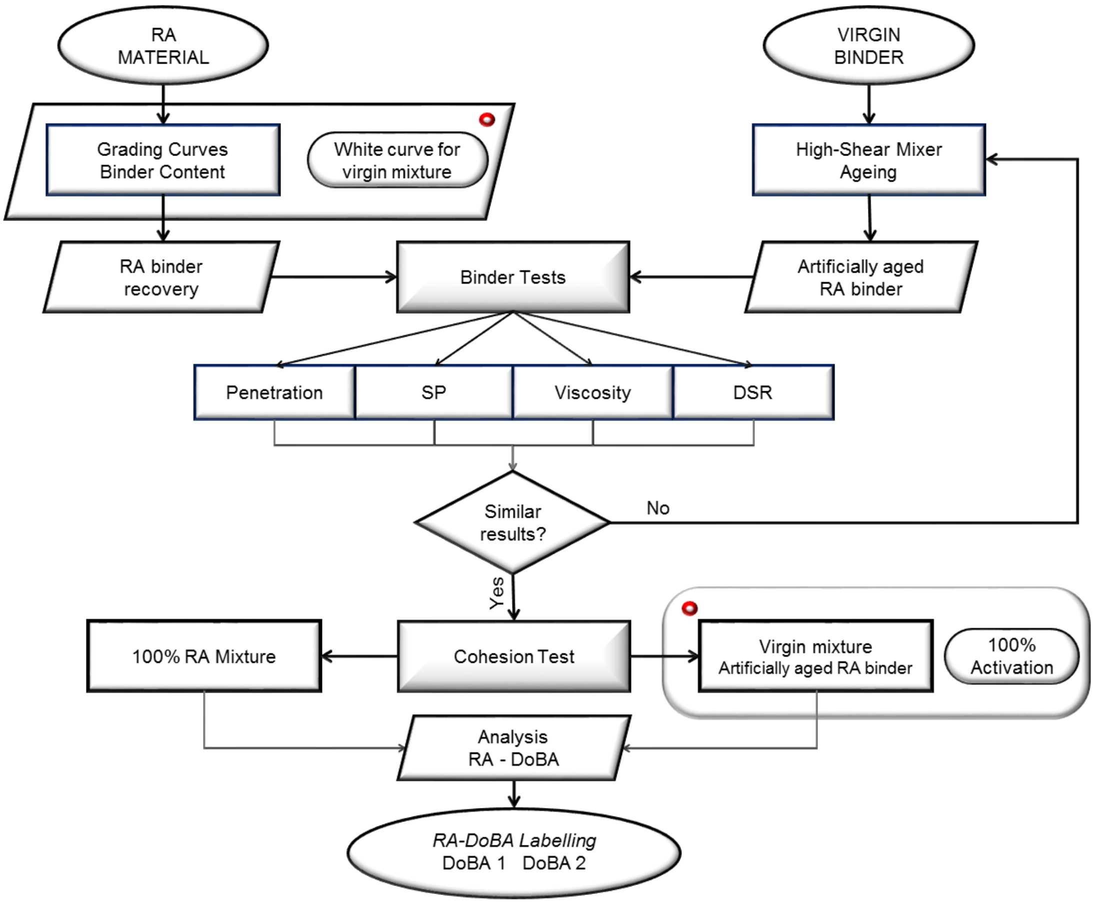


Figure 28: Flowchart RA-DoBA labelling framework

The primary objective of this study is the creation of an RA-DoBA labelling from ITS results coming from two different materials: RA and Artificial RA Mixture. To compare results, obtain the index and estimate activation values for a specific RA material, the Cohesion Test can provide an interesting variation of results for further analysis. The idea to get comparable results is by creating an artificial RA mixture with similar characteristics of an original RA, which includes the same conditions such as bitumen characteristics, grading curves and RA aggregates origin.

The principle of this investigation is if the artificial RA mixture is created, it can be assumed that full activation happens. The bitumen with similar characteristics of the RA binder is mixed with virgin aggregates, 100% of it will be involved in this blending, meaning that 100% activation occurs, independent of the mechanical test results. The results of the artificial RA using ITT is now comparable with 100% RA mixture ITT results making it possible to create the index for this investigation.

3.1.2. Aged binder creation

The first step is selecting the RA for characterisation is recovering the bitumen from the RA for simple tests such as Penetration, Softening Point, Viscosity and DSR Frequency Sweeps. These bitumen tests are the main point of comparison with the artificially aged bitumen which is achieved through some ageing procedure. A neat virgin binder 50/70 penetration was selected for the ageing procedure in order to create the artificially aged binder in this research.

The bitumen ageing procedures commonly used are Thin-film Oven Test – TFOT (EN 12607-2, 2014), Rolling Thin-film Oven Test – RTFOT (EN 12607-1, 2014), Rotating Cylinder Ageing Test – RCAT (EN 15323, 2007) and Pressure Ageing Vessel – PAV (EN 14769, 2012). In terms of long-term ageing methods, which would be appropriate to achieve the RA binder's characteristics, only PAV and RCAT are feasible options. The problem with these two procedures is that only a limited amount of material can be aged at the same time, and since the working plan intends to evaluate a wide range of specimens, the amount of aged bitumen is relatively large. Thus, a procedure proposed by Wu (2009) was chosen due to the processing facility, equipment availability and positive results obtained in the research.

This consists of a high-shear mixing (HSM) ageing simulation of the bitumen at a selected time and temperature. In the ageing procedure, the desired volume (4 litres in this study) of hot bitumen is placed in a clean tin on the hotplate below the mixer (entire system located in a fume cupboard) as shown in Figure 29. The shear mixing head is then lowered into the tin. The temperature of the hotplate is controlled to achieve the bitumen temperature at 163°C which is the temperature of the RTFOT. The mixer is operated at a speed of approximately 3500 rpm with the top surface of the binder being exposed to air. During the High-Shear ageing procedure, bitumen samples were taken at fixed times (every 6 hours in this research) to measure the conventional and rheological properties. When it reaches similar properties to the RA bitumen, the ageing procedure is finished.

Figure 29: Bulk ageing procedure using HSM

In order to compare the recovered bitumen from RA sources with the artificially aged binder (ArtRAb), samples were taken every 6 hours of ageing (up to 36 hours), and the following characterisation was performed:

Penetration: Needle penetration at 25^{0}C (EN 1426, 2015);

Softening Point: Ring and ball softening point (EN 1427, 2015);

Viscosity: Rotational viscosity determination - 100 to 200^{0}C (EN 13302, 2010);

DSR: Frequency sweep 0.1-10 Hz; Temperature sweep 0-80°C (EN 14770, 2012).

3.1.3. Cohesion Test: Experimental programme

The main objective of this study is the creation of Indexes from ITT results coming from two different materials: RA and Artificial RA Mixtures. In order to compare results, obtain the indexes, and estimate the binder activation values for a specific RA material, the Cohesion Test can provide an interesting variation of results for further analysis. The idea to get comparable results by creating an artificial RA mixture with 100% full activation scenario and similar characteristics of an original RA which includes two main conditions. Firstly similar ageing of the bitumen and, secondly, similar grading curve of the mixture. The loose mixtures are then subjected to the cohesion test and the obtained results are compared in terms of Indirect Tensile Strength, Total Energy, Pre-peak Energy, Post-peak Energy and Flexibility Index.

3.1.3.1. Mixing and compaction

In order to analyse the RA variability, a range of mixing temperatures and times (Figure 30) were selected for testing those three RA sources (further details in Chapter 4).

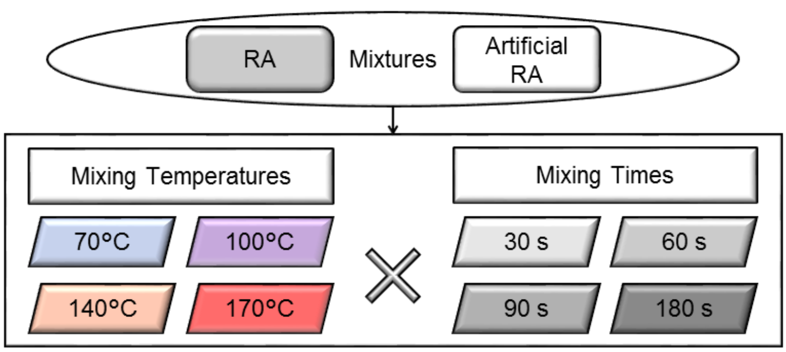


Figure 30: Cohesion Test – Procedures

The Figure 30 shows what types of mixtures are analysed in the proposed methodology: Reclaimed Asphalt and Virgin Reference, here called Artificial RA. The second part of the flowchart shows the different mixing times and temperatures selected to perform the methodology. All the situations were combined forming Table 4 where the RA1 material was tested first, and then the total amount of samples were adjusted for the RA2 and RA3.

Table 4: Cohesion test variations and number of samples tested

MIXING		RA1		RA2		RA3		ART 1	ART 2	ART 3
Temperature	**Time**	*DRY*	*WET*	*DRY*	*WET*	*DRY*	*WET*	*DRY*	*DRY*	*DRY*
70°C	30s	3	3	-	-	-	-	-	-	-
	60s	3	3	3	3	3	3	5	5	3
	90s	3	3	-	-	-	-	-	-	-
	180s	3	3	3	3	3	3	-	-	-
100°C	30s	3	3	-	-	-	-	-	-	-
	60s	3	3	3	3	3	3	5	5	3
	90s	3	3	-	-	-	-	-	-	-
	180s	3	3	3	3	3	3	-	-	-
140°C	30s	5	4	-	-	-	-	-	-	-
	60s	5	4	5	4	3	3	5	5	3
	90s	5	4	-	-	-	-	-	-	-
	180s	5	4	5	4	3	3	-	-	-
170°C	30s	5	4	-	-	-	-	-	-	-
	60s	5	4	5	4	3	3	-	-	-
	90s	5	4	-	-	-	-	-	-	-
	180s	5	4	5	4	3	3	5	5	3
		64	56	32	28	24	24	20	20	12
	TOTAL	280								

As the idea is to measure and understand the reactivated bitumen from the RA, it is important to identify the main mechanisms that can provide better results. In this sense, four different temperatures were chosen from those commonly used in HMA (170°C) to lower temperatures (70°C) which can also present the potential reactivation of the bitumen. In addition to these temperatures, four different mixing times were chosen for RA1, starting with 30 seconds (usual in asphalt plants) up to 180 seconds. These variations were chosen due to the differences found in the degree of blending by Bowers, Moore, *et al.* (2014) when mixing at different times.

There are some common steps that apply to all mixtures: The RA is dried in oven at 40°C for 48 hours; RA is properly selected using a rifle box and quartering; the specimens are compacted using a Marshall Compactor, 50 blows each side of the sample; mechanical mixing (chosen due to high manufacturing requirements); and conditioning the RA materials at oven temperatures for 4 hours prior mixing. These procedures were adopted in accordance with previous studies (Campher, 2012). The Marshall moulds were used for compaction, the usual sizes of its specimens are 100mm in diameter and 63.5mm in height.

The maximum density of the RA material is determined (EN 12697-5, 2009) and in order to achieve the target height, the air voids content are estimated firstly from previous results from RILEM TC SIB 237 - TG-6 (Tebaldi *et al.*, 2018) and then from specimen trials. Then, is possible to determine the amount of material that should be placed in each mould for further compaction to produce the final samples.

A different procedure is performed when manufacturing artificial RA mixtures: All mixtures are pre-blended at high temperatures (170°C) as if they are virgin materials; then conditioned in the oven for 1 hour in order to reach the desired temperature (except for 170°C mixtures where compaction is carried out just after mixing); 60 seconds mixing again for those mixtures with temperatures lower than 170°C, then compaction using Marshall Compactor with the same procedure as used for the RA mixtures. The 60s mixing time prior to compaction was chosen due to the results presented by RA to allow both cases to be comparable.

3.1.3.2. Cohesion test: Indirect Tensile Test (ITT) and indexes VS DoBA

The Indirect Tensile Test (ITT) has proven to be an important test procedure for characterising materials such as Portland cement concrete and asphalt mixtures, due to the difficulty of obtaining the tensile strength directly. The arrangement of this test is through the application of two diametrically opposite concentrated compression forces on a cylinder specimen. The vertical loading produces both a vertical compressive stress and a horizontal tensile stress on respective diameters of the specimen. This test became very popular in the world due to the ease and speed of execution. From the ITT, load and vertical deformation are recorded and used to analyse different parameters in this research: Indirect Tensile Strength (ITS) (Peak-load); Total Energy (whole test); Energy (Pre and Post-peak load); and Flexibility Index (whole test). The properties analysed were chosen based on the literature where researchers have found that the ITT results are affected by the optimum binder content (OBC) of the mixtures, where different binder contents – from the OBC of the design – presented reduction on results based on ITS values (Harvey, 2010; Choudhary *et al.*, 2012; Jitsangiam *et al.*, 2012; Katman *et al.*, 2012). The idea behind the investigation of the DoBA does match with those results related to the OBC, due to the fact that the activation of the binder from the RA will be acting during the test, and the portion of inactive bitumen can be considered as part of the optimum binder that is missing in the mixtures – in the case of the literature review where researchers compared mixes with OBC and reduced percentage of bitumen. In this sense, supported by the literature as well as aiming to investigate other parameters such as the Total Energy and Flexibility Index, the present thesis includes these properties for analysis in order to have extra results that are also related to the whole test and not only the peak load. This provides an idea about the energy absorbed during the procedure and its effect before and after the cracking.

3.1.3.2.1. Indirect Tensile Strength

The test follows the standard EN 12697-23 (2017) Bituminous mixtures — Test methods for hot mix asphalt — Part 23: Determination of the indirect tensile strength of bituminous specimens. The cylindrical test specimens were used in this research, 100mm in diameter and height between 35mm and 75mm (Figure 31). The tests were carried out with temperature control of the samples, 25°C in this research following the procedure developed by Campher (2012). The peak load from each specimen is used to analyse the results for the ITS parameter and is determined according to Equation 35. The load is applied through the 12.7 mm wide loading strip with adequate curvature of the cylindrical specimen, the current standards that uses the Equation 35 do not consider the influence of these loading strips in the ITS.

$$\mathrm{ITS} = \frac{2 \times P}{\pi \times D \times H} \qquad \text{(Equation 35)}$$

Where,

ITS = indirect tensile strength, expressed in mega pascals (MPa);
P = is the peak load, expressed in newton (N);
D = is the diameter of the specimen, expressed in millimetres (mm);
H = is the height of the specimen, expressed in millimetres (mm).

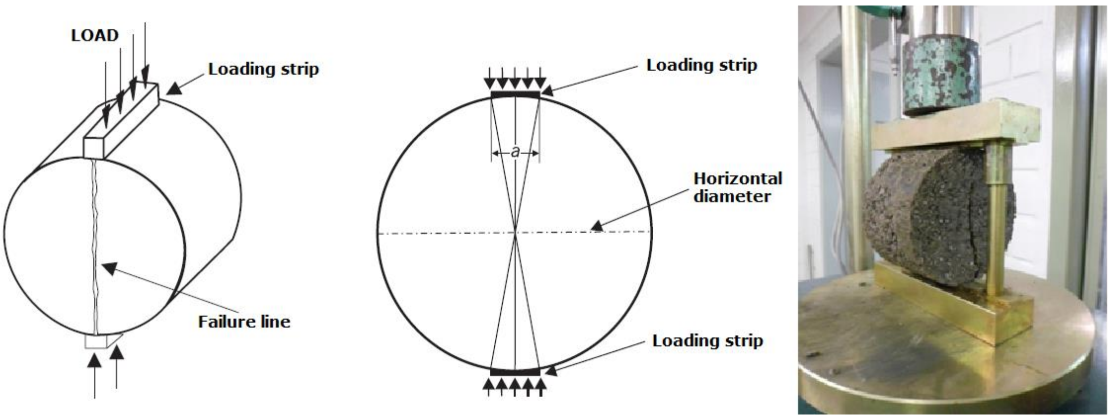


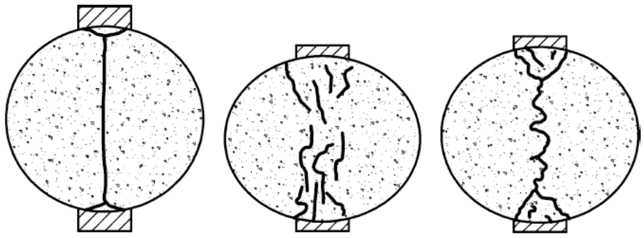
Figure 31: ITS Test scheme

According to the current standard specifications, the use of the Equation 35 to calculate the ITS for asphalt mixtures assumes that the test specimen breaks due to the uniform tensile stress generated along the requested diameter that equals with the maximum allowable tension, which is in an elastic behaviour throughout the test. Increasing the loading strip width to the same applied force P reduces the requesting tensile stress. The effect of the loading strip width on the strength of asphalt mixtures at different temperatures is presented by Falcão and Soares (2002). Due this, the standard EN 12697-23 (2017) recommends to record the type of failure according to **Figure 32**, categorised as:

a) Clear tensile break: Specimen clearly broken along a diametrical line, except perhaps for small triangular sections close to the loading strips;

b) Deformation: Specimens without a clearly visible tensile break line;

c) Combination: Specimens with a limited tensile break line and larger deformed areas close to the loading strips.



a) b) c)

Figure 32: ITS types of failure

3.1.3.2.2. Test Energy

The use of an energy approach for characterisation, as opposed to a strength-based approach, allows for better characterisation of the cracking processes in asphalt mixtures. The test energy in the ITT can be associated as the energy required to create a unit surface area of a crack (AASHTO TP105, 2013). The energy (E) is determined by first calculating the work (W), defined as the area under the load-displacement curve (Putman and Amirkhanian, 2004; Vasconcelos *et al.*, 2012). This work can be divided into pre-peak ($W^{Pre\text{-}peak}$), associated with energy necessary to initiate a crack, and post-peak fracture work ($W^{Post\text{-}peak}$) associated with energy needed to propagate the crack, as shown in Figure 33. The total work can be estimated according to Equation 36:

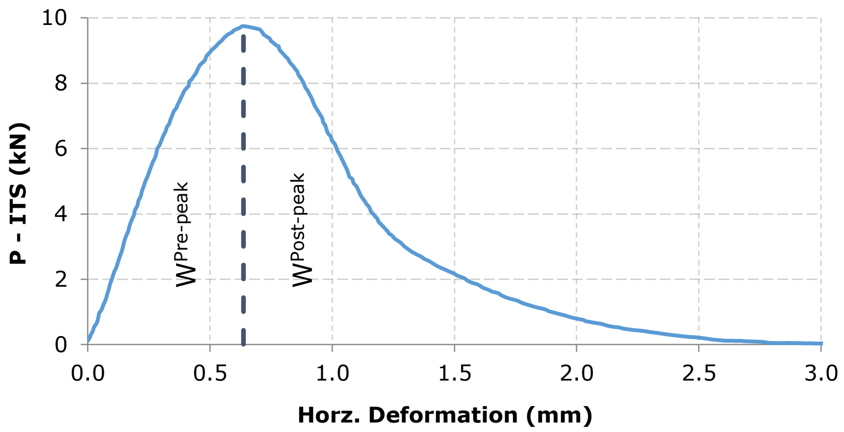


Figure 33: Fracture work definition

$$W = W^{\text{Pre-peak}} + W^{\text{Post-peak}} = \int_0^{\Delta_{\text{Pmax}}} P.dx + \int_{\Delta_{\text{Pmax}}}^{\Delta_{\text{Pfinal}}} P.dx \qquad \text{(Equation 36)}$$

Where,

W = is the total work expressed in kN.mm;

P = is the load, expressed in kilo Newton (kN);

x = is the horizontal displacement, expressed in millimetres (mm).

With the W results, the total energy can be calculated by normalising the total work by the area of fracture surface that is generated during the test. This area can be estimated as a product of the height of the specimen (H) and the length of new crack formed during the test. This crack length is often referred to as ligament length – the specimen diameter (D) in the ITT on cylindrical specimens. Total energy calculations are determined for the pre-peak and post-peak regions in order to distinguish the crack propagation process from the complete test, Equations 37 to 39 are presented below:

$$E_{\text{Total}} = E^{\text{Pre-peak}} + E^{\text{Post-peak}} \qquad \text{(Equation 37)}$$

$$E^{\text{Pre-peak}} = \frac{W^{\text{Pre-peak}}}{H \times D} \qquad \text{(Equation 38)}$$

$$E^{\text{Post-peak}} = \frac{W^{\text{Post-peak}}}{H \times D} \qquad \text{(Equation 39)}$$

Where,

E = is the energy expressed in J/m^2;
H = is the specimen height (thickness), expressed in millimetres (mm);
D = is the specimen diameter, expressed in millimetres (mm).

3.1.3.2.3. Flexibility Index

The flexibility index (FI) is a parameter that can describe fundamental fracture processes and overall patterns of load-displacement curves and can determine the cracking potential of asphalt concrete mixtures (Al-Qadi *et al.*, 2015; Ozer *et al.*, 2016). The authors show that low-temperature fracture testing is variable for distinguishing between different mixtures and provided evidence that fracture energy alone cannot be used to differentiate between some mixtures. However, fracture testing at an intermediate temperature (25°C) provides the desired distinction according to the authors. The conclusion is attributed to the nature of the fracture energy parameter. Depending directly on the shape of the load-displacement curve, the fracture energy is a function of both the strength (defined by peak load) and ductility (defined as the maximum displacement at the end of the test) of the material. If the material displays a high peak load, it may compensate its fracture energy for the lack of ductility in the post-peak region of the load-displacement curve. This is a potential explanation for why brittle asphalt mixtures with high-RA amounts may display similar or sometimes higher fracture energy values than their reference mixtures used for comparison. Figure 34 presented by Al-Qadi *et al.* (2015) illustrates a comparison of two mixtures, the fracture energy values of the two materials are virtually identical; however, the mixtures have distinctive load-displacement characteristics that may significantly differentiate their cracking response.

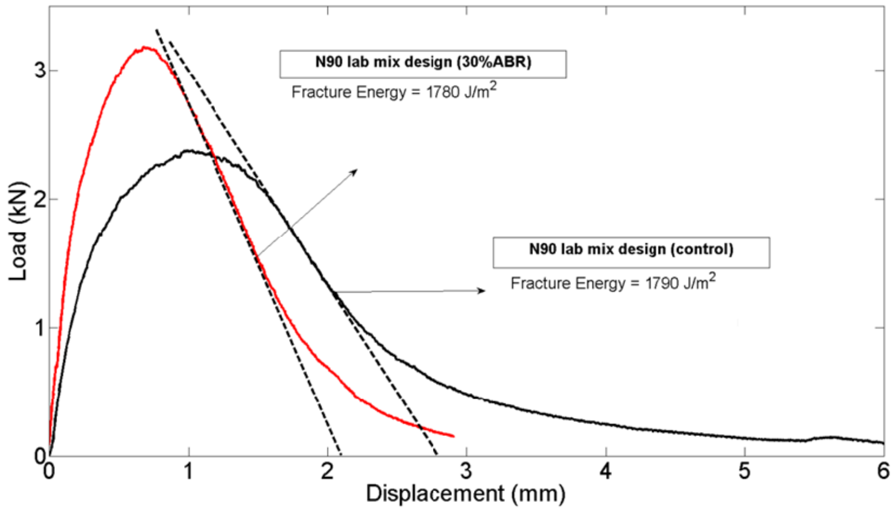


Figure 34: Load-displacement curves from fracture test (Al-Qadi et al, 2015)

As already mentioned, the FI is a parameter developed to describe fundamental fracture processes of load-displacement curves as well as determine the cracking potential of asphalt concrete mixtures. In order to calculate FI, the authors (Al-Qadi et al., 2015; Ozaer et al., 2016) provide the Equation 40:

$$FI = \frac{G_f}{m} \quad \text{(Equation 40)}$$

Where,

FI = is the Flexibility Index;
G_f = is the fracture energy expressed in J/m^2;
m = is the slope of the curve at an inflection point.

3.2. Binders characterisation and tests

A wide range of tests are performed on bitumen, from specifications tests to more fundamental, rheological and mechanical tests. In total, six bitumens are included in this research for investigation, two reference binders (virgin and pure RA1) and four recycled binders (applying different DoBAs). As a wide variety of bitumen is manufactured, it is necessary to have tests to characterise different materials. In this sense, the conventional properties of the binders are determined together with the rheological properties in order to characterise the materials. Moreover, the selected recycled and reference binders have their performance-related properties investigated, with tests regarding fatigue, rutting and thermal cracking. Therefore, the whole binders' experimental work is to be used further in the RA DoBA verification with the recycled mixtures.

3.2.1. Conventional properties

To specify particular binders as suitable for pavements, most countries use simple measures of physical characteristics which are easy to implement in laboratories. The two most common specifications are Penetration-graded, measuring the "hardness" by standard needle penetration test, and Viscosity-graded, through the resistance to flow by viscosity tests (Bernucci *et al.*, 2008; Koenders, 2015).

According to Airey (1997), the primary purposes of these specifications are to grade bitumen according to its consistency, so do not address specific distress modes or ensure long-term field performance. The author also mentions that because both specifications were developed based on empirical tests, it was difficult to have a reliable correlation between the criteria of specification and pavement performance. The traditional tests used to characterise bitumen are shown as follow.

3.2.1.1. Penetration

Penetration is a measure of the bitumen consistency through the Needle Penetration Test (EN 1426, 2015), expressed as the distance in decimillimetre which a standard needle penetrates vertically into a standardised volume sample of the bitumen under specified conditions such as temperature, load and load duration. The usual conditions are a loading time of 5 seconds; applied load is 100 grams and the test temperature at 25°C. In each test, three individual penetration measures are carried out. The average of the three values is recorded and accepted if the difference between the three measures does not exceed a specified limit in the standard. The consistency of the bitumen is higher, the smaller the penetration of the needle.

3.2.1.2. Softening Point

The softening point (SP) is an empirical measurement which relates to the temperature at which bitumen softens when heated under certain specific conditions and reaches a particular flow condition (beginning of fluidity range). The SP is determined through the Ring and Ball Test (EN 1427, 2015). A steel ball with specified dimensions and weight is placed in the centre of a bitumen specimen that is contained inside a standard metallic ring. The entire assembly is placed in a water bath or glycerine in a glass beaker. The bath is heated at a controlled rate of 5°C/minute. When the bitumen softens enough to no longer support the weight of the ball, the ball and the bitumen movie towards the bottom of the beaker. The temperature is marked at the instant the softened bitumen touches the bottom plate from the standard test set. The test is conducted with two samples of the same material at the same time in the same bath. If the temperature difference between the two samples exceeds the values determined by standard (1°C when results below 80°C or 2°C above), the test must be redone.

3.2.1.3. Fraass Breaking Point

In 1937 the researcher Fraass proposed a test method to qualify bitumen on condition of freezing temperatures (as low as -30°C), which primarily consists in seeking to determine the temperature that causes the bitumen to reach a critical stiffness that results in cracking. Many countries that have very cold winters have maximum values of "Fraass temperature" in their bitumen specifications (Bernucci *et al.*, 2008; Koenders, 2015).

It is the temperature at which the bitumen, when subjected to bending, tends to break more pronouncedly than flow. In the standard test (EN 12593, 2015), a steel plate of 41mm × 20mm, coated with a thin layer of bitumen, bent under standard conditions, is subjected to decreasing temperatures. The breaking point is the temperature at which the first crack appears in the bitumen film. This test measures the minimum temperature at which the material resists bending.

3.2.1.4. Viscosity

The structural model of the binder as a dispersion of polar molecules in a non-polar medium helps to understand the effect of temperature. At very low temperatures the molecules are unable to move about amongst each other, and the viscosity is very high, the binder in this situation behaves almost like a solid. As the temperature increases, some molecules start to move and there may even be a flow between the molecules. With the movement increasing, the viscosity is reduced, and at high temperatures, the binder behaves as a liquid, in a reversible transition (Bernucci *et al.*, 2008).

Currently, the viscometer most used in the United States and Europe for bitumen measurement is called the Brookfield Viscometer, which gives the viscosity-temperature curve over a wide range of determination on the same sample.

The Brookfield viscometer can measure the consistency properties related to pumping and storage. It also enables a chart of temperature-viscosity for the mix design to be obtained by measuring the behaviour of the fluid at different shear rates and stresses obtained by rotating coaxial cylinders that are immersed in the test sample. Essentially, the device consists of a thermostatically controlled chamber containing a sample of hot bitumen. Various types of rods, called spindles, can be utilised and, for each type of material or temperature range it is necessary to specify the correct spindle number. The torque required to rotate the spindle is measured and converted into the viscosity of the bitumen (Koenders, 2015). It is a measure of the dynamic viscosity expressed in centipoise (cP), the viscosity measurement unit in the international system is the Pascal second (Pa.s). The rotational viscosity of bitumen is usually determined at 135°C, but with this type of apparatus, the viscosity can be determined over a relatively wide range of temperatures, in this research was used between 100°C and 200°C. The test is regulated by the European Standard EN 13302 (2010).

3.2.1.5. Temperature susceptibility

Temperature susceptibility indicates the sensitivity of the consistency of bitumens to temperature variations. This is an essential property of bitumens since if they are very susceptible to changes in state or properties depending on temperature variation, they will not be desirable in pavements. The penetration index (PI) was developed by Pfeiffer and van Doormaal in 1936, and measures the temperature susceptibility of a bitumen that can be derived mathematically, from the standard penetration (at 25°C) and SP values (Equation 41), as given in the following equations (Koenders, 2015; Southern, 2015):

$$PI = \frac{1950 - 500\log(pen) - 20SP}{50\log(pen) - SP - 120} \quad \text{(Equation 41)}$$

Bernucci *et al.* (2008) classify that values higher than +1 indicates oxidised bitumen (low sensitivity to high temperatures and brittle at lower temperatures), and smaller values (-2) indicate high sensitivity to temperature variation. According to Southern (2015), the values of penetration index for unmodified bitumens range from around -3 for highly temperature-susceptible bitumens to around +7 for highly-oxidised bitumens with low-temperature susceptibility. For paving-grade bitumens used for highways, the typical range is -1.5 to +1.0.

3.2.2. Bitumen ageing

Bitumen properties change over the lifetime of the pavement due to the combined action of heat during the manufacturing process, evaporation of volatile components, and reactions with atmospheric oxygen (Bernucci *et al.*, 2008; Southern, 2015). Bitumen ageing is one of the principal factors causing the deterioration of asphalt pavements. Important ageing related modes of failure are traffic and thermally induced cracking, and ravelling (Lu and Isacsson, 2002).

Bitumens suffer short-term ageing during the mixing, handling, and application stage of the asphalt mixture. Therefore, the properties of the binder will change from the fresh condition to the point where the asphalt is placed into the pavement. The long-term ageing of the bitumen takes place through the whole service life of the pavement, which will be subjected to various environmental factors (Lu and Isacsson, 2002; Airey, 2003; Southern, 2015).

The primary mechanisms related to ageing are oxidation, loss of volatiles, exudative hardening and physical hardening. Oxidative hardening is considered to be the leading cause of bitumen ageing. These effects are due to the chemical composition and structure of bitumens, which can be divided into asphaltenes and maltenes. The asphaltenes are the heavy particles of bitumen (high molecular weight), therefore, the higher the asphaltene content, the lower the penetration and the higher the softening point and viscosity. During hardening, asphaltenes content increases, producing changes in bitumen properties and rheology and obtaining a harder binder (Airey, 1997).

The phenomenon of hardening over time is a slow process, and there is a need for a laboratory ageing and conditioning procedure. In this sense, several methods have been developed to simulate the ageing process and provide a sample of aged bitumen that can be tested within a reasonable period (Southern, 2015). Various ageing and conditioning regimes are standardised for use in specifications and are intended to be used to simulate Short-Term Ageing (STA) and Long-Term Ageing (LTA). The methods selected for this study are shown below.

3.2.2.1. Rolling Thin-film Oven Test – RTFOT

The test follows the European standard EN 12607-1 (2014) and was designed to reproduce STA. It was developed by California Division of Highways in 1963 as an improvement of TFOT. This test also measures the ageing by oxidation and evaporation, but is more severely by continually exposing a new portion of the bitumen to the effect of air.

In this test, 35g of (in a thin film of approximately 1.25mm) bitumen is continuously rotated in a glass container at 163ºC for 75 minutes while blowing hot air into each sample bottle at its lowest travel position. A maximum of 8 containers can be tested at the same time due to equipment restrictions.

3.2.2.2. Pressure Ageing Vessel – PAV

The test follows the European standard EN 14769 (2012). The PAV was developed as part of the SHRP project to simulate LTA. The procedure is carried out using bitumen that has already been subjected to STA, RTFOT in this research, followed by oxidation of the residue in a pressurised ageing vessel. The PAV apparatus consists of the pressure ageing vessel and a forced draft oven. The pressure vessel is designed to operate under the pressure and temperature conditions of the test.

In the Superpave specification AASHTO MP-1 (1998), the PAV procedure entails ageing bitumen in 140mm diameter steel trays within a heated vessel, pressurised with air to 2.07MPa for 20h at temperatures between 90 and 110 °C. The vessel should accommodate 10 sample trays and uses a rack that fits into the vessel. The method can prepare relatively large quantities of the binder; each tray can hold 50g of bitumen. After 20h, the pressure is gradually released, the trays are removed from the rack and placed in an oven at 163°C for 30 minutes to remove entrapped air from the samples, after this the samples are ready for further testing. In this study, the temperature selected was 90°C.

3.2.3. Rheological testing

As already mentioned, the DSR is used to characterise the bitumens rheology, obtaining the viscous and elastic behaviour by measuring the G* and δ. The G* is a measure of the total resistance of a material to deformation when exposed to repeated pulses of shear stress, and consists of both elastic (recoverable) and viscous (non-recoverable) components. The δ is an indicator of the relative amounts of recoverable and non-recoverable deformation. Both values (G* and δ), for bitumens, are highly dependent on the temperature and frequency of loading (Asphalt Institute, 1994). In this sense, the unaged, STA and LTA binders were tested for the Frequency and Temperature Sweep test in the DSR.

3.2.3.1. DSR - Frequency & Temperature Sweep Test

In a frequency sweep test, the dynamic loading apparatus applies a range of frequencies to the bitumen sample at a fixed strain/stress. In order to obtain representative results, the strain/stress applied has to be within the limit of the linear viscoelastic region.

Temperature sweep tests are used to investigate the temperature susceptibility of the asphalt binders. In this test, frequency and strain are fixed, and temperature changes over the desired range. Typically, this test is combined with frequency sweep test so that a complete characterisation of the material is obtained.

The details and conditions applied to this research as follow:

- Plates diameter (ø mm) and gap: 8ø and 2mm gap; 25ø and 1mm gap;
- Frequencies (Hz): 0.1, 0.16, 0.25, 0.4, 0.63, 1, 1.59, 2.52, 4, 6.38, 10;
- Temperatures (°C): 0, 10, 15, 20, 25, 30 and 40°C using 8ø plates;
  30, 40, 50, 60, 70 and 80°C using 25ø plates;

The data obtained from DMA from DSR tests need to be represented in a useful form, and there are different types of demonstration, the most common plots are isochrones, isothermals, master curves, black diagrams and Cole-Cole diagrams (Airey, 1997).

3.2.3.2. Bending Beam Rheometer (BBR)

In order to characterise binders at low temperature, the BBR is widely used over the world. The test procedure is in accordance with the European standard EN 14771 (2012) as already mentioned in Section 2.4.1.3. In this research, the average results from three tested beams was obtained in order to determine the creep stiffness (S) and the relaxation capacity (m-value) of the binders. The test temperatures vary for each binder, in this study they were between -12 and -30°C.

3.2.4. Bitumen performance-related tests

3.2.4.1. Rutting: Multiple Stress Creep and Recovery (MSCR)

The MSCR test was selected to be performed in the recycled and reference binders. The European Standard EN 16659 (2015) describes the test procedure and analysis method. The details were already exposed in Section 2.4.2. Although this test has been developed in order to investigate the rutting resistance on RTFOT aged binders, it was also tested on the neat and PAV state for analysis purpose in this research. Regarding the test temperatures, the European standard EN 16659 (2015) recommends to run the MSCR test at an appropriate temperature from 50-80°C. In this research two temperatures were chosen due the nearest high critical temperature of the investigated binders and the reference binder as well as for analysis comparison: 60°C for all rejuvenated and virgin binders, in all stages of ageing (neat, STA and LTA); and, 50°C for STA binders for further comparison with rutting tests on asphalt mixtures to investigate the RA DoBA. Also, two repetitions for each bitumen to reduce the possible errors were tested, an average of the two measurements is applied.

3.2.4.2. Fatigue: Linear Amplitude Sweep (LAS)

The LAS test was selected to be performed in the recycled and reference binders. The LAS test is carried out following the standard AASHTO TP 101 (2014), the details were already exposed in Section 2.4.2. The test is run using the DSR in two steps: a frequency sweep (from 0.1 Hz to 30 Hz with a strain level of 0.1%) followed by an amplitude sweep (at 10 Hz with a linear increase of strain from 0.1% to 30%). The temperature selected for general fatigue tests is commonly 20°C, and for being related to the PG intermediate critical temperatures, it was also chosen for this investigation. Furthermore, three repetitions for each bitumen to reduce the possible errors were tested, an average of the three measurements is applied. Although the tests are required to be performed only on LTA samples (RTFOT+PAV), neat and RTFOT aged samples were also tested. Analysing the test results, the fatigue resistance is obtained using the viscoelastic continuum damage (VECD) theory. Therefore, the obtained results are used to correlate with the asphalt mixtures fatigue tests.

3.2.4.3. Low-temperature cracking: Thermal cracking potential (BBR)

In order to characterise thermal cracking resistance of binders, the stiffness and m-value were obtained using the BBR. The thermal cracking potential is then determined with the binders critical temperatures and evaluated according to the analysis exposed in Section 2.4.2.

3.2.5. Additive dosage and binders blend design

In this part of the study, the RA binder is rejuvenated using an additive (polyol ester oil) specially made for the asphalt industry. The RA binder is rejuvenated by adding two different percentages of additive to define the optimum dose. The blending protocol is based on the Standard ASTM D4887 (2016) where this practice covers the procedure for preparation of hot recycled bituminous blends for testing in the laboratory and involves an iterative trial blend process followed by the preparation of batch blends. According to the standard, the batch blends can be used for extensive evaluation such as viscosity, penetration, ductility, ageing properties (such as RTFOT), composition analysis, solubility analysis, and other user-selected tests. In this study, the optimum rejuvenator dosage is defined using the penetration and the softening point laws as well as matching the specifications regarding Fraass breaking point and Penetration Index. Therefore, the investigation includes the following tests (encompassing other variables):

- Fundamental properties: penetration, softening point, Fraass breaking point and rotational viscosity test;
- Chemical properties: determination of asphaltenes (BS 2000-143, 2004);
- Rheological properties: DSR and BBR tests.

The target reference binder has been defined as a 50/70 penetration grade bitumen due to the widespread use of this type of binder worldwide. According to the specification (EN 12591, 2009), the bitumen has to respect some specifications and, in this sense, the requirements for a 50/70 penetration grade bitumen are illustrated in Table 5:

Table 5: Binder specification

Property	**Binder 50-70**
Penetration at 25°C (dmm)	50-70
Softening Point (°C)	46-54
Penetration Index	-1.5 to +0.7
Fraass Breaking Point (°C)	≤ -8

Thus, according to the Table 5, the rejuvenated binder should respect these requirements to guarantee a comparison with the target bitumen. The optimal rejuvenator dose is defined as the dose needed to reach the defined target for the property under study, and in this section, the methodology used to define it is exposed below:

- The RA binder is extracted and recovered according to the European standard EN 12697-4 (2015);

- The RA binder is heated up in an oven at a temperature no higher than the softening point temperature + 80 °C for 30 minutes;
- The binder is mixed for homogenisation and to remove bubbles;
- Placement of the RA binder on a container at a temperature between 100°C and 135°C;
- The desired amount of additive is added to the hot plate with RA binder for further mixing of 30 seconds using a glass rod;
- The container with the target blend sample is replaced in an oven at 135 °C for 10 minutes and stirred for 30 seconds at 5 minutes intervals;
- Finally, the sample is ready to be tested.

Other procedures for the optimum dosage are available in literature such as the PG critical temperatures. However, in Europe, the bitumens are classified by their penetration and softening point characteristics, and because these two tests are faster than other tests to be performed the needle penetration and the ring and ball softening point laws were chosen in this research. Moreover, the apparatus to run the tests is simpler and less expensive than the DSR and BBR rheometers, not requiring any specialised training as well as high possibility to be widely reproduced.

In this sense, two percentages by weight of the additive were added in the recovered binder (100% RA binder) in order to determine the conventional properties. The percentage values of additive were chosen according to manufacturer's guidelines and laboratory experience, those results are detailed in further chapters.

Although three different RA's were studied in the first phase of this study, only one material was used from this stage – the RA1. This is due to the fact that to cover the investigation and to be able to analyse more cases of blended binders and mixtures involving DoBA results, more than one type of material would limit the lab activities in terms of time and execution of the experimental programme.

3.2.5.1. Penetration law

The penetration law is based on the results coming from needle penetration test at 25°C, and at least two outcomes are required: the penetration of the both RA binder and the rejuvenated binder, in this last situation adding two percentages o additive. Once the results are available, a graph is plotted with the logarithm of penetration values on the vertical axis and the percentages of the additive on the horizontal axis to draw the regression line for those points.

Once defined the equation of the straight line, the Equation 42 is applied with the aim of calculating the optimal dose of the additive:

$$Dose(\%) = \frac{(logPEN - q)}{m} \qquad \text{(Equation 42)}$$

Where,

PEN = penetration target (e.g. 50 and 70dmm in the chart);
q = the vertical value interception;
m = the gradient or slope of the straight line.

3.2.5.2. Softening Point law

The method aims to determine the ideal dose to reach a softening point target. As in the penetration law, at least two SP results are required to calculate the right amount of additive: the SP of the RA binder and the SP of the rejuvenated binder. However, it is preferable testing two binders rejuvenated with two different percentages of the additive. The results are then drawn in a graph with the softening point temperatures on the vertical axis and the percentages of the additive on the horizontal axis. Once defined the equation of the straight line, the Equation 43 is applied with the aim of calculating the optimal dose of the additive:

$$Dose(\%) = \frac{(Soft.Point - q)}{m} \qquad \text{(Equation 43)}$$

Where,

Soft.Point = softening point target (e.g. 46 and 54°C in the chart);
q = the vertical value interception;
m = the gradient or slope of the straight line.

The difference between the mathematical equations for penetration and SP laws is the existence of the logarithm in the penetration method. This is because the European methodology, followed to draw the blending charts and to determine the blending laws, presents a difference regarding logarithm between these expressions (Section 2.3.1). It is essential to understand that because the bitumens specification are set for both penetration and softening point, in this sense is possible that the percentage defined by the penetration law does not respect the requirement for the softening point and vice versa. In this situation, it is indispensable to choose the amount of additive which respects all the requirements for the selected target binder according to the specifications.

3.2.5.3. Blend design

Binder blend design is performed following the European and USA recommendations described in Section 2.3.1. The application is regularly considered when mixtures with high percentages of RA are under study. The procedure is usually performed for penetration, softening point, viscosity and PG critical temperature: when the binders are tested, the results are substituted in the European and USA models and the properties of the binder in the final mixture can be predicted.

When the binder testing plan is finished, blending charts and blending laws are tailored. Blending charts are graphs in which the horizontal axis represents the percentage of RA in the asphalt mix from 0% to 100%, while the vertical axis represents the binder property under study. In this sense, the 0% RA is the property of the virgin binder, and the 100% RA is the property of the recycled binder (Figure 35).

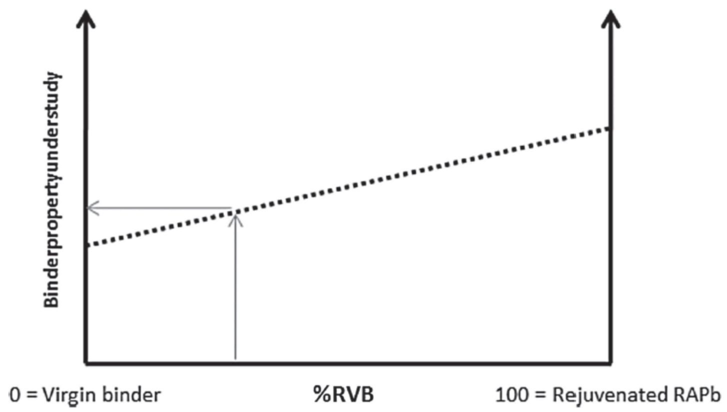


Figure 35: Blending chart example (Jiménez del Barco Carrión et al., 2015)

Jiménez del Barco Carrión *et al.* (2015) introduce a parameter called Replaced Virgin Binder (RVB). The authors mention that a fact to be taken into account is that the %RA to be replaced in the blending laws are the real percentage of virgin binder that must be replaced by the rejuvenated binder. According to those authors, the RVB is influenced by:

- RA content in the final mixture, the total percentage by weight;
- The DOB, the real ratio of blending between the RA binder and the virgin binder. To calculate the RVB, authors considered different percentages of DOB;
- Bitumen content in the RA as well as in the final mixture;
- Additive ratio, the ratio between the amount of additive and the RA binder.

In this research is applied the use of the DoBA due to the processes under investigation (Section 3.1). Therefore, the RVB (%) is calculated applying the Equation (44):

$$RVB(\%) = 100 \times \frac{RA_{in\ the\ mix} \times DoBA \times RA_{bit.content} \times (1 + REJ_{ratio})}{binder\ content\ in\ the\ mixture} \qquad \text{(Equation 44)}$$

Where,

$RA_{in\ the\ mix}$ = is the total RA percentage to add in the mixture by weight, in decimals;
DoBA = the assumed degree of binder activation of the RA, expressed in decimals;
$RA_{bit.content}$ = bitumen content in the RA, expressed in decimals;
REJ_{ratio} = the ratio REJ/$RA_{bit.content}$ for each additive;
Binder content in the mixture = designed final binder content in the mixture.

Applying the Equation 44 and depending on the materials considered, the percentage of the RVB for various mixtures can be determined. The %RVB can be then replaced in the blending laws to evaluate the value of each property in the final mixture. Thus, it can be judged if the mixture is acceptable based on the binder properties.

3.3. Mixtures characterisation and tests

As a wide variety of mixtures is manufactured, it is necessary to have tests to characterise different materials. The mixtures included in this thesis are the same related to the investigated binders: two reference mixtures (control virgin and control RA1) and four recycled mixtures (produced assuming four different DoBAs).

In this sense, the mixtures have their performance-related properties investigated, regarding stiffness modulus, fatigue, rutting and water sensitivity. Unlike what was proposed in the binder studies, thermal cracking resistance of the mixtures had not been investigated due to equipment unavailability.

Therefore, the whole mixtures experimental work is to be used in the RA DoBA Study verification with the recycled binders.

3.3.1. Mixtures plan

The design of an asphalt mixture has until now consisted in choosing, through experimental procedures, a so-called "optimum" binder content, from a predefined granulometric range. In the case of asphalt mixtures, there are numerous aspects to be considered, and the "optimal" content varies according to the evaluation criteria. Therefore, the most convenient is to name the binder content dosed as a design content, as a way of emphasising that its definition is conventional (Bernucci *et al.*, 2008).

The design content of asphalt binder varies according to the dosing method and is a function of parameters such as compaction energy, type of mixture, the temperature at which the pavement will be subjected, among others. The most widely used method worldwide makes use of impact compaction and is called the Marshall method, developed in the 1940s. During the 1980s, several US high-traffic highways showed premature deformations, which were attributed to the excess binder in the blends. This subject was addressed in the US study on asphalt materials, called the Strategic Highway Research Program (SHRP), which resulted in the Superpave. SHRP proposed a methodology that consists in estimating a probable design content through the determination of the air voids and the knowledge of the granulometry of the available aggregates (Asphalt Institute, 1995).

For the development of the asphalt mixtures of this research, it has been found necessary to follow procedures not wholly conventional in the matter of performing the mixtures design. Using the initial results found in the DoBA study as well as the design of the bitumens, the design procedures of the asphalt mixtures had to follow a determinant demand: the amount of RA - 100%. For this reason, further details of the materials are presented in Chapter 4, regarding grading issues and binder content of the RA.

As previously mentioned, the optimum binder content is a crucial factor to obtain a good performance of the asphalt mixtures. Due to the fact that the production of mixtures with 100% RA and the DoBA studies of the RA are the primary objective of this research, it was necessary to maintain the original characteristics of RA.

Therefore, the application of any mixture design, either by the Marshall Method or Superpave, could result in radical changes in the asphalt mixtures and consequently the final bitumen that surrounds the RA aggregates. However, as shown in the previous section regarding the binders, the use of an additive was considered to rejuvenate the properties of RA bitumen. This additive was carefully selected in order to be an agent in which a low percentage was sufficient to modify the final properties of the mixtures, but at the same time would not affect the volumetric properties of the produced specimens.

In the UK, a standard used to predominate in the specification of asphalt mixtures, the BS 4987-1 (2005) Coated macadam (asphalt concrete) for roads and other paved areas — Part 1: Specification for constituent materials and for mixtures. The document contains a range of recipe mixtures for all type of coated macadams to be laid as base, binder or surface course. According to Read and Whiteoak (2003), a recipe specification defines a mixture in terms of target aggregate grading and target binder content, they are simple to use and apply to mixtures if the traffic level is light or moderate.

Nowadays, the standard mentioned above has been already replaced by the European Standard EN 13108-1 (2016), although remains in use due to the simplicity and importance for first guidance on choosing the mixture type. For this reason and the limitations imposed by the primary aims of this research, the BS 4987-1 (2005) and EN 13108-1 (2016) standards were utilised in this study to obtain the mixtures details in terms of grading curves and binder content. In addition, the Brazilian standard DNIT 031 ES (2006) from the National Department of Transport Infrastructure (DNIT) has been also used for mixture design purposes.

Having the information from those standards as well recognising the necessities that should be applied in the research program, the following Table 6 presents three grading curves and bitumen content to be considered according to the RA characteristics (exposed in further chapters).

Table 6: Mixtures design – grading curves and bitumen content targets

Asphalt concrete AC 20			0/20mm dense binder course			Curve DNIT - B (Binder/Surface course)		
Sieve (mm)	% passing		Sieve (mm)	% passing		Sieve (mm)	% passing	
40	98	100	40	100	100	38.1	100	100
31.5	91	100	31.5	100	100	25.4	95	100
20	86	100	20	95	100	19.1	80	100
14	-	-	14	65	85	9.5	45	80
10	53	71	10	52	72	4.8	28	60
6.3	38	56	6.3	38	56	2	20	45
2	22	36	2	20	40	0.42	10	32
0.25	10	20	0.25	6	20	0.18	8	20
0.063	3	9	0.063	2	9	0.075	3	8
Bitumen content:	3-8%		Bitumen content:	4.1-5.3%		Bitumen content:	4.5-7.5%	

3.3.2. Specimen preparation

3.3.2.1. Maximum Density

Density is one of the most critical parameters in the construction of asphalt mixtures. Since the density of an asphalt mixture varies throughout its life, the voids must be low enough initially to prevent permeability of air and water and high enough after a few years of traffic to prevent plastic flow (Brown, 1990).

The maximum density of an asphalt mixture is used in the calculation of void volume, asphalt absorption by the aggregates, the specific effective mass of the aggregate and the effective asphalt content of the mixture (Bernucci *et al.*, 2008). Choosing a binder content above the ideal will reduce the volume of voids, causing the instability of the mixture and the exudation of the binder. The choice of content below the ideal will increase the void volume, making the pavement excessively permeable and accelerating the deterioration process. Therefore, caution is required in determining the maximum density of an asphalt mixture, so that the design content of the chosen asphalt binder eliminates the risk of pathologies, thus ensuring the safety of road users.

In this research, the major investigation is not related to the manufacturing process that includes mixing virgin aggregates and bitumen, but in the effect of the bitumen on recycled mixtures. Therefore, the RA being used as the primary material on this study, the maximum density becomes a fundamental property to be determined from those recycled mixtures to control the manufacturing process.

The EN 12697-5 (2009) Standard specifies the test methods for determining the maximum density of a bituminous mixture. The test methods described are intended for use with loose bituminous materials containing paving grade bitumens, modified binders or other bituminous binders used for hot mix asphalt. In this research, the volumetric procedure using a pycnometer was applied.

3.3.2.2. Gyratory compaction and compactability

The compactability of asphalt mixture in the laboratory is one crucial factor that influences the mixture design. The formation of a structure for asphalt mixture mainly depends on the compaction methods and the specified levels of compaction. Meanwhile, the way in which how the asphalt mixture is compacted in the field significantly influences the compaction degree, which is associated with the construction quality of asphalt pavement. Therefore, understanding the compactability of asphalt mixture is needed for both design of asphalt mixture and construction of asphalt pavement (Micaelo, 2008; Hu *et al.*, 2017).

Compactability, which is used for evaluating how easy it is to compact a mixture in the field (Micaelo, 2008; Leiva and West, 2008), is one of the aspects representing for the workability of the asphalt mixture. As gyratory compaction method is widely accepted for mixture design, and due to the high productivity of test specimens, the method was selected for the compactability investigation and specimens manufacturing for further performance-related tests. The European standard EN 12697-31 (2007) is followed for the specimen preparation using the gyratory compactor.

The EN 12697-10 (2002) standard, which designates the methods to be used in measuring the compactability of bituminous mixtures, defines compactability as "the ratio of the density or void content to the applied compaction energy". The standard specifies the measurement of compactability based on the monitoring of compaction, through the density, porosity or thickness of the test specimen, with the compaction energy applied by one of three laboratory compaction methods (gyrations, impact or vibration).

In the scope of the Superpave method, compaction limits are defined at three moments of compaction of the test specimen with the gyratory compactor. According to the traffic and temperature classes to which the pavement is predictably subjected, the N_{ini} (initial), N_{des} (desired) and N_{max} gyration numbers, respectively, in ascending order, are defined. For a number of gyrations equal to N_{ini}, the degree of compaction (a function of the theoretical maximum density) must be less than 89%, for N_{des} equal to 96% and Nmax less than 98%. With the limits of N_{des} and N_{max}, the desired air voids value for the bituminous mixture in service is specified, between 4% (after construction) and 2% (after traffic compression). The degree of compaction presents an almost linear variation as a function of Log N. Therefore, the Superpave method defines N_{ini} and N_{max} according to N_{des}, according to Equations 45 and 46:

$$LogN_{ini} = 0.45 \times LogN_{des} \qquad \text{(Equation 45)}$$

$$LogN_{max} = 1.10 \times LogN_{des} \qquad \text{(Equation 46)}$$

According to the EN 12697-10 (2002) standard the compactability of a bituminous mixture, measured with the gyratory compactor, is determined by the logarithmic regression adjustment (Equation 47), where k represents the compactability. Considering the limits of the degree of compaction equal to 89% and 96% for N_{ini} and N_{des} respectively, the minimum value of the compactness k is equal to 0.01.

The compactability (k) has been investigated on those mixtures selected according to the primary DoBA study and binders design.

$$v\left(N_g\right) = v(1) - (k \times lnN_g) \qquad \text{(Equation 47)}$$

Where,

N_g = the void content for a number of gyration N_g, expressed in percent (%);
$\nu(1)$ = the calculated void content for one gyration;
k = the compactibility (method using a gyratory compactor);
N_g = the number of gyrations.

3.3.3. Performance-related properties

3.3.3.1. Modulus: Indirect Tensile Stiffness (IT-CY)

In order to characterise the stiffness of the six mixtures, the IT-CY test was selected, the test details were presented in Section 2.4.3 and is in accordance with the European standard EN 12697-26 (2012). The temperature frequently used to conduct the IT-CY test is 20°C, and was selected for the tests. Furthermore, 3 temperatures were also tested in order to observe the stiffness variation between the mixtures (0°C, 10°C and 30°C) in this research. The cylindrical specimens used for the test measured 50mm thickness x 100mm diameter and all test specimens were subjected to 24 hours conditioning at the test temperature before testing. Five specimens of each mixture were tested, the average values are reported.

3.3.3.2. Rutting: Repeated Load Axial Test (RLAT)

In order to characterise the rutting resistance of the six mixtures, the RLAT test was selected, the test details were presented in Section 2.4.3 and is in accordance with the British Standard BS DD 226 (1996). The cylindrical specimens used for the test measured 50mm thickness x 100mm diameter and all test specimens were subjected to 24 hours conditioning at the test temperature before testing, and three specimens of each mixture were tested, the average values are reported. The following test parameters were applied in the RLAT testing:

- Test temperature: 50°C;
- Test duration: 7200 seconds (3600 cycles);
- Axial stress: 100 kPa;
- Conditioning stress: 10 kPa for 600 seconds;

3.3.3.3. Fatigue: Indirect Tensile Fatigue Test (ITFT)

In order to characterise the fatigue resistance of the six mixtures, the ITFT test was selected, the test details were presented in Section 2.4.3 and is in accordance with the British Standard BS DD ABF (2003). The cylindrical specimens used for the test measured 50mm thickness x 100mm diameter and all test specimens were subjected to 24 hours conditioning at the test temperature (20°C) before testing. The fatigue performance of the asphalt mixtures is evaluated by using the following two fatigue parameters based on the fatigue laws obtained for the mixtures (Read and Collop, 1997; Airey, 2004):

- Strain (microstrain): 1,000,000 cycles;
- Fatigue cycles: 100 microstrains.

3.3.3.4. Water sensitivity: Indirect Tensile Stiffness Ratio (IT-CY$_{Ratio}$)

The sensitivity to water of the recycled and control mixtures was evaluated based on the Interim Guideline Document For The Assessment And Certification Of Thin Surfacing Systems For Highways (BBA, 2013), determining the ratio of conditioned to unconditioned indirect tensile stiffness modulus (IT-CY) value measured using the NAT. The following procedure has been developed specifically for the assessment of thin surfacing systems by the Highways Agency Product Approval Scheme (HAPAS) to protect against water damage. The cylindrical specimens used for the test measured 50mm thickness x 100mm diameter, three specimens of each mixture were tested, the average values are reported

The testing procedure involves measuring the non-destructive IT-CY in the dry condition, designated as IT-CY_U, and subsequently the same samples having undergone a water immersion regime as follows:

1. The specimens are tested for IT-CY in a dry condition, after at least 4h conditioning at 20°C;

2. The specimens are placed in the vacuum desiccators and covered with distilled water at a temperature of 20±1°C, the apparatus is sealed, and a partial vacuum of 510±25 mm Hg for 30±1 minutes is applied;

3. The specimens are removed and placed in a hot water bath at a temperature of 60±1°C for 6±1 hours;

4. The specimens are removed from the hot water bath and immediately placed in a cold water bath at a temperature of 5±1°C for 16±1 hours;

5. The specimens are removed from the cold water bath and directly placed in a water bath at a temperature of 20±0.5°C for 2 hours;

6. The specimens are removed from the water bath, surface dried and the conditioned stiffness determined at a test temperature of 20±0.5°C for the first conditioning cycle and designated as IT-CY_{C1}.

7. The procedure given in clauses 3 to 6 is repeated and the conditioned stiffness of the specimen determined for the IT-CY_{C2} and IT-CY_{C3}.

The stiffness ratio is calculated for the specimens for each conditioning cycle as:

$$ITCY_{Ratio,Ci} = \frac{ITCY_{Ci}}{ITCY_U} \quad \text{(Equation 48)}$$

Where,

Ci = conditioning cycle i=1, 2, 3;
$ITCY_{Ci}$ = conditioned stiffness after conditioning cycle ci, and;
$ITCY_U$ = unconditioned stiffness modulus.

3.4. Summary and conclusions

The methodology of this thesis is divided into different steps/tasks related together as shown in Figure 36. As it can be seen in Figure 36, there are three different levels of materials evaluation: DoBA Study, binders and asphalt mixtures.

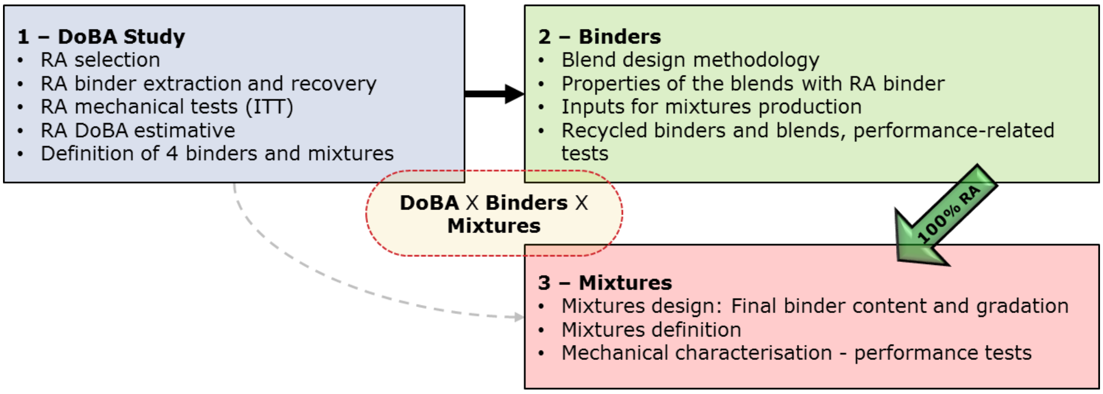


Figure 36: Summarised methodology

These three levels are connected through the development of the blend design of binders and asphalt mixtures by using the results from the DoBA study. In this regard, the first step of the methodology is the characterisation of the RA materials and perform the DoBA Study. Next, this characterisation and DoBA results are used to perform the blend design with the RA binder and define the final mixtures. The results of the blend design are used as inputs for the asphalt mixtures. Finally, binders and mixtures are produced and tested for the subsequent correlation of the three levels (DoBA x binders x mixtures). Figure 37 presents the set of tests performed in all the selected binders and mixtures, regarding conventional and performance-related properties.

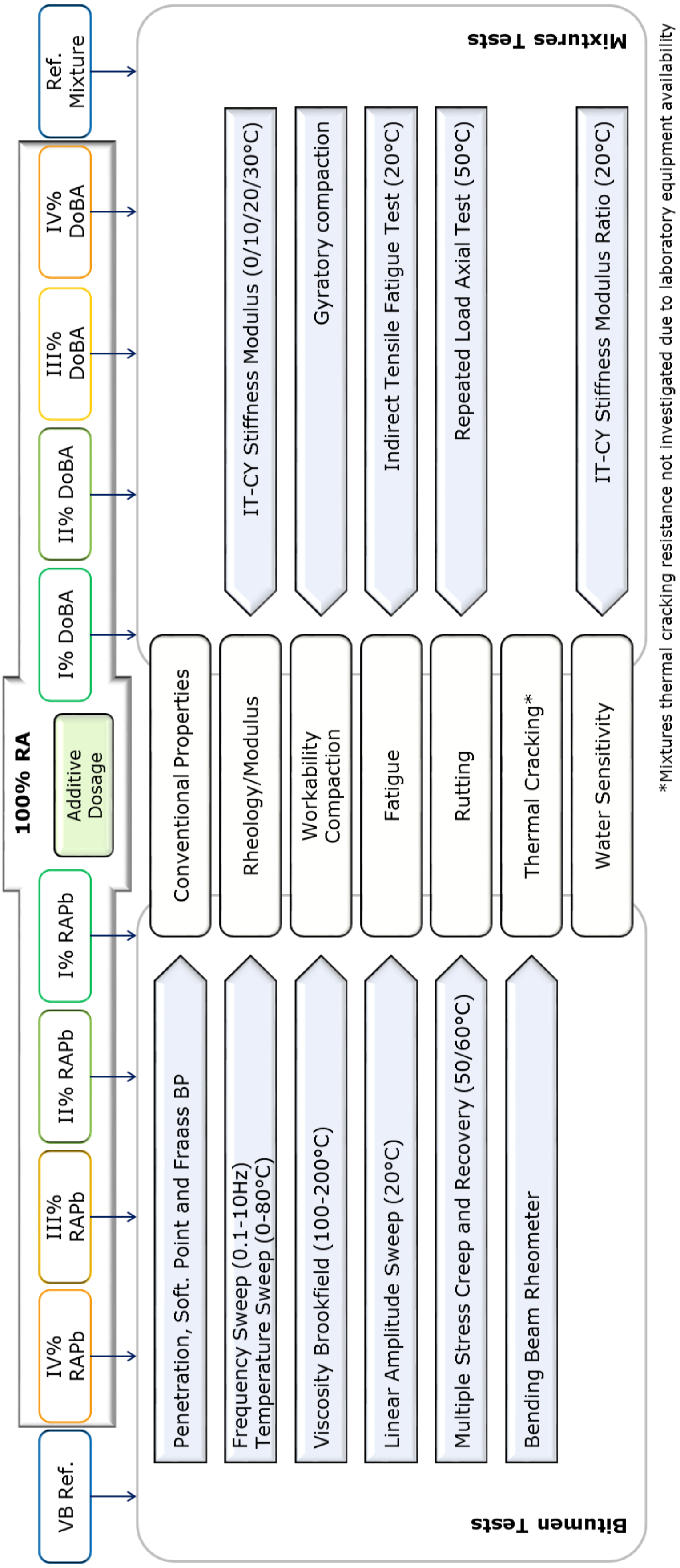


Figure 37: Laboratory experimental programme

4. DEGREE OF BINDER ACTIVATION

4.1. Introduction

Once the research methodology and the experimental programme were defined, the DoBA investigation could be carried out. This chapter shows the process and results found regarding the DoBA for different sources of RA. The objectives are to provide a characterisation of the selected RA's and virgin materials and, create two RA binders in the lab to produce artificial RA mixtures. Next, the DoBA investigation is presented with the procedures adopted during the samples manufacturing along with the influence of the different mixing times and temperatures. Following this, the DoBA investigation is presented with the Indirect Tensile Test data analysed by using different parameters (ITS, Total Energy, Pre-peak Energy, Post-peak Energy and Flexibility Index). These parameters were obtained to assess the DoBA, according to the different production temperatures, for the RA-DoBA labelling proposals. Furthermore, the use of an additive was also investigated in order to determine its effects in the RA DoBA and also to apply in further binders and mixtures designs (Chapters 5 and 6).

4.2. Materials

Any project requires for its viability, a vast knowledge of the characteristics, properties and behaviour of the materials to be used. It should be characterised with the operating conditions to which this material is submitted to obtain the properties required for such application and also understand how to determine these values and what limitations and restrictions exist on their usage. In this sense, a wide range of laboratory tests were performed to characterise the materials:

- Reclaimed Asphalt: three sources of RA were selected with different original characteristics. Two materials were considered with original straight bitumen in its composition, differing in their penetrations with RA1 < 20 dmm < RA2. RA3 was considered to have a modified bitumen in its original composition.

- Virgin bitumen: two virgin bitumens were selected for the DoBA study and the ageing procedure. One neat bitumen with penetration 50/70 and another modified PMB 60/85. The VB 50/70 was also used to produce the binder's blends and further asphalt mixtures, shown in Chapters 5 and 6, respectively.

- Virgin aggregates: two sources of virgin aggregates were used according to the RA original aggregates characteristics.

4.2.1. RA selection

In order to obtain the RA properties and characteristics, a wide range of tests were performed according to the following Figure 38:

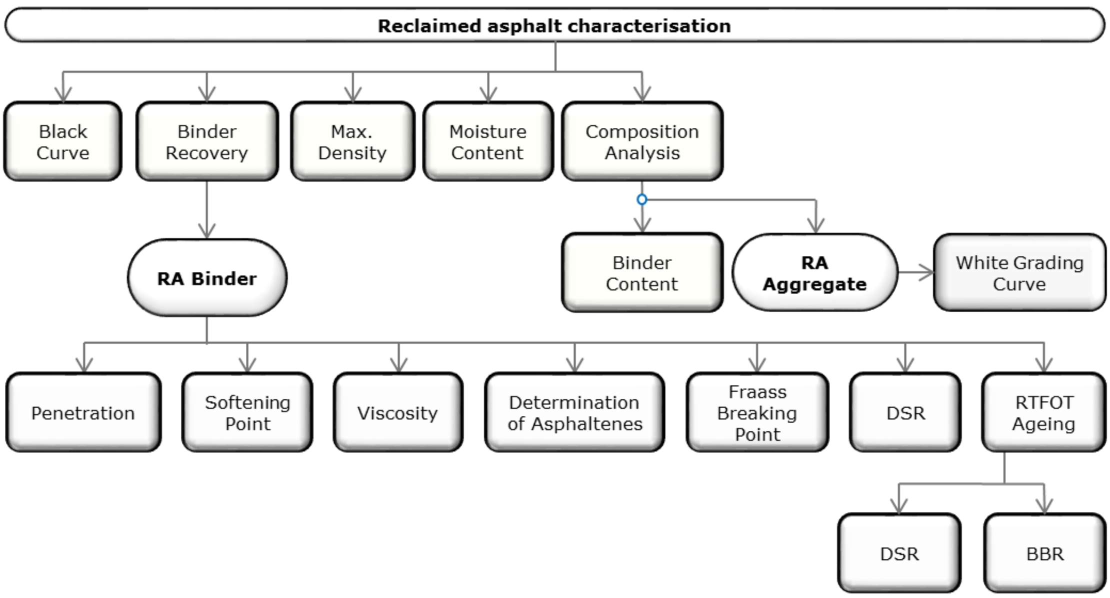


Figure 38: RA characterisation flowchart

4.2.1.1. Grading Curves and Composition Analysis

The grading curves of the RA's were determined using the procedures detailed in the European standard (EN 12697-2, 2015) in terms of, firstly, the grading of the original material (known as a Black Curve) and, secondly, on the recovered aggregate after binder extraction (known as a White Curve). Figure 39 presents the grading curves of the three RA samples (Black and White curves). The grading envelope for a standard Asphalt Concrete 20 mm asphalt mixture (AC 20), 0/20mm dense binder (BS 4987-1, 2005) and the Curve DNIT – B (DNIT 031 ES, 2006) are included in Figure 40 together with the RA White curves.

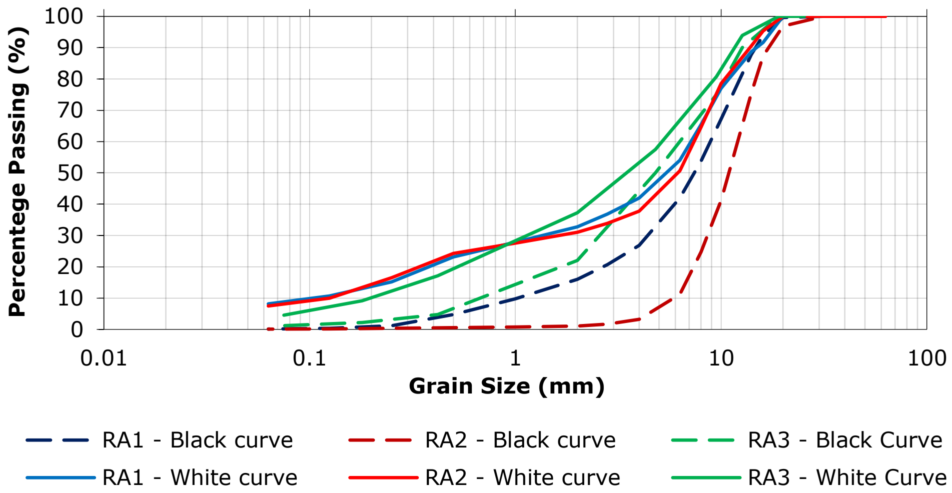


Figure 39: RA grading curves

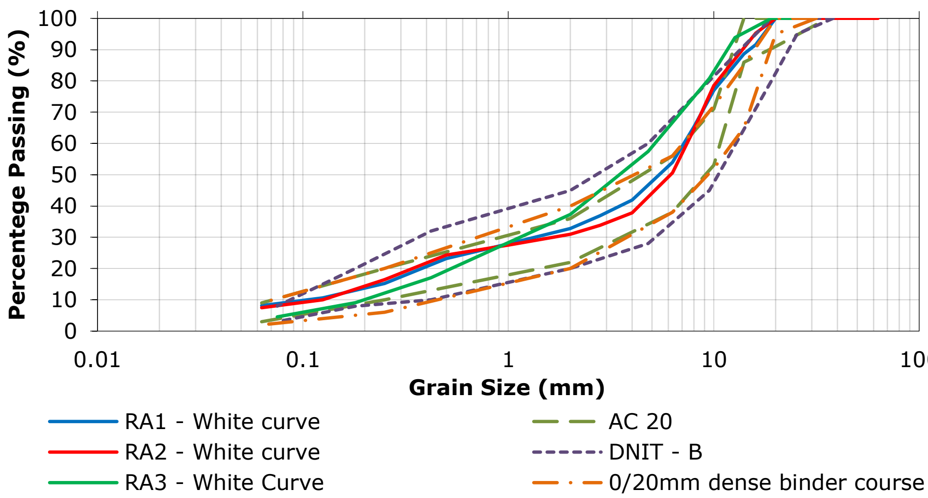


Figure 40: RA white curves and agencies designed curves

The particle size distribution confirms the most common finding in RA grading, the low amount of fine material, a characteristic that occurs due to the presence of the bitumen in the finer particles of the material and the subsequent formation of lumps. However, after the bitumen extraction, the white curve shows a significant amount of fines in the materials, with about 30% of the particles equal or smaller than 2mm. Surprisingly, RA1 and RA2 presented very similar white curves, meaning that the same original curve may have been used but probably handled differently after the milling process of the RA (e.g. processing, crushing and fractionating).

In addition to the grading curves, the grading limits for the asphalt mixture AC 20, 0/20mm dense binder and the Curve DNIT – B are also shown as the RA1 and RA2 white curves fitted almost entirely inside of the boundaries (AC 20 and 0/20mm dense binder) and its therefore possible that the design curve was initially adopted. RA3, originally from Brazil, is fitted almost entirely inside of the limits of the DNIT – B curve. In this sense, it is now critical to understand the properties of the binder from each source of RA.

4.2.1.2. RA composition analysis, moisture content and maximum density

The results of the composition analysis that provided the RA white grading curves have also provided information about the original binder content of the mixtures. Moreover, other essential characteristics for a better understanding of the RA's were determined and presented in Table 7 together with the binder content and the respective standards followed for each procedure.

Table 7: Binder content, moisture content and maximum density

Property	RA1	RA2	RA3	Standard
Binder Content (%)	4.4	4.7	4.41	(EN 12697-1, 2012)
Moisture Content (%)	0.14	0.10	0.19	(EN 1097-5, 2008)
Maximum Density (g/cm^3)	2.48	2.42	2.55	(EN 12697-5, 2009)

The results in Table 7 are essential to understand the material in their future applications, where the grading curves and binder content from the original mixture are important. For RA1 and RA2, the bitumen content found is relatively similar as well as their original grading curves presented in Figure 40. Moreover, the bitumen contents found in these two sources is between the limits suggested by the standards in Table 6 (3-8% for AC 20 and 4.1-5.3% for 0/20mm dense binder). Regarding the binder content of RA3, 4.41% is outside the boundaries of the DNIT – B curves 4.5-7.5%, but still close to the minimum specified, suggesting again to be the original curve adopted by the constructor.

Considering the moisture content, its determination is valuable when recycling these type of materials and necessary when choosing the application method (cold, warm or hot). If this property is not considered, the recycling process can be compromised affecting the performance of the new material. The results show only residual moisture in the three materials. However, the procedures of this investigation include the drying of the RA's before their use to avoid residual moisture content affecting the results.

Another property that it is not only for characterisation but is also crucial for understanding the origin of the materials is the maximum density. Particularly in the methodology of this research where the major investigation is not related to the manufacturing process, that includes mixing virgin aggregates and bitumen, but to the effect of the bitumen on mixtures containing 100% RA. As presented in the previous chapter, the maximum density of an asphalt mixture is used in the calculation of the voids volume, bitumen absorption by the aggregates, the specific effective mass of the aggregate and the effective bitumen content. Therefore, the maximum density of the materials is a crucial value for the manufacturing process of the DoBA Study, becoming the first guideline for the production and thereby controlling the specimens compaction and air voids. All the three RA's can be considered to have close values of maximum density, although this does not mean that the original virgin aggregates were similar, it may indicate that they are also not entirely different as they do not exhibit a considerable difference (e.g. 2.1

g/cm^3 and 2.7 g/cm^3). Further investigation of the RA aggregates, washed from bitumen, was performed and the results are presented in the following sections.

4.2.2. Binders

This section shows the characterisation of the virgin binders (VB 50/70 and PMB 60/85) and comparison with the RA binders. In order to study conventional and rheological properties of bitumen through a wide range of temperatures and loading times, the following tests have been undertaken:

- Needle penetration at 25°C (EN 1426, 2015);
- Ring and ball softening point (EN 1427, 2015);
- *Fraass breaking point determination (EN 12593, 2015);
- *Determination of asphaltenes (BS 2000-143, 2004);
- Rotational viscosity determination from 100 to 200°C (EN 13302, 2010);
- DSR - Frequency sweep from 0.1 to 10 Hz (EN 14770, 2012);

*Tests performed only on RA1b, RA2b and VB 50/70 due to the availability of the test.

4.2.2.1. Conventional test results

Conventional test results are shown in Table 8, the asphaltenes content is also included when available.

Table 8: Binders properties

	RA 1	RA 2	RA 3	VB 50/70	PMB 60/85
Penetration (dmm)	16.3	24.6	22.0	64	61
Softening Point (°C)	64.9	61.1	66.5	47.6	60
Penetration Index	0.37	-0.31	0.43	-1.07	1.57
Fraass Breaking Point (°C)	-3	-7	-	-8	-
Asphaltenes content (%)	21.3	20.8	-	15.76	-

Given the results, it is possible to see that the recovered binders RA1 and RA2 are stiffer than the VB 50/70 due to the lower penetration and higher SP. The penetration index shows that both materials are in the typical range for paving-grade bitumens (-1.5 to +0.7) according to the European Standard EN 12591 (2009), but the RA binders may be less susceptible to variations in temperature, as its value is closer to zero. Fraass breaking point results imply that the RA binders are slightly more brittle than VB 50/70 at low temperatures. Lastly, the asphaltenes content shows the expected higher content in the oxidised bitumens and confirms what was presented in the literature review: "The asphaltenes are the heavy particles of bitumen (high molecular weight), therefore, the higher the asphaltenes content, the lower the penetration and the higher the softening point and viscosity" (Airey, 1997).

Regarding the results from RA3 binder and the virgin binder PMB 60/85, the same situation for penetration (lower value for the RA binder) and softening point (higher for the RA binder) were found. It is important to highlight that the virgin binder PMB 60/85 used in this investigation is the same material used initially in the RA3 mixture. The penetration index value found for the virgin binder is a typical value for a PMB and, according to Airey (2004) indicates the improved (low) temperature susceptibility resulting in increased flexibility at lower temperatures and increased stiffness at high temperatures.

4.2.2.2. Viscosity

Viscosity measurements have been undertaken at temperatures ranging from 100 to 200^{0}C using a Brookfield Viscometer following the European Standard EN 13302 (2010). Figure 41 displays these results for all five bitumens with the respective regression power line from each material. VB 50/70 presented the lowest viscosity; however, all other bitumens met the Superpave binder specifications where the maximum value of 3 Pa.s should be respected at 135°C (AASHTO MP-1, 1998), and, from this temperature, the three recovered binders begin to present their differences.

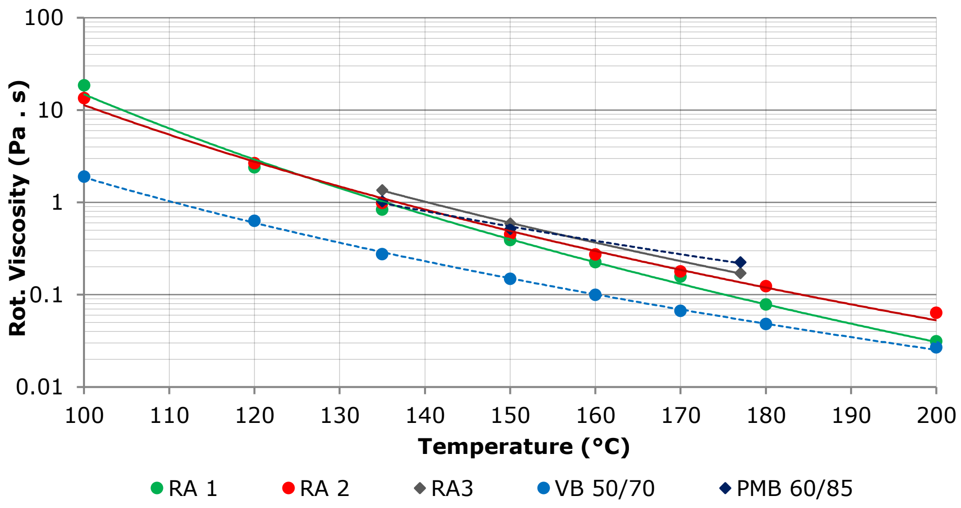


Figure 41: Viscosity - RA's and virgin binders

The results show the differences between neat and modified bitumens, aged or not, where the modified binder is providing higher viscosity even in its virgin form. Moreover, comparing RA3 and PMB 60/85, the significant variance to notice is the slope of the curves, which can be observed at 155°C where virgin and old bitumen are crossing the regression lines. Therefore, for the ageing bitumen procedure, only a change in the slope should be attempted, and not just on ageing or oxidation like in the other case for VB 50/70 and the RA1 and RA2 binders.

4.2.2.3. Rheological properties – DSR and BBR

The bitumens have been tested using the DSR for Frequency and temperature sweep tests from 0 to 80°C at frequencies from 0.1 to 10 Hz, according to Section 3.2.3.1. Figure 42 shows the results in the form of master curves for complex modulus and phase angle at a reference temperature of 25°C. Master curves have been produced through manual adjustment of shift factors.

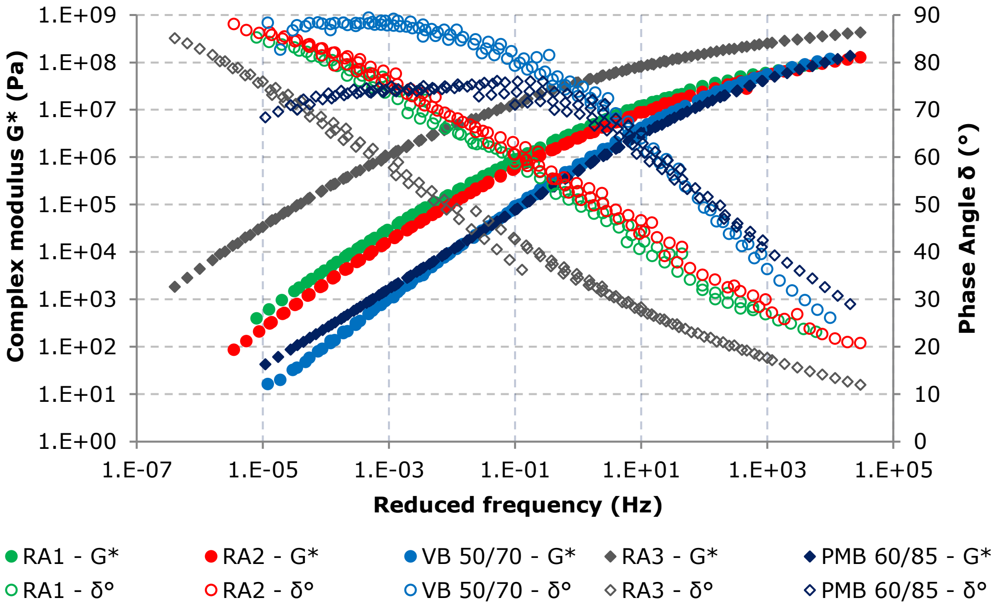


Figure 42: Complex modulus and phase angle master curves

The binder RA3 presents the highest complex modulus, followed by RA1 and RA2. Considering RA1, RA2 and the VB 50/70, the three bitumens present similar behaviour at lower temperatures, slightly higher stiffness for the RA1. These results could be expected in view of the conventional test results. The differences between RA1 and RA2 are even less noticeable looking at the phase angle, with values slightly lower for RA1. In summary, supporting the conventional properties (penetration and softening point), the rheological properties also show the RA1 being slightly stiffer than the RA2. Phase angle master curves clarify the differences in the elastic and viscous behaviours. For all reduced frequencies, the VB 50/70 presents higher phase angles which represents more viscous and therefore less elastic behaviour. For the DoBA Study and the ageing procedure to produce an RA binder, the VB 50/70 selected can be considered similar to the aged RA1 and RA2 binders by analysing the shape and slope of the curves under comparison. Therefore, the VB 50/70 bitumen was used in the next tasks of the investigation.

Regarding the RA3 and the PMB 60/85, there is a big difference between the materials regarding complex modulus and phase angle. Although the origin of the bitumens is the same (RA3 binder is originally the PMB 60/85), the degree of oxidation that RA3 presents is quite high. One hypothesis for the reason for this is that during the handling of the RA3 binder, it may have been overheated in the oven during the preparation of the samples for testing in the DSR. Unfortunately, this result could not be repeated due to the amount of binder sent to the laboratory that performed the test. Despite this, the selected material was continuously used in the research simply by knowing the origin of the binders under analysis.

4.2.3. Virgin aggregates

Firstly, to understand and recognise the type of the RA original aggregates, the composition analysis carried out to obtain the white grading curve also provided washed aggregates for the visual assessment. In this sense, the following analysis is presented considering each source of RA:

RA1: the clean aggregate presented an appearance similar to a type of granite with predominantly grey and pink colours. Therefore a crushed granite from Bardon Hill/UK available in the lab was selected. In order to investigate the material, the absorption of the virgin aggregates is essential to reproduce a similar mixture to the RA. In this sense, the water absorption was determined according to the European Standard EN 1097-6 (2013) in both virgin and RA1 aggregates (after total binder extraction). A range of 3 aggregate sizes (#12.5, #8 and #4mm) were sieved and separated for the analysis, showing results equal to 0.4% (#12.5mm), 0.6% (#8mm) and 0.8% in both materials, meaning that the chosen virgin aggregate can absorb the same amount of binder as the original RA1.

RA2: the aggregates found after extraction of the bitumen had a mineral origin that was not available in the laboratory to reproduce the mixtures. The extracted material seemed to have more than one source of stone (granite and basalt) and was difficult to separate and estimate the amounts of each available in the original mixture. Because of this, problems of absorption of the binder could be found. RA2 was therefore subjected to additional binder extraction to recover the aggregates for its reuse in the study. Therefore, the aggregates from RA2 were entirely washed from bitumen using dichloromethane (DCM - CH_2Cl_2) as a solvent, washed with water and finally dried in an oven for 24h at 105°C.

RA3: the virgin aggregates were obtained through the crushing process of basaltic rock, collected directly from the quarry located in Santo Antonio da Patrulha/RS, Brazil. In this case, the materials from RA3 (virgin binder and aggregates) were available to use in the University of Santa Maria because of the original mixture design had been performed by the university together with the constructor.

4.3. Artificially aged binders

The creation of the artificial RA binder was carried out using the High-Shear Mixer (Section 3.1.2). Every 6 hours of ageing a sample of the bitumen was taken in order to measure the properties using Penetration, SP, Viscosity and DSR Frequency & Temperature Sweeps. The first analysis was conducted based on the results of Penetration at 25°C and SP, the dashed lines representing the targeted values from RA1 and RA2 in Figure 43, and RA3 in Figure 44:

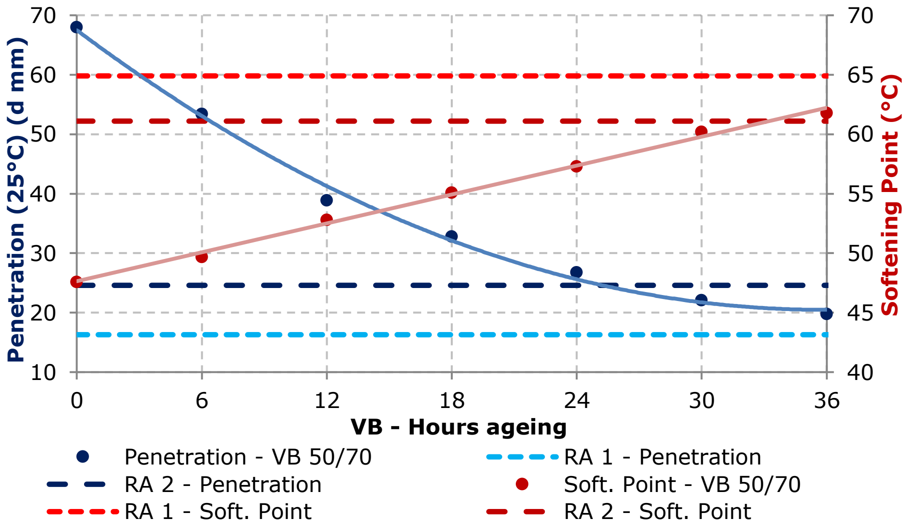


Figure 43: VB 50/70 ageing procedure – Penetration and SP

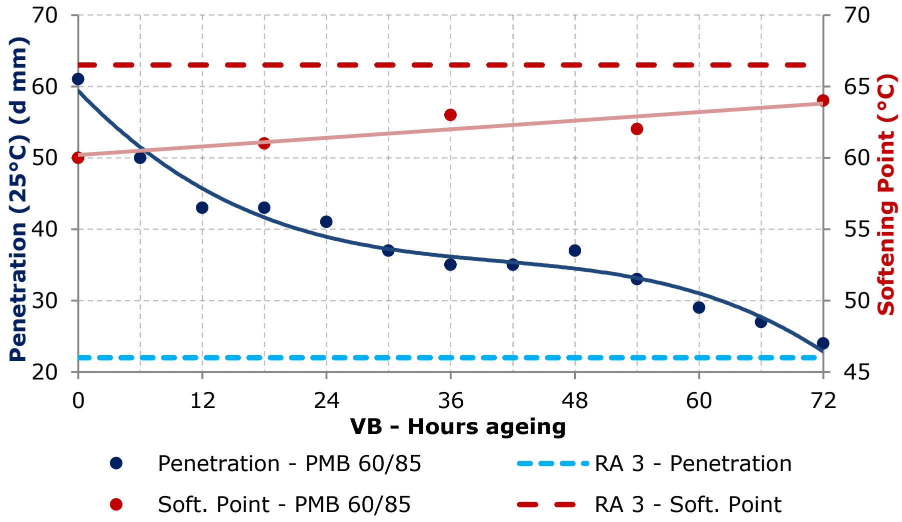


Figure 44: PMB 60/85 ageing procedure – Penetration and SP

As can be seen, the test was finished after 36 hours of ageing the VB 50/70. The dashed lines represent the targeted values coming from the RA binder results. Penetration of virgin binder is 68dmm which after 36 hours decreased to 19.8dmm, a slightly higher than the RA1 (16.3dmm) and lower than RA2 (24.6dmm). The ring and ball test determined 61.8°C for SP after 36 hours of ageing, lower than the RA1 (64.9°C) and relatively the same as RA2 (61.1°C).

The ageing procedure of the PMB required extended time to reach similar properties to the RA3 binder. Although the procedure was more time consuming, the results obtained were promising, the original penetration of 61dmm was reduced to 24dmm, close to the value of RA3 (22dmm). A similar analysis can be made for the SP, from 60°C (unaged PMB 60/85) reaching 64°C after 72 hours of ageing, near to the 66°C found in the RA3 binder. In summary, the ageing procedure adopted was not affected by the type of bitumen, regarding penetration and SP, where the good relationship of the results was found. However, ageing a PMB is more time consuming, and other properties could provide extra support for these results.

The next results presented are related to the viscosity measurements. Figure 45 shows the viscosity of the VB 50/70 during the ageing procedure together with the regression lines for all aged bitumens (VB HSM XXh).

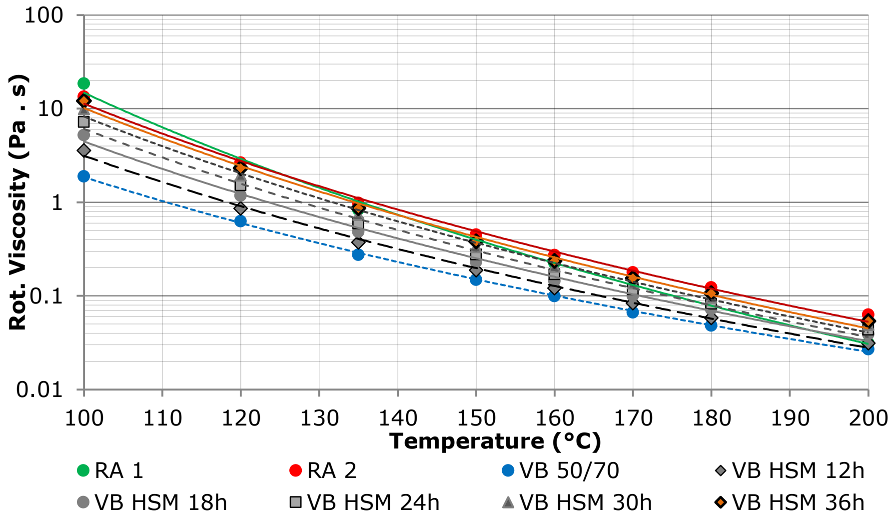


Figure 45: VB 50/70 ageing procedure – Viscosity

From the test results, it can be seen that the viscosity increased every 6 hours for each temperature, less evident at 200°C but distinguishable at other temperatures. From these results, the regression lines of RA1 and VB HSM 36h cross each other at the temperature around 137°C, but the measurements showed values almost equal at temperatures of 120, 135, 150, 160 and 170°C, meaning that in this range of temperatures the viscosity can be considered the same. Comparing with the RA2 results, the recovered binder is always slightly more viscous but still with similar properties. Therefore, the viscosity characteristic was achieved after 36 hours of ageing, allowing working at temperatures ranging from 120 to 170°C.

Next, Figure 46 shows the viscosity results of the PMB 60/85 during the ageing procedure together with aged bitumens (PMB HSM XXh). As already mentioned, comparing RA3 and PMB 60/85, the significant variance to notice is the slope of the curves, which can be observed at 150°C where virgin and RA bitumen cross their regression lines. Therefore, for the ageing bitumen procedure a change in the slope should be attempted. In this sense, the results show the changing in the slope of the lab aged bitumens instead of a shift in the lines towards more viscous materials (as in the VB 50/70 ageing). After 72h of ageing, the measured values were accepted together with the Penetration and SP outcomes.

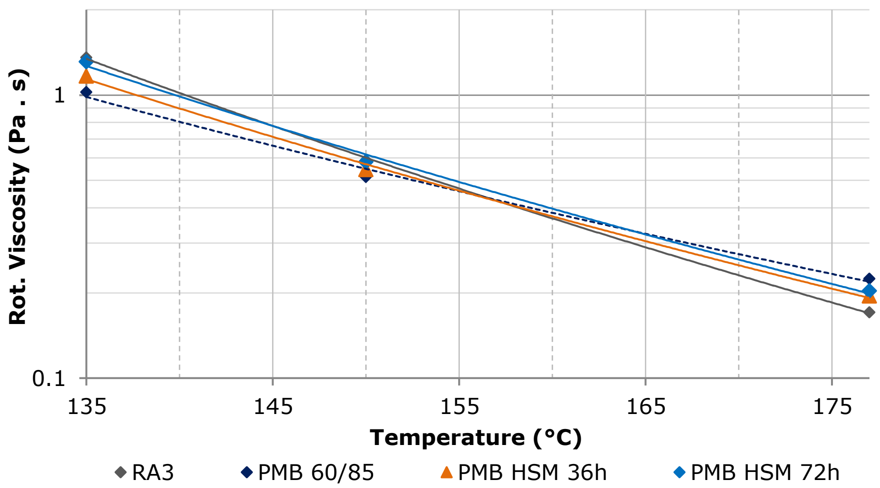


Figure 46: PMB 60/85 ageing procedure – Viscosity

With all the results collected from binders, Table 9 and Table 10 were tailored for the VB 50/70 and the lab aged bitumens:

Table 9: Binder's VB 50/70 ageing characterisation

Binder / Test	VB 50/70	VB HSM - Ageing					
		6h	12h	18h	24h	30h	36h
Penetration 25°C (dmm)	64	53.4	38.9	32.8	26.8	22.1	19.8
Softening Point (°C)	47.6	49.7	52.8	55.1	57.3	60.2	61.8
Penetration Index	-1.07	-1.13	-1.08	-0.92	-0.87	-0.67	-0.58
Fraass Breaking P. (°C)	-8	-	-	-	-	-	+2
Rot. viscosity (Pa.s)							
@100°C	1.89	2.74	3.58	5.23	7.21	9.89	12.17
@120°C	0.63	0.70	0.86	1.18	1.52	1.94	2.33
@135°C	0.28	0.31	0.37	0.49	0.60	0.74	0.87
@150°C	0.15	0.15	0.19	0.23	0.28	0.33	0.38
@160°C	0.10	0.01	0.12	0.14	0.17	0.20	0.24
@170°C	0.07	0.07	0.08	0.10	0.12	0.14	0.15
@180°C	0.05	0.05	0.06	0.07	0.08	0.10	0.11
@200°C	0.03	0.03	0.03	0.04	0.04	0.05	0.05
Log(G*@0.4Hz-25°C)	5.492	5.612	5.875	6.032	6.177	6.310	6.388
Asphaltenes Content (%)	15.76	-	-	-	-	-	19.67

Table 10: Binder's VB 50/70 – RA and ageing comparison

Test \ Binder	VB 50/70	VB HSM 36h	RA 1		RA 2	
				Relative Differ.		Relative Differ.
Penetration 25°C (dmm)	64	19.8	16.3	+21%	24.6	-19.5%
Softening Point (°C)	47.6	61.8	64.9	-4.8%	61.1	+1.1%
Penetration Index	-1.07	-0.58	0.37	-	-0.31	-
Fraass Breaking P. (°C)	-8	+2	-3	+5°C	-7	+9°C
Rot. Visc. @120°C (Pa.s)	0.630	2.328	2.394	-2.8%	2.644	-11.9%
@135°C (Pa.s)	0.275	0.868	0.836	3.8%	0.984	-11.8%
@150°C (Pa.s)	0.149	0.382	0.391	-2.3%	0.450	-15.1%
@170°C (Pa.s)	0.066	0.154	0.157	-2%	0.177	-13%
Log(G*@0.4Hz-25°C)	5.492	6.388	6.320	+1.1%	6.152	+3.8%
Asphaltenes Content (%)	15.76	19.67	21.3	-7.7%	20.8	-5.4%

Tables Table 11 and Table 12 were tailored for the PMB 60/85 and the lab aged bitumen:

Table 11: Binder's PMB 60/85 ageing characterisation

Test \ Binder	PMB 60/85	PMB HSM - Ageing						
		12h	18h	36h	48h	54h	60h	72h
Penetration 25°C (dmm)	61	43	43	35	37	33	29	24
Softening Point (°C)	60	-	61	63	-	62	-	64
Penetration Index	1.57	-	0.85	0.76	-	0.44	-	0.16
Rot. viscosity (Pa.s)								
@135°C	1.03	-	1.13	1.17	-	1.17	-	1.32
@150°C	0.51	-	0.52	0.55	-	0.54	-	0.58
@177°C	0.23	-	0.19	0.20	-	0.19	-	0.20
Log(G*@0.4Hz-25°C)	5.38	-	5.47	-	-	5.54	-	5.55

Table 12: Binder's PMB 60/85 – RA and ageing comparison

Test \ Binder	PMB 60/8	PMB HSM 72h	RA 3	
				Relative Differ.
Penetration 25°C (dmm)	61	24	22	+9.1%
Softening Point (°C)	60	64	66	-3.0%
Penetration Index	1.57	0.16	0.41	-
Rot. Visc. @135°C (Pa.s)	1.03	1.32	1.36	-3.0%
@150°C (Pa.s)	0.51	0.58	0.59	-1.7%
@177°C (Pa.s)	0.23	0.20	0.17	+17%
Log(G*@0.4Hz-25°C)	5.38	5.55	7.42	-25%

The last analysis made was regarding the rheological properties using DSR frequency & temperature sweeps. Figure 47 shows the complex shear modulus and phase angle master curves of all tested VB 50/70 bitumens, developed using 25°C as the reference temperature.

From the master curves can be seen that the bitumen VB HSM 36h shows almost the same behaviour as the RA1 binder, since they are almost overlapped entirely, but is slightly stiffer than RA2 (minimum variation only at high temperatures but still similar for both recovered binders). The same behaviour was noted for the phase angle master curves, for both RA1 and RA2, meaning that the viscous and elastic behaviours are also similar at the analysed temperatures. The measured Log(G*) at 0.4Hz and 25°C is used to compare the results as it can be used to predict penetration value using the correlation proposed by Gershkoff (1991). Those results were presented in Table 9 and Table 10. In addition, the Appendices 3, 4 and 5 present the complex modulus and phase angle raw data for comparison between RA1, RA2 and RA3 binders with their respective artificially aged bitumen.

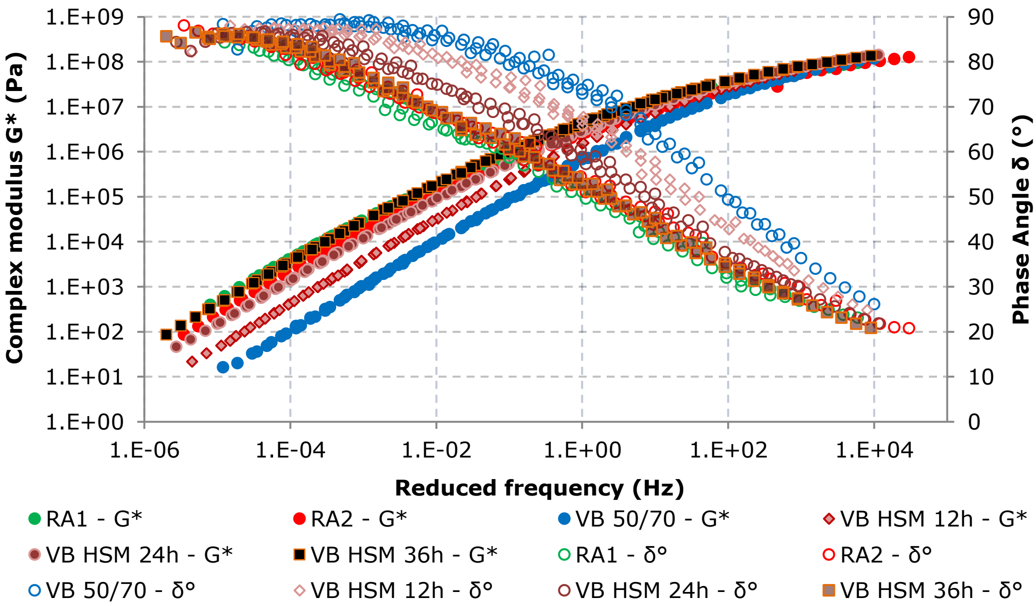


Figure 47: VB 50/70 Ageing procedure – Complex modulus and Phase angle

Figure 48 shows the G* and δ master curves of the PMB 60/85 and RA3 bitumens, also developed with 25°C as the reference temperature.

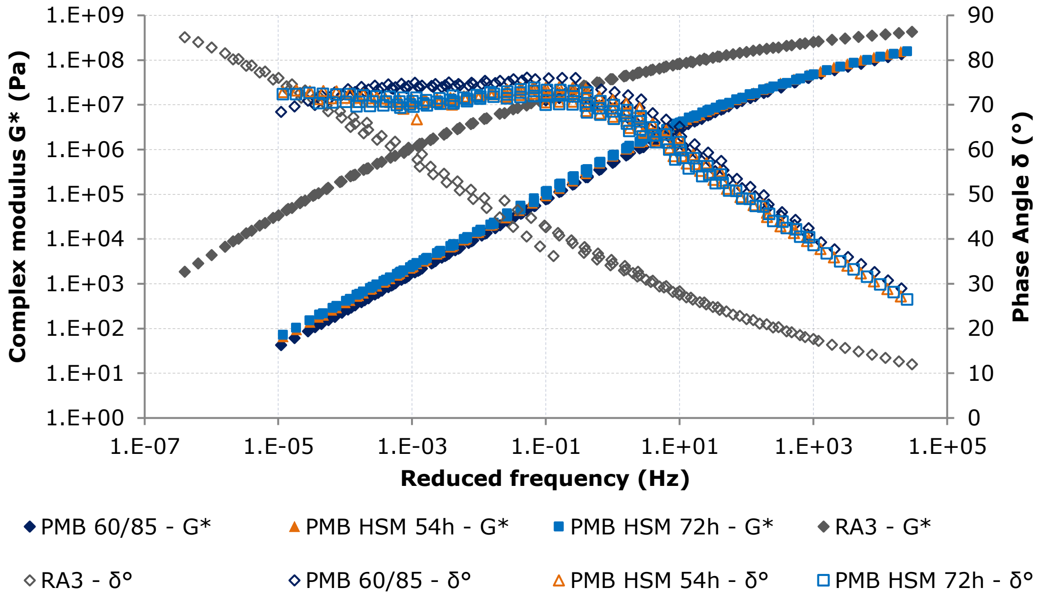


Figure 48: PMB 60/85 Ageing procedure – Complex modulus and Phase angle

Different from the results obtained in the ageing of the neat bitumen, the PMB results of the G* and δ were not evident after several hours of ageing. Although the penetration and SP results have been satisfactory achieved, the ageing does not seem to have the same effect on DSR rheology. Analysing the master curves of G* and δ, after subjecting the binder to 72h at high temperatures, there was no significant increase in binder stiffness or change in viscoelastic properties. One possible explanation for this may be the temperature chosen for the ageing in the laboratory with 163°C possibly being a relatively low temperature for a PMB. Also, the G* master curves of the RA3 bitumen showed very stiff behaviour with extreme properties which may not be able to be achieved with artificial ageing. Despite these results, the other investigated tests (Pen, SP and Viscosity) showed satisfactory results and for this reason the binder ageing for 72h was adopted for the next step of the DoBA Study. Furthermore, these results together with the VB 50/70 case, can be analysed in a way which it will be promising to identify which properties become more critical for the understanding of the activation of the RA binder, physical or rheological approaches.

4.4. Samples manufacturing

4.4.1. RA samples

The cohesion test procedure was performed first with the RA1 material using the methodology described in Section 3.1.3. Marshall moulds were used for compaction with specimens of 100mm in diameter and 63mm in height (as target). Using the maximum density of the material, the target height and the air voids content (estimated according to previous results from RILEM TG237 (Tebaldi *et al.*, 2018), the amount of material that should be placed in each mould for compaction was determined. The procedures for selecting material until the compaction of the first RA was as follows:

1. Selecting RA: the material was collected from a large amount stored in the laboratory and protected from the weather. For each selection, the material is subjected to the quartering procedure, using a riffle box, in order to collect homogeneous samples. After passing the material in the riffle box, the final amount of RA obtained should be enough to compact six Marshall samples (due to the mixer limitations);

2. Samples weight: Table 13 shows the amount of material placed in each sample, according to the maximum density of the material and the estimated final air voids content for the final specimens from RA1:

Table 13: Cohesion Test RA1 – Samples weight

Producing Temperature (°C)	Estimated air voids content (%)	Samples weight (g)
70	15.0	1007.3
100	12.0	1042.8
140	10.0	1066.5
170	10.0	1066.5

3. Conditioning in oven: 4 hours in an oven at the specified temperature;

4. Mixing procedure: the inclined mechanical mixer was used, and the mixing temperature was controlled. Figure 49 shows the equipment and the material during the mixing process. The amount of RA in the picture corresponds to six specimens;

Figure 49: Cohesion Test RA – Mechanical mixer

5. Compaction: after mixing the material, the compaction starts. Before placing the Marshall mould in the compactor, the temperature of each sample was recorded immediately before starting the compaction. The moulds were also placed in the oven before compaction for conditioning;

6. Production: with the results obtained from the RA1 samples, the procedure was adjusted according to the necessities of this research regarding mixing times and estimated air voids content. The procedure was then performed on the other sources of RA – RA2 and RA3.

4.4.2. Artificial RA samples

A different procedure was performed when manufacturing artificial RA mixtures: All mixtures are initially blended at high temperatures (170°C) as they are virgin materials; then conditioned in an oven for 1 hour in order to reach the desired temperature (except for 170°C mixtures where compaction is carried out just after mixing); 60 seconds mixing again for those mixtures with temperatures lower than 170°C, then compaction. The 60s mixing prior to compaction was chosen due to the results found for the RA (Section 4.5) which could also be compared between both cases.

The artificial mixtures composition was reproduced according to the original RA grading white curve and bitumen content, already identified. **Figure 50** presents the final grading curves reproduced with virgin materials in the laboratory:

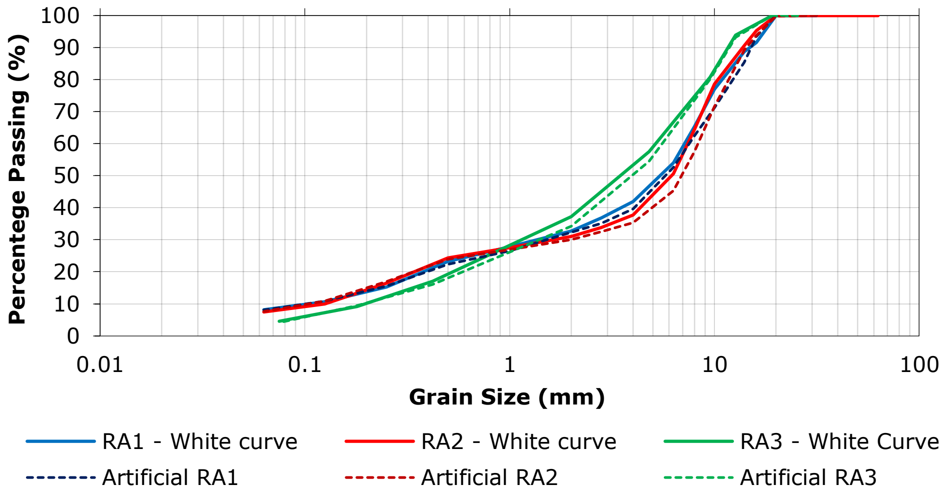


Figure 50: Artificial RA - grading curves

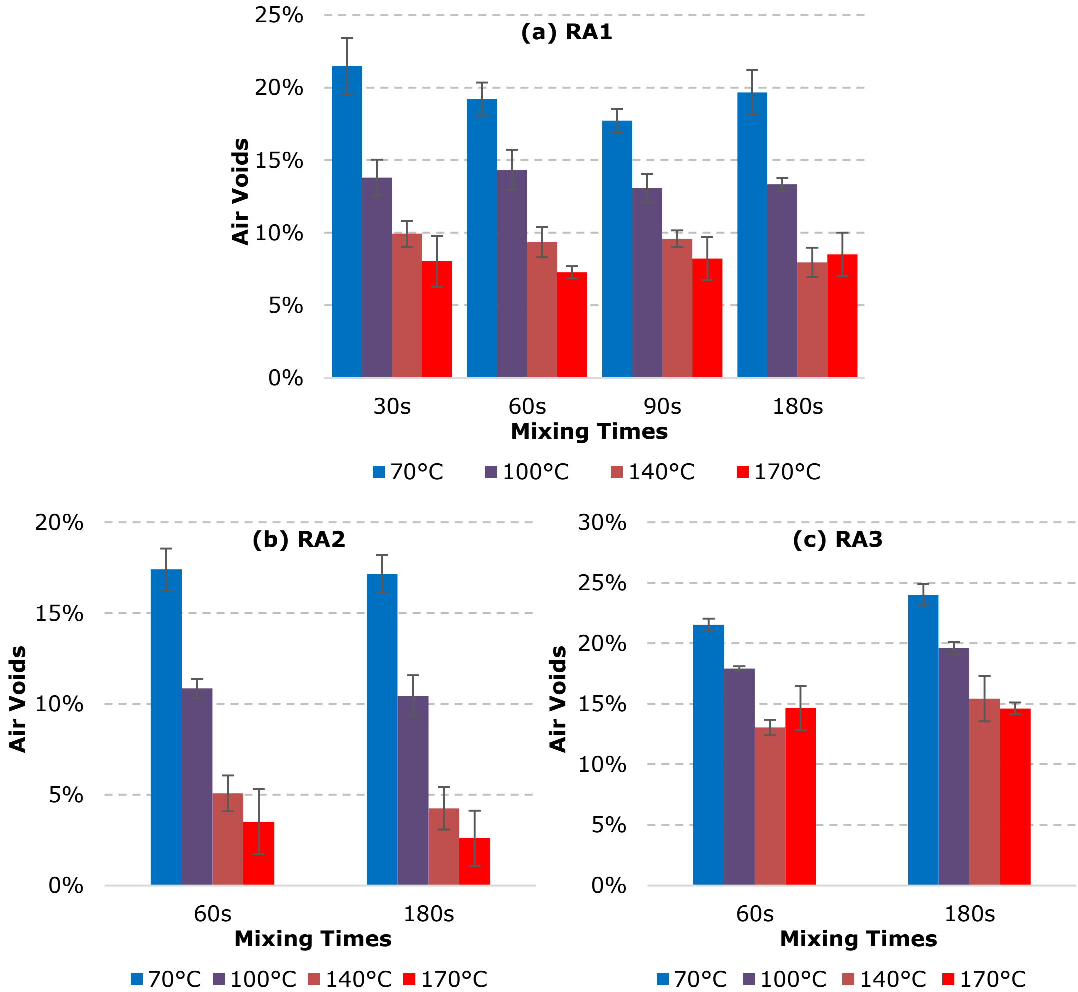
Following the grading curves reproduced in the lab, the final bitumen content of those mixtures was equal to 4.4% (RA1), 4.7% (RA2) and 4.4% (RA3).

4.5. Influence of compaction temperature and time

This section presents the compacted samples characteristics of air voids, compaction temperatures and ITS results for the first analysis. The air voids were calculated according to the European Standard EN 12697-6 (2012). The average air voids content (together with error bars showing the range in air voids associated with one standard deviation above and below the average) for the RA's compacted specimens are presented in Figure 51.

The results show that the average air voids content decreases as a function of increasing mixing (and conditioning) temperature for each group of mixing times.

However, the influence of mixing times (30 seconds to 180 seconds) appears to have less effect on the resulting average air voids content for RA1 with similar values being found for the different mixing times at each particular mixing temperature. Due to the outcomes of the RA1 air voids, the mixing times were adjusted for the other sources of RA by removing the 30s and 90s mixing times.



(Error bars show 1 standard deviation level)

Figure 51: Cohesion Test – Air Voids

The influence of mixing temperature for the study can clearly be seen in Figure 51 with the higher temperatures resulting in a softening of the RA binder (reduction in binder viscosity) thereby improving the workability of the material and aiding in the compaction of the RA samples and the resultant lower air voids content. Volumetric proportions (air voids) of compacted RA samples could therefore also be used as a measure of binder mobilisation (or activation). The results also show that there are smaller differences in air voids content between 140 and 170°C compared to the differences found from 100 to 140°C. This would imply that the amount of binder activation in RA (at least for the three RA's used in this study) potentially reaches an optimum (or threshold) at high mixing temperatures.

The ITS results for RA's as a function of mixing temperature and time are shown in Figure 52. All the specimens used to determine the ITS results were conditioned for 24 hours at 25°C inside a temperature-controlled cabinet prior to subjecting the specimens to the ITT. These initial results were used to determine the mixing times selected to produce the control pseudo-RA mixtures for the DoBA investigation.

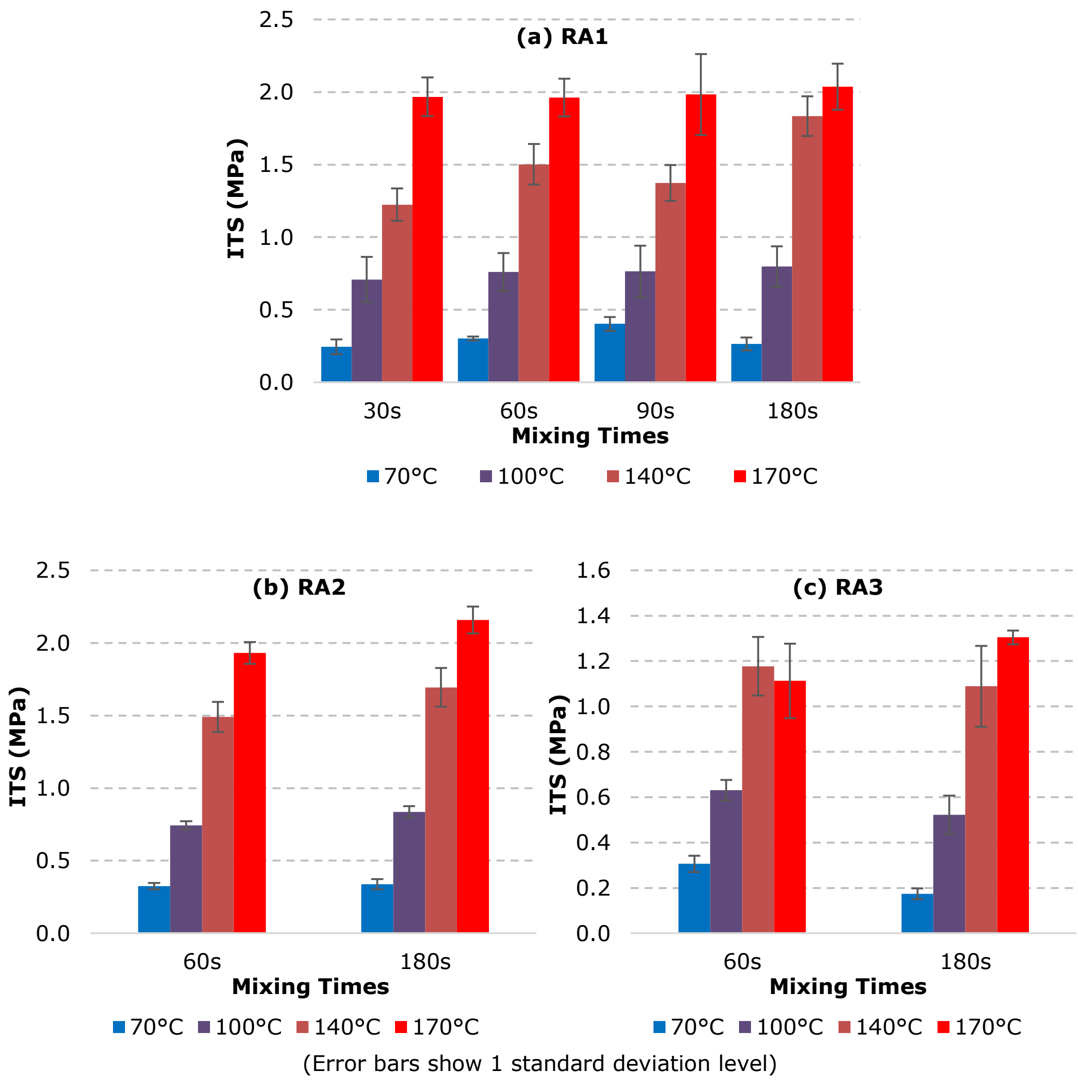


Figure 52: Cohesion Test – ITS by mixing temperatures and times

The results show that the ITS values increase with increasing conditioning and mixing temperature regardless of the mixing times. The highest tensile strength values were consistently found for the RA1 and RA2 specimens produced at 170°C, indicating the significant temperature dependent of the RA material regarding proposed binder activation (increased material cohesion) and increased density (higher degree of compaction). In the RA3 case, some variations in the results were found at high temperatures, the same trend as RA1 and RA2 was found with 180s mixing time, whereas by mixing for 60s showed minimal differences at 140°C and 170°C possibly due to the PMB binder and its polymer activity starting when a certain temperature is surpassed. Same as seen for the air voids content, mixing times were found to be less important and influential compared to temperature.

Considering each of the specific mixing temperatures for the RA1, where the first specimens were manufactured and evaluated for the experimental programme optimisation, the results at 70°C show some variation with average ITS values tending to increase with longer mixing times, although the differences cannot be considered significant. In terms of a visual assessment of the RA material during mixing and compaction, there is little evidence of significant binder activation with the material remaining opaque in colour with evidence of uncoated fine fractions on the surface of the compacted specimens (Figure 53).

Figure 53: RA1 specimen – 70°C

It is possible that increased mixing time (180 seconds) could have led to the disintegration of RA lumps and the release of larger amounts of fine material which, without the required degree of binder activation, resulted in materials harder to compact (higher air voids as shown in Figure 51a) and less internal material cohesion (lower ITS values in Figure 52a). Also, in Figure 53 can be seen the specimen after testing, the rupture type can be said as similar to the type C presented in **Figure 32** (Section 3.1.3.2), with a combination of tensile break line and some deformed areas close to the loading strips (effect of the compression).

Regarding the conditioning and mixing temperature at 100°C, the variation, in this case, was quite low. The ITS results were not affected by the mixing time. However, it is possible to affirm that at this temperature, the specimens began to show superior consistency with the apparent contribution of the bitumen when handling the material. Herein not only the compaction spearheaded the results (such as 70°C), but also the reactivation of the bitumen. A 100°C manufactured specimen from RA1 is presented in Figure 54. In Figure 54 can be observed the clear tensile break, with the specimen broken along a diametrical line with some small compressed region close to the loading strip.

Figure 54: RA1 specimen – 100°C

Considering the results for those specimens produced at 140°C, the behaviour of the material seems to be wholly influenced by the bitumen. The appearance after any of the four different mixing times starts to become similar to a virgin asphalt mixture, as shown in Figure 55 from RA1. The evidence in this visual assessment is noticeable during the mixing process, with the RA material presenting loose coarse aggregates, the clustering phenomenon in the fine aggregates as well as the shining black colour of the bitumen. Regarding the mixing times, significant variation was found, the ITS increased with the increase of mixing time, different from all other temperatures. As in the case of the lowest temperature, the time could have led to the disintegration of RA lumps, but in this situation to improve the final mixture with more bitumen involved and a more homogeneous blend. A clear tensile break is observed with the specimen broken along a diametrical line.

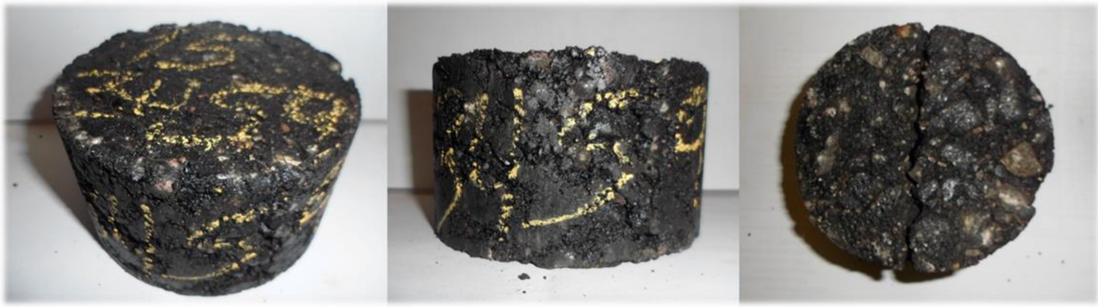


Figure 55: RA1 specimen – 140°C

Finally, specimens compacted at 170°C, the material showed similar appearance to a virgin asphalt mixture with a significant amount of bitumen involved. The mixing times do not show any influence on the final ITS results. A 170°C manufactured specimen from RA1 is presented in Figure 56. Can be observed the clear tensile break, with the specimen broken along a diametrical line with some small compressed region close to the loading strip.

Figure 56: RA1 specimen – 170°C

The last analysis is regarding the water sensitivity where the specimens were soaked in water for 24 hours at 25°C before testing to determine the ITS and the ITS Ratio. It is predicted that compacting the mixtures under normal compaction temperatures results in the inappropriate and insufficient coating, bonding, compaction or occurrence of structural cracks. According to the European standard EN 13108-1 (2016), the minimum ITS ratio value for an Asphalt Concrete is 0.70 or 70% of the value obtained from the unconditioned samples. Results of the ITS Ratio for the RA1 is presented in Figure 57:

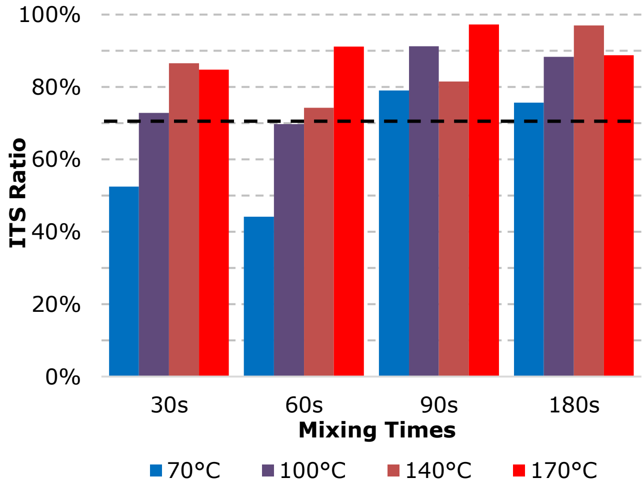
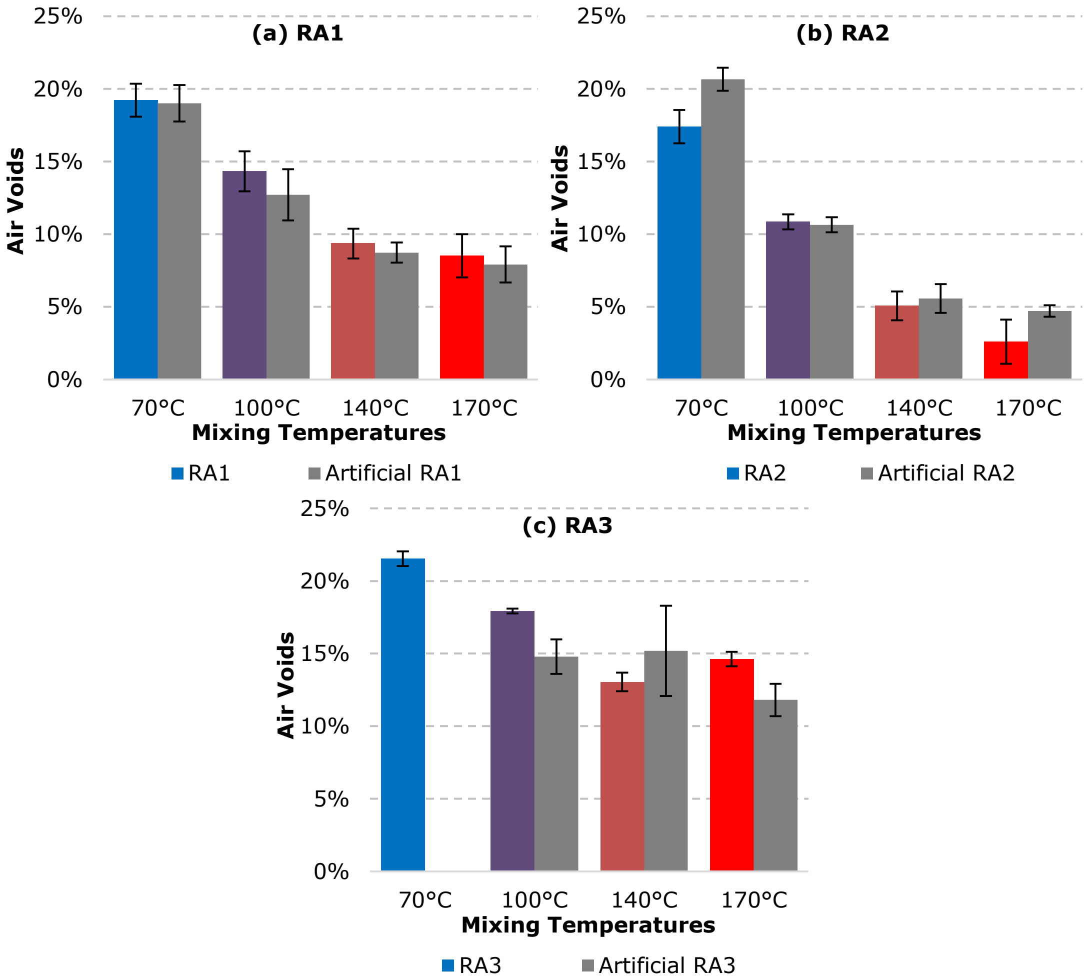


Figure 57: ITS Ratio – RA1

As can be seen from the figure, the ratio for the specimens produced at the specified temperatures of 100°C to 170°C was superior or equal to 70% for all mixing times applied. For specimens produced at 70°C, the minimum was not reached when mixing for 30s and 60s. It is possible that higher mixing times (90s and 180s) have led to the disintegration of RA lumps and released fine RA material which resulted in more homogeneous samples, reducing the moisture susceptiblity. However, the specifications determine the 70% ITS ratio for HMA, and analysing an RA without treatment at low temperatures with high air voids content, only two situations at the lowest temperature failed to achieve the minimum ratio. For this reason, understanding that the water sensitivity analysis could not provide any further information for the DoBA study, the other sources of RA were only tested in their unconditioned condition.

4.6. Assessment of the DoBA of the selected RA's

Based on the results presented in the previous section, just one mixing time was chosen to manufacture the artificially aged RA mixtures and to analyse the DoBA: 60 seconds for 70°C to 140°C and 180 seconds for 170°C due to the manufacturing limitations and comparison capability (Section 4.4). Figure 58 shows the air voids of the produced samples.



(Error bars show 1 standard deviation level)

Figure 58: Cohesion test RA's and Artificial RA's – Air Voids

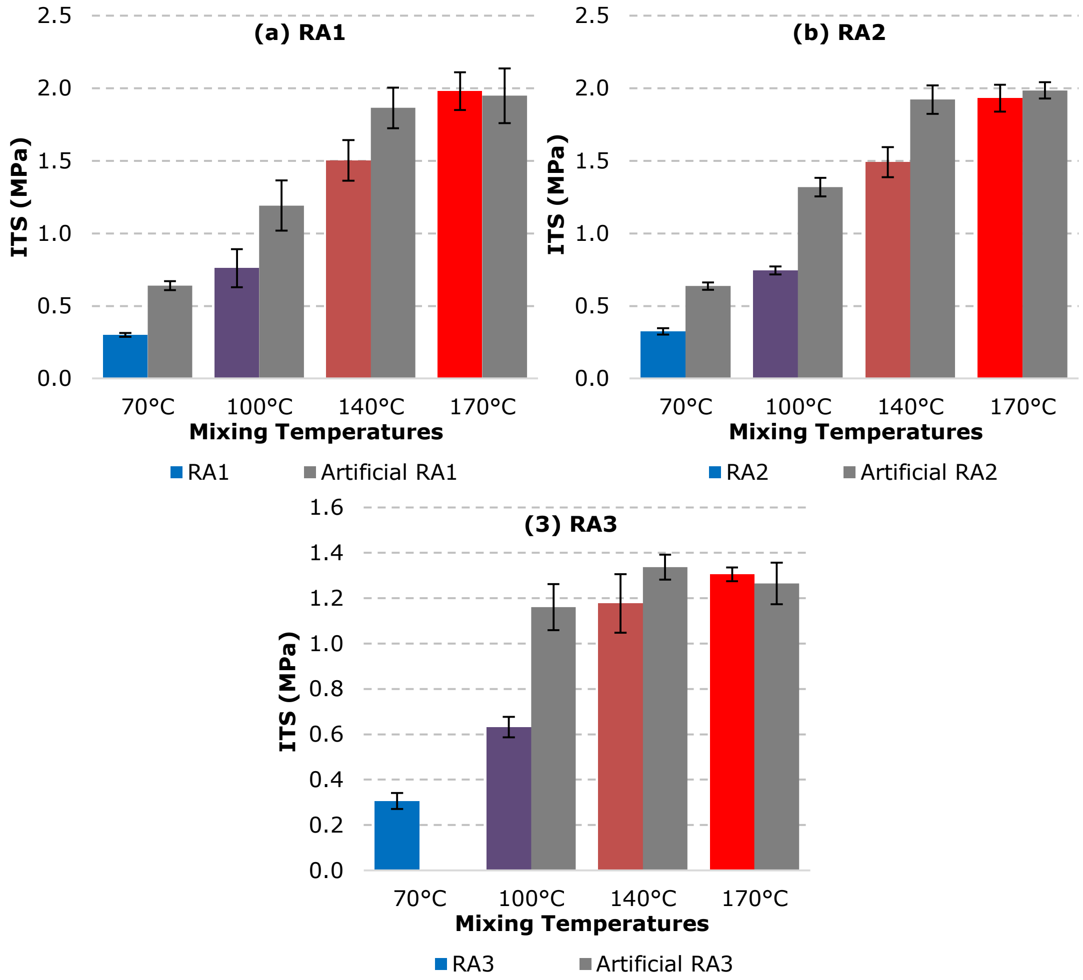
From these results it can be seen that the values were controlled during the manufacturing process in order to have comparable cases regarding volumetric properties since it is known that the voids can considerably affect the mechanical results.

In order to analyse statistically some results, the Student's t-test was applied for testing a hypothesis on the basis of a difference between sample means. Table 14 presents the results found for the cases where the RA and Artificial RA means do not differ significantly (statistically the same). The analysis was based on the t-Test: Two-Sample Assuming Unequal Variances.

Table 14: Air voids - Student's t-test analysis

t-Test: Two-Sample Assuming Unequal Variances					
Case	**Material**	**Variance**	**Degrees of freedom (df)**	**t-value**	**t critical two-tail**
RA1 - 70°C	RA1	1.284	4	0.246	2.776
	Art.RA1	1.583			
RA1 - 100°C	RA1	1.894	8	1.667	2.306
	Art.RA1	3.103			
RA1 - 140°C	RA1	1.061	10	1.257	2.229
	Art.RA1	0.476			
RA1 - 170°C	RA1	2.214	5	1.330	2.570
	Art.RA1	0.074			
RA2 - 100°C	RA2	0.725	13	0.077	2.160
	Art.RA2	0.085			
RA2 - 140°C	RA2	0.984	6	-0.707	2.447
	Art.RA2	1.369			
RA3 - 140°C	RA3	4.2E-05	3	-0.956	4.302
	Art.RA3	9.7E-04			

In terms of the ITS results (Figure 59), a similar tendency for all three cases can be seen with peak load increasing in accordance with the temperature increase which may be the result of the increased mobilisation of the binder raising the ITS. Furthermore, the ITS results confirm the importance of temperature conditioning where for the higher the temperature there is a smaller difference between RA1-RA2 and Artificial RA1-RA2 values. This means that the obtained maximum values for Artificial RA's are close to the maximum possible values for these RA's. It is important to note the similar results for RA1 and RA2 which can be explained by the similar properties of the two binders and mixtures grading.



(Error bars show 1 standard deviation level)

Figure 59: Cohesion test RA's and Artificial RA's – ITS

Comparing RA3 (Figure 59c) with the other two sources of RA, it can be concluded that smaller values of ITS can be attributed to the finer grading curve. However, this does not mean that the bitumen activity is lower and the evaluation with the artificial RA becomes important. For the three RA's, considering the deviation, from 140°C the lab produced RA's achieved the maximum tensile strength which may mean that maximum activation can be reached from warm temperatures. Furthermore, the RA3 also showed better activation at lower temperatures of at 100°C for the artificial RA. Despite this, the Artificial RA3 could not be produced in the lab at 70°C due to laboratory limitations during the compaction procedure.

In order to analyse statistically some results, the Student's t-test was also used. Table 15 presents the results found for the cases the observed difference between the RA and Art.RA means is not convincing enough to say that they differ significantly. The analysis was based on the t-Test: Two-Sample Assuming Unequal Variances.

Table 15: ITS - Student's t-test analysis

t-Test: Two-Sample Assuming Unequal Variances					
Case	**Material**	**Variance**	**Degrees of freedom (df)**	**t-value**	**t critical two-tail**
RA1 - 170°C	RA1	0.011	6	0.351	2.447
	Art.RA1	0.021			
RA2 - 170°C	RA2	0.006	5	-0.904	2.571
	Art.RA2	0.007			
RA3 - 140°C	RA3	0.017	3	-1.969	3.182
	Art.RA3	0.003			
RA3 - 170°C	RA3	9.2E-04	3	0.958	3.182
	Art.RA3	0.004			

Although the ITS results were promising, further analysis of the entire stress versus displacement was analysed for some cases of RA1 and Artificial RA1 produced in the laboratory and presented in Figure 60 for comparison. The horizontal deformation was obtained through the vertical deformation recorded during the test together with the Poisson ratio – 0.35 adopted in this research.

Can be seen in the charts plotted in Figure 60 that there are differences between original and lab produced RA’s. For the 170°C conditioning temperature, it can be seen that although the maximum ITS results are similar for both materials, the test curves for the laboratory aged binder shows a greater capacity to absorb and dissipate the energy acquired during the test.

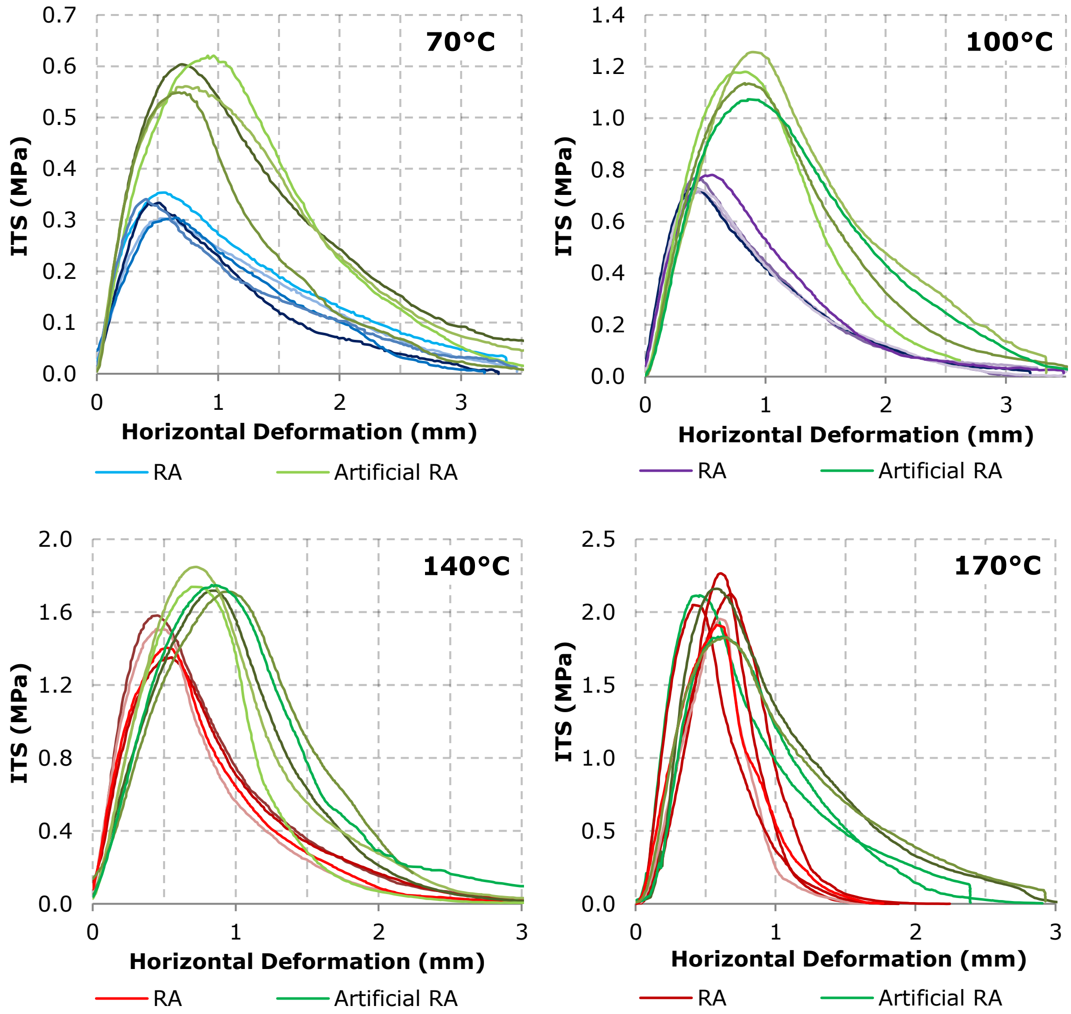
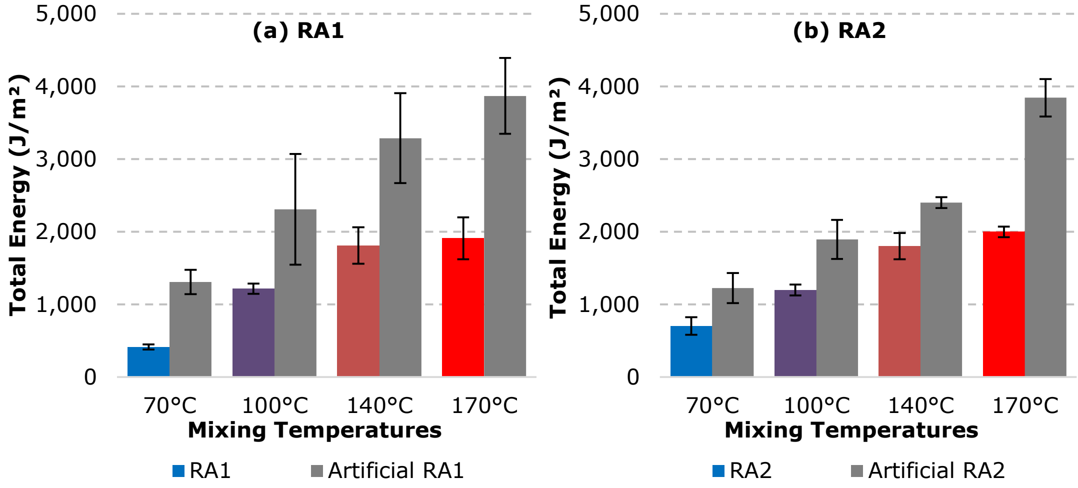


Figure 60: Cohesion test RA1 and Artificial RA1 – ITT deformation curve

The energy absorption capacity can also be observed at other temperatures with larger areas below the stress-displacement curves for the Artificial RA specimens. However, this is partly due to the higher loads associated with the peak strength of the specimens. In this sense, the figure presenting results at 170°C shows the brittle properties of the RA and its lower capacity to store energy after the first crack, in distinction to artificial RA mixtures that can still withstand the test for a longer time. For this reason, other parameters were included in this investigation related to the shape and magnitude of the curves: total energy, pre-peak energy, post-peak energy and flexibility index. This analysis was only undertaken for RA1 and RA2.

Figure 61 shows the Total Energy (E_{Total}) results determined according to Section 3.1.3.2.2 from Chapter 3. As in the ITS results, higher E_{Total} values were obtained for the Artificial RA's, also, a similar trend in the chart columns can be observed (energy increasing with the temperature increase).

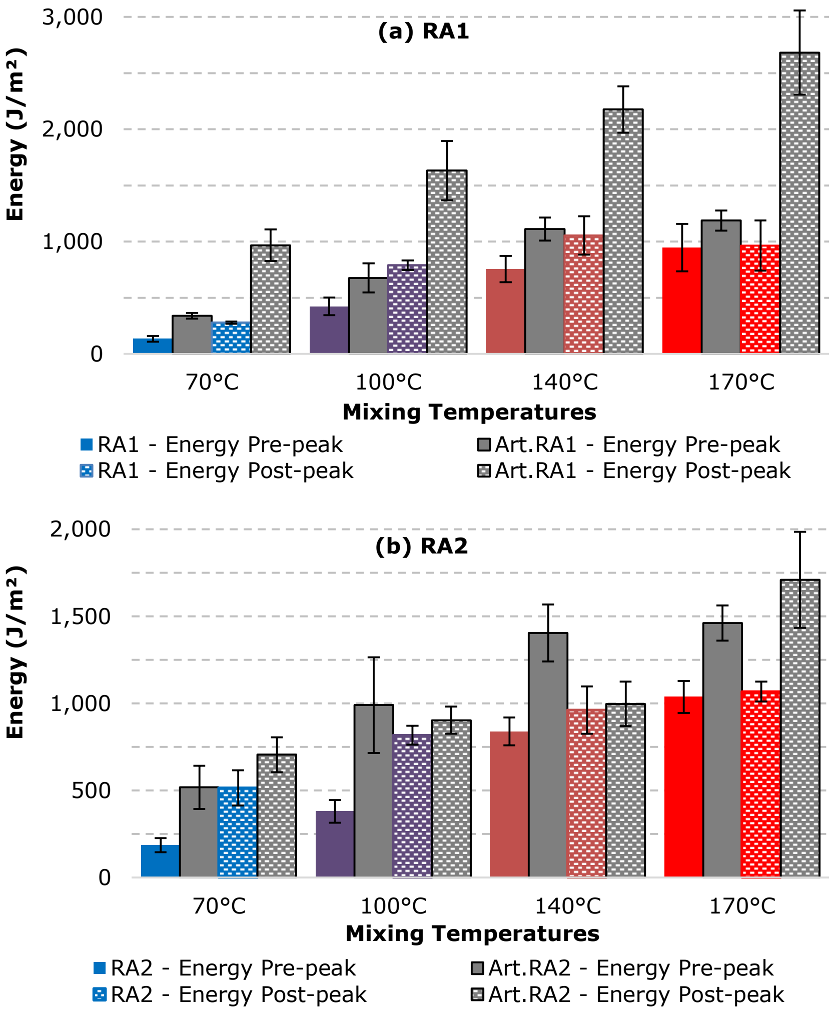


(Error bars show 1 standard deviation level)

Figure 61: Cohesion test RA's and Artificial RA's – Total Energy

The analysis carried out, in addition to the ITS, becomes interesting here at 170°C where the RA's show lower energy absorption capacity, meaning that the 100% RA samples are not that tough even though they have high ITS, where the material cracks with a short load displacement during the test. Due to this situation, it becomes essential to analyse the Pre and Post peak load regions (through the Total Energy) that can provide an overall assessment that considers the load-displacement connected to the energy absorption on each sample, before and after the crack initiate. The energy parameters were obtained through the procedures presented in Section 3.1.3.2.2 and are presented in Figure 62.

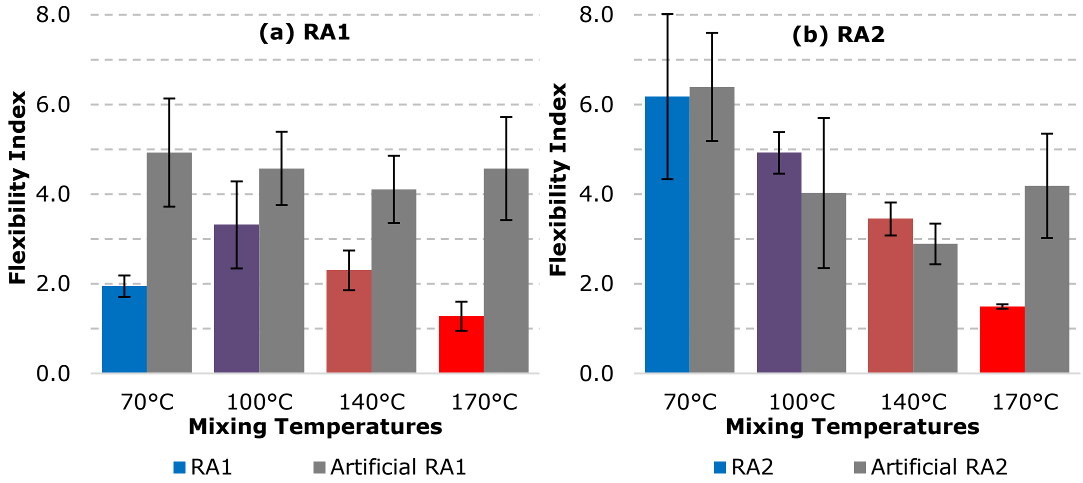
The main differences that can be observed in Figure 62 are in the $E^{Post\text{-}peak}$ results for Art.RA1 as the other results seems to be in equilibrium for the two RA's selected. It is also essential to observe that the values $E^{Post\text{-}peak}$ present high variation when comparing artificial and original materials, without obeying linearity in respect to the temperature changes that may affect the DoBA analysis – aim of the investigation.



(Error bars show 1 standard deviation level)

Figure 62: Cohesion test RA's and Artificial RA's – Pre and Post-peak Energy

Figure 63 shows the last parameter analysed, the Flexibility Index (FI), determined according to Section 3.1.3.2.3 in Chapter 3. The FI is a simple index parameter that can be correlated to fundamental crack growth mechanisms and has the ability to distinguish mixes with distinct design characteristics that may influence cracking resistance.



(Error bars show 1 standard deviation level)

Figure 63: Cohesion test RA's and Artificial RA's – Flexibility Index

The range of FI values for the mixtures was in the range of 1 to 6 with increasing brittleness with lower FI values. Both RA's behave differently when comparing the final results, RA1 shows some variation when the specimens were conditioned and produced at different temperatures, with the brittleness increasing from 100°C to 170°C. The laboratory artificial RA1 presents an almost constant index for all four temperatures, where the samples do not seem to be affected by the temperature production regarding flexibility. However, in the case of mixtures RA2 and Artificial RA2, the expected trend was found for nearly all situations where the FI decreases with increasing production temperature. Moreover, all cases presented a high variation of results and, due to this together with the inconsistency in the results, FI does not seem to be a promising parameter for the evaluation of the RA binder activation, since the increase in the fragility of the material opposes the idea that a larger amount of binder involved would strengthen the compacted mixture.

4.6.1. RA DoBA Labelling

In order to estimate the DoBA for each temperature and materials studied, the parameters investigated (Section 4.6) were applied in the Equation 49. The equation was developed to be used in accordance with the framework proposed in

Section 3.1, resulting in the DoBA obtained through mechanical tests. The calculated DoBA's are presented in Table 15,

Table **16** and Table 17, for RA1, RA2 and RA3 respectively.

The results assume that Artificial RA reached the maximum DoBA value on each analysis, with the DoBA as a mechanical relationship, the ratio between both materials (RA and Artificial RA) could then be achieved. In addition, as the results were obtained through mechanical tests, the DoBA from these outcomes are called in this thesis as DoBA'.

$$DoBA' \, (\%) = 100 \times \frac{Y_{RA} \, (X°C)}{Y_{Art.RA} \, (X°C)} \qquad \text{(Equation 49)}$$

Where,

Y_{RA} (X°C) = parameter result "Y" for the RA at a specific temperature "X";

$Y_{Art.RA}$ (X°C) = result of the artificial RA at the same temperature analysed for RA.

Table 15: DoBA' estimation for RA1

Temperature (°C)	Analysis	RA1	ART. RA1	DoBA' (%)
70	ITS Peak Stress (MPa)	0.302	0.639	47.2
	E_{Total} (J/m²)	412.0	1306.8	31.5
	$E^{Pre\text{-}peak}$ (J/m²)	134.1	339.9	39.5
	$E^{Post\text{-}peak}$ (J/m²)	277.9	966.9	28.7
	Flexibility Index	1.949	4.928	39.5
100	ITS Peak Stress (MPa)	0.760	1.192	63.8
	E_{Total} (J/m²)	1212.9	2307.4	52.6
	$E^{Pre\text{-}peak}$ (J/m²)	422.7	676.2	62.5
	$E^{Post\text{-}peak}$ (J/m²)	790.2	1631.2	48.4
	Flexibility Index	3.313	4.573	72.4
140	ITS Peak Stress (MPa)	1.502	1.864	80.6
	E_{Total} (J/m²)	1809.3	3287.2	55.0
	$E^{Pre\text{-}peak}$ (J/m²)	754.9	1110.8	68.0
	$E^{Post\text{-}peak}$ (J/m²)	1054.5	2176.4	48.5
	Flexibility Index	2.300	4.104	56.0
170	ITS Peak Stress (MPa)	1.980	1.949	101.6
	E_{Total} (J/m²)	1909.4	3869.4	49.3
	$E^{Pre\text{-}peak}$ (J/m²)	945.9	1187.0	79.7
	$E^{Post\text{-}peak}$ (J/m²)	963.5	2682.4	35.9
	Flexibility Index	1.276	4.567	27.9

Table 16: DoBA' estimation for RA2

Temperature (°C)	Analysis	RA2	ART. RA2	DoBA' (%)
70	ITS Peak Stress (MPa)	0.330	0.637	51.2
	E_{Total} (J/m²)	700.1	1223.2	57.2
	$E^{Pre\text{-}peak}$ (J/m²)	185.8	518.0	35.9
	$E^{Post\text{-}peak}$ (J/m²)	514.3	705.2	72.9
	Flexibility Index	6.176	6.389	96.6
100	ITS Peak Stress (MPa)	0.744	1.221	61.0
	E_{Total} (J/m²)	1196.8	1893.7	63.2
	$E^{Pre\text{-}peak}$ (J/m²)	380.2	990.2	38.4
	$E^{Post\text{-}peak}$ (J/m²)	816.6	903.5	90.4
	Flexibility Index	4.923	4.025	122
140	ITS Peak Stress (MPa)	1.490	1.922	77.5
	E_{Total} (J/m²)	1800.7	2401.5	75.0
	$E^{Pre\text{-}peak}$ (J/m²)	839.2	1404.4	59.8
	$E^{Post\text{-}peak}$ (J/m²)	961.5	997.1	96.4
	Flexibility Index	3.446	2.889	119
170	ITS Peak Stress (MPa)	2.158	2.018	107.0
	E_{Total} (J/m²)	2104.0	3844.6	54.7
	$E^{Pre\text{-}peak}$ (J/m²)	1036.6	1461.2	70.9
	$E^{Post\text{-}peak}$ (J/m²)	1067.4	1709.4	62.4
	Flexibility Index	1.492	4.183	35.7

Table 17: DoBA' estimation for RA3

Temperature (°C)	Analysis	RA3	ART. RA3	DoBA' (%)
70	ITS Peak Stress (MPa)	0.306	-	-
100	ITS Peak Stress (MPa)	0.631	1.160	54.4
140	ITS Peak Stress (MPa)	1.177	1.337	88.0
170	ITS Peak Stress (MPa)	1.300	1.265	102.7

From the tables, the charts in Figure 64 (RA1), Figure 65 (RA2) and Figure 66 (RA3) were produced representing the DoBA' percentage ranges as well as the active binder content for the studied temperatures and cases. The four parameters are plotted following the principles of Equation 63.

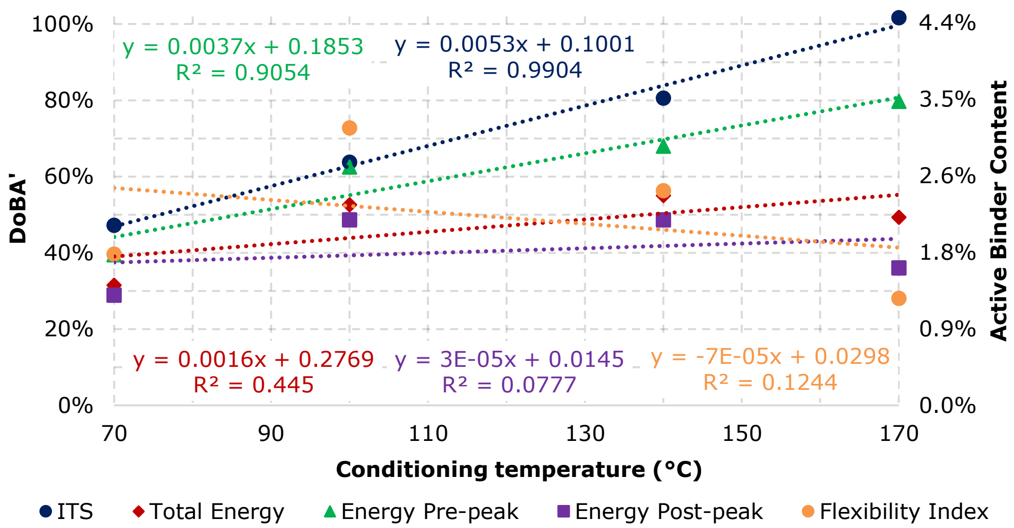


Figure 64: DoBA' estimation for RA1

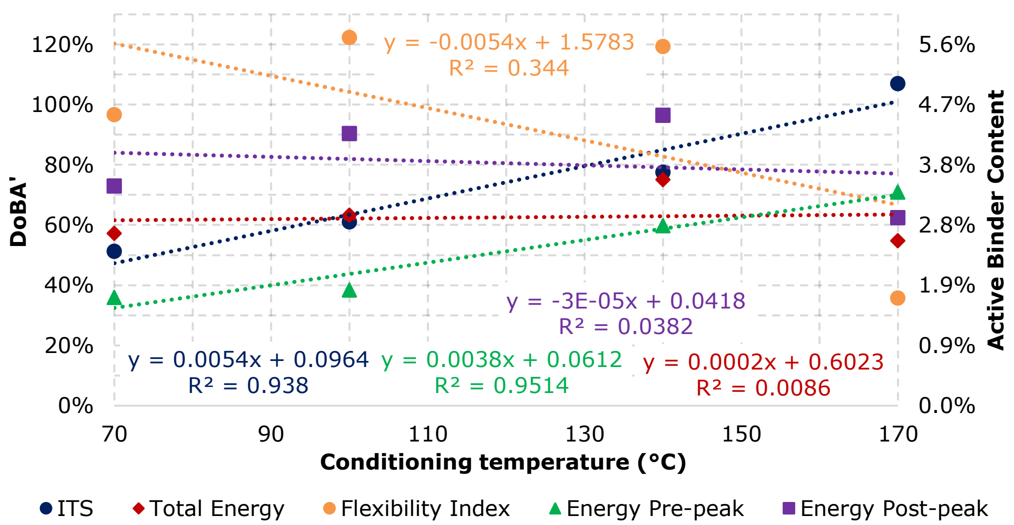


Figure 65: DoBA' estimation for RA2

Analysing the estimated DoBA' from RA1 and RA2, it can be seen a similar trend in the regression lines for ITS and $E^{Pre\text{-}peak}$. Moreover, it is possible to observe the good correlation and the high value of R^2 (>0.9) from the linear regression analysis for both RA1 and RA2. The same analysis can be made for the RA3 and the ITS as parameter in Figure 66.

However, the results of E_{Total}, $E^{Post\text{-}peak}$ and FI do not show the same tendency of the other parameters, where for example from 100°C the values of DoBA' ratio start to decrease. Furthermore, the RA2 E_{Total} and $E^{Post\text{-}peak}$ results are higher for lower temperatures regarding DoBA which does not appear to be what actually occurs during the conditioning, mixing and compacting of the samples. What may explain this dispersion in the results is the more significant activation of the binder as well as its brittle properties that can affect the speed of cracking appearance during the test. The same analysis is not observed for the Artificial RA since the bitumen aging mechanisms are different from an original RA binder, therefore, the E_{Total}, $E^{Post\text{-}peak}$ and FI do not seem to be suitable for the DoBA ratio for the RA's investigated.

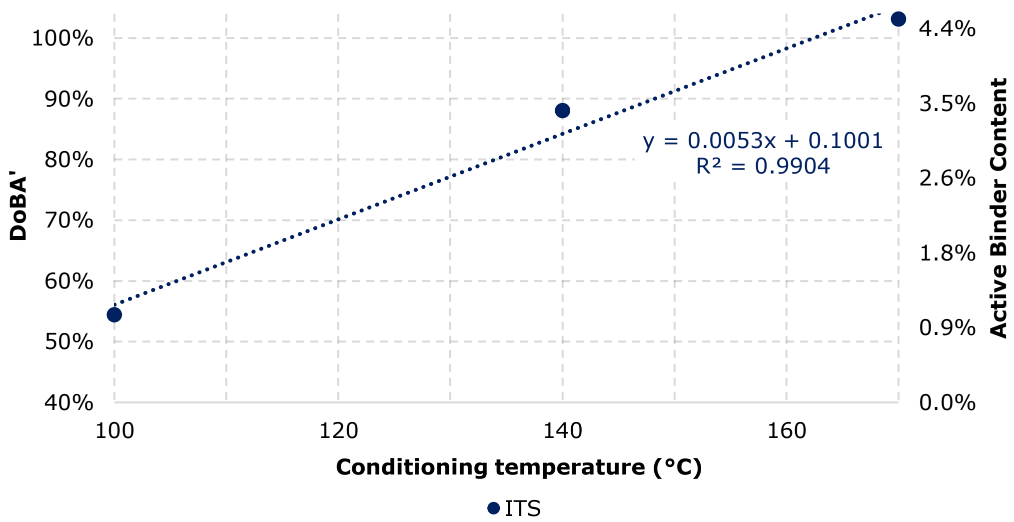


Figure 66: DoBA estimation for RA3

In summary, the analysis of the parameters is promising since the trends found are consistent with the expected values and confirm the main idea, already observed in the literature review, that the temperature is the most important factor in the activation of RA bitumen. Inevitably, the temperature increase should gradually boost the DoBA and not diminish it as was found for three analysed parameters (E_{Total}, $E^{Post\text{-}peak}$ and FI). Moreover, the total control of conditioning, mixing and sample production leaves no room for rapid ageing of the material in the oven, which could drastically alter the properties of the RA binder at high temperatures.

4.6.2. RA DoBA Labelling – Supplementary analysis

In addition to the investigation presented above, another way of analysis was elaborated. This consisted of a more straightforward method to use the results obtained from the Cohesion Test. In order to create the index to estimate the DoBA, only results from RA's samples produced at 170°C are used as a reference, which means that 100% DoBA is assumed for those materials conditioned at this temperature. The idea emerged from results found in the first DoBA' analysis (Table 18) and it is understood that the ITS parameter could provide a better application of the results due to the similarity in the results found. In this sense, for a more simplified investigation in future applications and possible replication of the methodology, the supplementary analysis using the ITS is presented.

Table 18: DoBA' estimation for RA's – ITS

Analysis	Temperature (°C)	DoBA' RA1	DoBA' RA2	DoBA' RA3
ITS Peak Stress	70	47.2%	51.2%	-
	100	63.8%	61.0%	54.4%
	140	80.6%	77.5%	88.0%
	170	101.6%	107%	102.7%

The following analysis is elaborated according to the previous results, by comparing the RA's and the Artificial RA's for each case at 170°C (highest activation), making it possible to assume these values as reference. Applying the Equation 50 and using the ITS_{RA}170°C as reference, the DoBA" can be estimated:

$$DoBA'' (\%) = 100 \times \frac{ITS_{RA}\ (X°C)}{ITS_{RA}\ (170°C)} \quad \text{(Equation 50)}$$

Where,

ITS_{RA} (X°C) = the ITS result of the RA at a specific temperature "X";

ITS_{RA} (170°C) = the ITS result of the RA produced at 170°C.

Once the analysis was finished, Table 19 was created and the chart in Figure 67 was drawn:

Table 19: DoBA" estimation for RA's – 170°C as reference

Analysis	Temperature (°C)	DoBA" RA1	DoBA" RA2	DoBA" RA3
ITS Peak Stress	70	15.2%	15.1%	23.4%
	100	38.4%	34.5%	48.4%
	140	75.9%	69.0%	90.2%
	170	100%	100%	100%

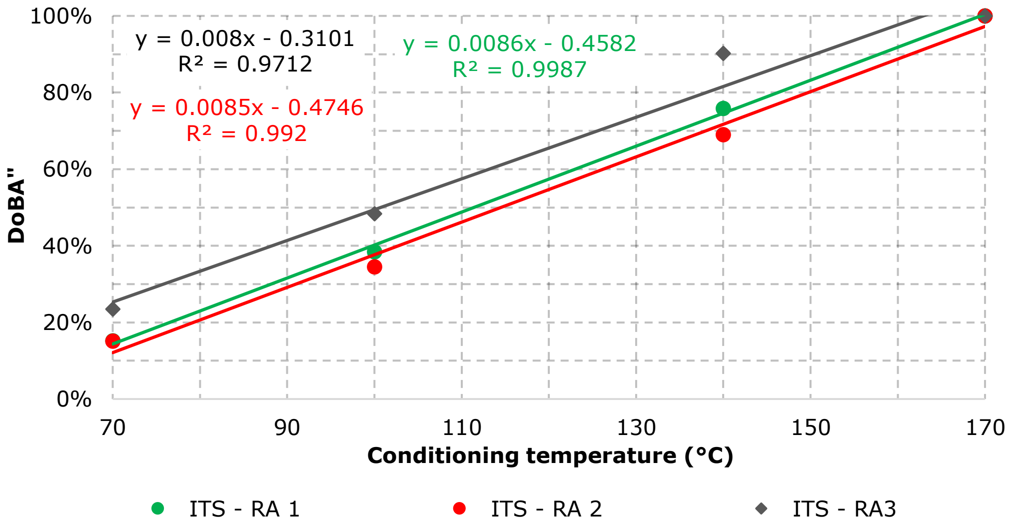


Figure 67: DoBA" estimation for RA1 – 170°C as reference

The results show the high similarity in all the results for the RA's investigated. A good correlation was found when using the 170°C as a reference, even if the ITS results were of different magnitudes for all the materials (RA's and Artificial RA's). Although the results compared with Art.RA show differences, the linear regression lines are matched to the analysed temperature range (70-170°C). There are also small differences for the R3 case with the regression line slightly shifted to the left with these results indicating a higher activation at temperatures below 170°C than any other RA studied. This analysis shows a slightly different trend found with the Artificial RA's (Table 18), where the RA3 with PMB mobilised larger amounts of bitumen at warm temperature (140°C) in comparison to RA1 and RA2, but lower activation than RA1 and RA2 at 100°C.

4.6.3. DoBA: Additive application

Due to the results presented in the previous section, another investigation was carried out to explore the DoBA with the inclusion of an additive. The selection of an additive was due to the necessity of a rejuvenation in the RA binder for the final mixtures as well as the performance-related tests on designed binders and mixtures with 100% RA1. In this sense, the Cohesion Test procedure with the additive was performed in order to check the improvements provided by the additive regarding DoBA.

The additive selected for this investigation was *SYLVAROAD RP1000 Performance Additive* produced by Arizona Chemical Company and Kraton Corporation. The additive is a polyol ester oil made from Crude Tall Oil and Crude Sulphate Turpentine, pine chemicals produced by the pulp and paper industry. According to the company, the liquid additive mobilises the aged binder of RA, improves the thermal and fatigue cracking resistance as well as water sensitivity of mixtures containing RA and allows higher amounts of RA in an asphalt mixture, providing lower viscosity and restoring the RA binder properties (Arizona Chemical, 2014; KRATON, 2017).

At the time the additive was considered in the research, no binder design had been performed. Thus, to initiate the production of samples of 100% RA1 + Additive and also Artificial RA1 + Additive, the manufacturer's recommendations were applied. The manufacturer's guidelines suggest that the RA binder penetration doubles its value for every 5% of incorporated additive relative to the weight of binder present in RA. Therefore, targeting a final penetration of 60 dmm and having an initial value of 16 dmm (Section 3.2.2.1) for the RA1, 9% additive was added for this preliminary investigation.

The specimen manufacturing process followed the same procedure adopted previously (Section 4.4), with minor modifications for the use of the additive:

- RA mixtures: the same procedure of conditioning the materials in an oven; initial mixing for 60 seconds with RA1 only, then the additive is poured into the mixer for a further 60 seconds mixing and, finally, compaction.

- Artificial RA: the same method of production of the artificial mixtures presented in the previous section was used. The difference, in this case, is that the additive was premixed to the aged binder in the laboratory. The virgin aggregates and the rejuvenated aged binder were then mixed for further compaction.

The compacted samples were analysed, and their air voids are shown in Figure 68 together with the ITS results – tested at 25°C.

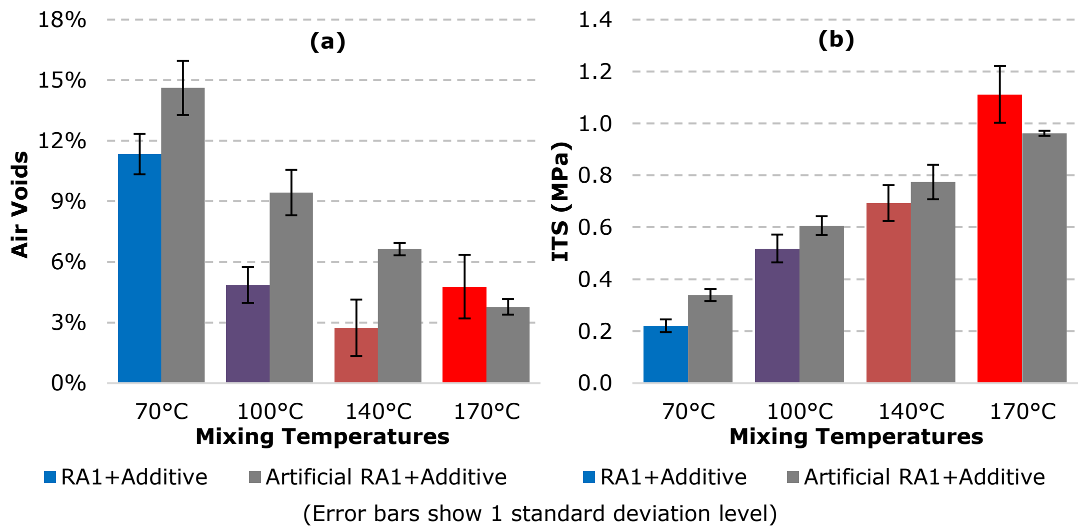


Figure 68: Additive application – (a) Air Voids (b) ITS

The air voids results obtained from the compaction of the samples present significant differences in two cases. For the 100°C and 140°C specimens, the Artificial RA presented twice the value of the RA samples air voids. In the 70°C and 170°C specimens, the differences presented are smaller, with higher voids in the case of artificial blends compacted at 70°C, and similar voids for specimens compacted at 170°C. With these results it can be said that the additive improved the workability of the RA1 mixtures, considerably reducing the air voids. At the same time, the artificial samples did not show satisfactory performance in comparison to the original RA1 (see Figure 59a compared to Figure 68b). Even so, the increase in compaction temperature shows the evolution of the compactability of the samples. These differences may directly affected the results of ITS in Figure 68b. The results can be seen to be very similar at all temperatures, with higher deviation only at 170°C. In the analysis of Figure 68, the ITS values of the Artificial RA1 may have been affected by the values of air voids as higher voids can compromise the resistance during the ITT.

In order to analyse statistically some results, the Student's t-test was also used. Table 20 presents the results found for the case only case (Air Voids - 170°C) where the observed difference between the RA and Art.RA means is not conclusive enough to say that they differ significantly. The remaining three ITS cases exposed are included to show that they are not statistically the same, despite being similar in Figure 68b.

Table 20: Additive application - Student's t-test analysis

t-Test: Two-Sample Assuming Unequal Variances					
Case	**Material**	**Variance**	**Degrees of freedom (df)**	**t-value**	**t critical two-tail**
Air Voids - 170°C	RA + Add.	2.492	6	1.497	2.447
	Art.RA + Add.	3.778			
ITS - 100°C	*RA + Add.*	*0.001*	*3*	*-3.810*	*3.182*
	Art.RA + Add.	*0.002*			
ITS - 140°C	*RA + Add.*	*0.005*	*4*	*-3.468*	*2.776*
	Art.RA + Add.	*0.001*			
ITS - 170°C	*RA + Add.*	*0.010*	*4*	*3.634*	*2.776*
	Art.RA + Add.	*0.001*			

Although some variations were observed, the DoBA analysis followed the same procedures and equations adopted in Sections 4.6.1 and 4.6.2, with the development of a ratio of DoBA’ to the artificial material as well as using the RA1 produced at 170°C as a reference. From these results, Table 21 was tailored (including DoBA’ without additive for comparison), and Figure 69 is presented:

Table 21: DoBA’ estimation for RA1 with additive

Analysis	Temperature (°C)	DoBA’ – Artificial RA1	DoBA’ Additive – Artificial RA1	DoBA” – 170°C RA1 Reference	DoBA” Additive– 170°C RA1 Reference
ITS Peak Stress	70	47.2%	65.1%	15.2%	21.2%
	100	63.8%	85.6%	38.4%	49.8%
	140	80.6%	89.4%	75.9%	66.5%
	170	101.6%	115%	100%	100%

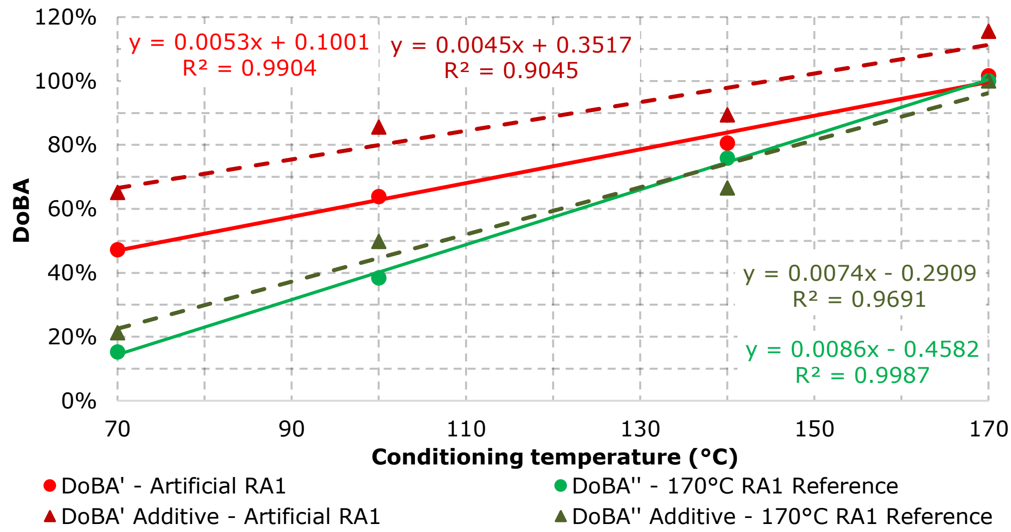


Figure 69: DoBA estimation for RA1 – Additive application

The results from estimated DoBA of the RA1 with an additive, show the good correlation between DoBA and conditioning temperature using ITS results. The linear trends (dash lines) show a high value of R^2 (>0.9) from the regression analysis for both RA1 using the artificial reference (DoBA’) as well as $RA1_{170°C}$ as reference in the final results (DoBA”). The regression lines can also be compared to the original values obtained previously (solid lines) with these lines showing an improvement in the DoBA. However, it should be remembered that the air voids content might be affecting the artificial RA1 results, thus, affecting the final DoBA’. Moreover, it is also known that another factor, namely the Degree of Blending (DOB), can be affecting the results because another material was added in order to blend with the RA1 binder. Regarding the DOB, the Artificial RA had all the bitumen and additive blended prior to manufacturing, whereas the original RA was directly mixed with the additive. In this last scenario it is not possible to know the final blend properties and how deeply the additive penetrates into the RA binder. Therefore, the effect of the DOB in this procedure cannot be measured. Despite this, the additive study in the DoBA investigation is still important to estimate how the RA binder behaves with the recycling agent and how it can support the binder and mixture design with the RA1. Therefore, the final chart presented in this section together with the previous results, are analysed to arrive at the following conclusions.

4.7. Summary and conclusions

The research in this chapter presents a methodology to assess the feasibility of using high RA percentages in asphalt mixtures. It is based on the RA binder availability which can reduce the amount of virgin binder that needs to be added. According to the laboratory investigation, the following conclusions can be drawn:

- Conventional equipment and methods can be used to mix, compact and test the mixtures with RA;

- The RA characterisation is essential when the focus is maximising RA content in new asphalt mixtures. This is especially relevant in terms of the recovered bitumen that guides the binder design for the new mixtures using virgin materials, rejuvenators or other available technologies;

- The procedure adopted to age the virgin bitumen to create an artificial RA binder proved to be efficient when adequately controlled. The objectives targeted in this procedure were achieved by providing bitumen characteristics similar to that of the RA binder. Penetration and SP tests can be applied as comparison methods to create a laboratory aged binder if a DSR is not available. The Rotational Viscometer can also be used;

- The first tests carried out showed that variations in conditioning and production temperatures is an important factor when considering 100% RA as higher temperatures increase ITS values. The same tendency was not found by increasing mixing times;

- The air voids content decreased when temperatures increased. This behaviour was expected and showed significant variations from 100 to 140°C. This highlights the importance of controlling the temperature during compaction and that the material does not need to be heated to very high temperatures. Between these temperatures (100 to 140°C) the RA bitumen began to be activated in larger amounts improving the workability.

Regarding the DoBA′ and DoBA″ determined using different parameters and methods, the following conclusions can be drawn:

- The ITS results showed a similar trend for 100% RA and Artificial RA with increasing peak loads in accordance with temperature rise. This highlights the importance of considering temperature conditioning;

- The Energy Pre-peak parameter proved to be an essential extra tool of analysis that considers other aspects of ITT such as the load-displacement and the energy absorption before the crack initiates and further crack propagation;

- The flexibility index, Total Energy and Energy Post-peak do not present satisfactory correlation with the temperatures as expected. This indicates that these parameters may not be realistic to use in the present methodology because they are opposed to what the literature proposes (RA bitumen is more easily mobilised when the temperature is increased);

- The DoBA′ index proposed to improve the binder and mixture design is an easy and simple tool to be performed. It provided an innovative idea of how to use the RA binder on recycled mixtures. Furthermore, the DoBA′ analysis was conducted on RA samples tested without any binder extraction, although the RA properties should be known. However, the procedure simulates the real mixing that RA materials are subjected (opposite from the literature discoveries where DoBA and DOB are assessed through binder results);

- The supplementary DoBA'' analysis (using $RA_{170°C}$ as reference) also presented satisfactory results, since a good correlation with the production temperatures was obtained. Also, this analysis does not require tests with Artificial RA. However, this method requires further investigations into the possibility of using high-temperature results as a reference as other sources of RA may present different outcomes;

- The additive application supported the DoBA' results obtained with Artificial RA, and as the additive manufacturer proposes, it can improve the RA bitumen mobilisation. In the methodology adopted, the additive application resulted in higher DoBA, and are to be used in the binder and mixture design.

5. RECYCLED BINDERS

5.1. Introduction

This chapter aims to compare the conventional and performance-related properties of the recycled binders to the virgin target bitumen. In the first part of the study, the RA1 binder is rejuvenated with an additive, assuming four DoBA percentages (x3 DoBA'; 1x DoBA"). The optimum rejuvenator dosage is defined using the penetration and the SP laws as well as matching the specifications in terms of Fraass breaking point and Penetration Index (EN 12591, 2009). The target binder has been defined as a 50/70 penetration grade bitumen with the specification details already presented in Section 3.2.3. The second part of the study presents the performance-related tests on the recovered and recycled binders in comparison with the reference binder. Therefore, the present chapter includes the following investigation according to Figure 70.

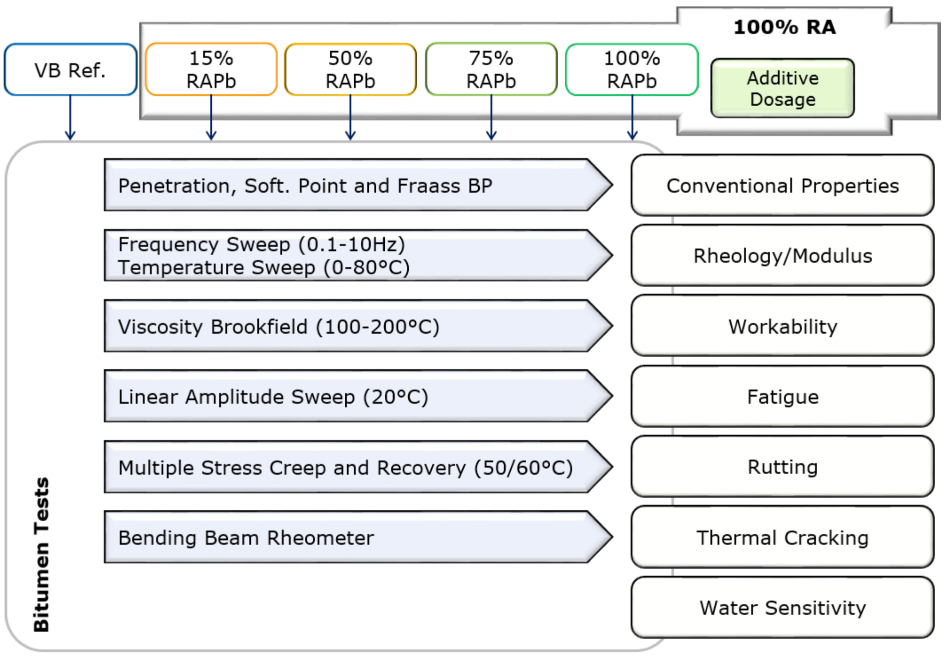


Figure 70: Binders – Experimental programme

5.2. Binders design and properties

5.2.1. Introduction: DoBA selection

The proposed methodology to understand and measure the bitumen activation from RA for new asphalt mixtures showed promising results. The outcomes can be used as a tool to better understand the RA in order to improve the binder and mixture design. In this sense, the DoBA investigation (Chapter 4) was completed in this research and all the results used to drawn the chart in Figure 71. The chart presents the DoBA from RA1 according to the conditioning procedures adopted and the different parameters investigated:

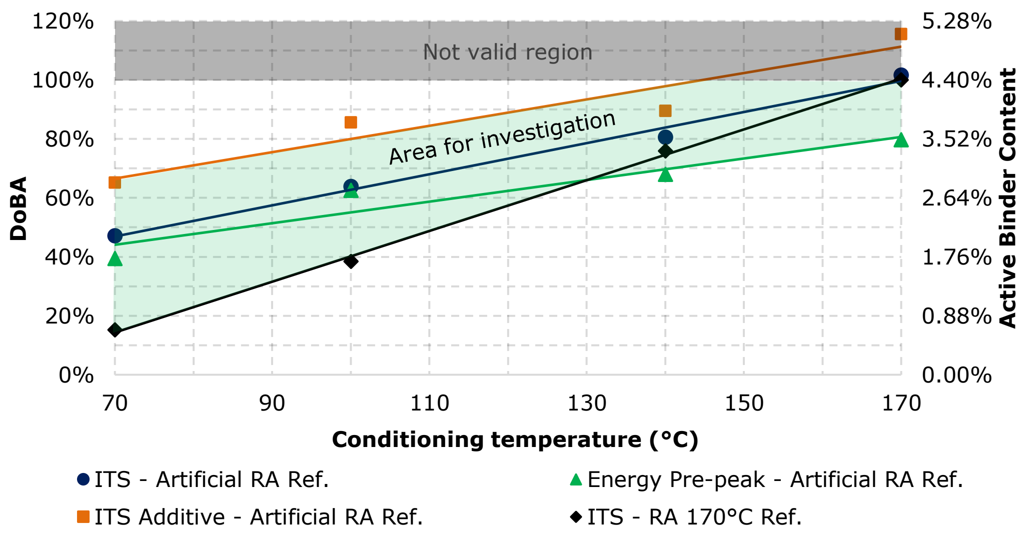


Figure 71: DoBA estimation for RA1 – Final chart

Figure 71 was used to select four different DoBA percentages to produce RA mixtures containing 100% RA to test their mechanical performance:

<u>**Case I** – 100% DoBA’ at 170°C</u>: maximum value found for the ITS parameter, although other parameters have been analysed, the desire to use only ITS values prevailed for being the fastest parameter to obtain the data.

<u>**Case II** – 75% DoBA’ at 140°C</u>: analysing all the results at 140°C, the DoBA’ found was in a range of 60-90%, then the midpoint 75% was chosen, also because of the

ITS values in both DoBA' cases (Artificial RA and $RA_{170°C}$ as references) being very close to this scenario;

Case III – 50% DoBA' at 70°C: the first case for the 70°C conditioning production, as at this temperature the wider range of DoBA was found (15-65%), 50% DoBA' was selected due to the analysis with the artificial RA1 in terms of ITS and Energy Pre-peak;

Case IV – 15% DoBA" at 70°C: 15% DoBA" was selected for being the lowest value found in the proposed methodology, in this situation using the $RA1_{170°C}$ ITS as a reference.

5.2.2. Optimal additive dosage

The penetration and SP tests were used in order to optimise the dosage of additive in the RA1 binder. In this sense, two percentages by weight of the rejuvenator were added to the RA1 binder and hand mixed to determine the conventional properties. The percentage values were chosen according to manufacturer's guidelines and laboratory trials as 6 and 8%. The results of the tests on the RA1 binder and the two rejuvenated binders are presented in Table 22:

Table 22: RA1 + Add. – penetration and SP dosage

Binder	Penetration 25°C (dmm)	Softening Point (°C)
RA 1	16.3	64.9
RA 1 + 6% Add.	47	54.1
RA 1 + 8% Add.	64	50.1
VB 50/70	64	47.6

From the results can be seen that adding 8% of additive, results in properties are closer to the VB and match the specifications for both penetration and SP. For the dosage, two graphs have been tailored, one for each property (Penetration and SP). The graphs are presented below in Figure 72:

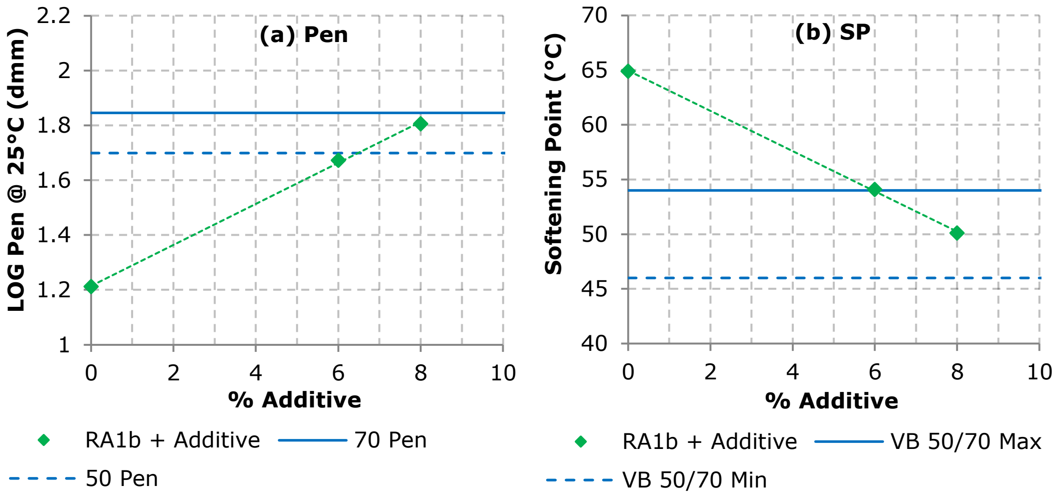


Figure 72: Additive dosage – Penetration (a); Softening Point (b)

Furthermore, with the outcomes presented in Figure 72, Table 22 presents the dosage limits for each property applying the equations and having the specification parameters as reference. The results show the penetration law results in a lower required dosage than the amount determined by the softening point law. However, regardless of the dosage found, both values are within the limits for the two properties studied based on the requirements for a 50/70 bitumen. In this sense, the optimum dosage adopted for this research was 7.5% which represents a binder with 60 dmm in penetration, which is the midpoint of the specification. Furthermore, considering the midpoint of the softening point, 8.1% additive was considered very close to the maximum amount of additive for 70 dmm penetration, which is one of the limits.

Table 22: RA1 + Add. – Penetration and SP dosage

	Penetration		**Softening Point**	
Limit (min-max)	50 dmm	70 dmm	46°C	54°C
Additive	6.5%	8.4%	10.3%	5.9%
Target midpoint	7.5%		8.1%	

Therefore 7.5% of additive was added to the recovered RA1 binder in order to obtain the final binder properties. The results are presented in Table 23 (including outcomes with 6% and 8% of additive):

Table 23: Additive dosage – conventional properties

Binder	Penetration 25°C (dmm)	Softening Point (°C)	Fraass BP (°C)	Viscosity at 135°C (Pa.s)	Asphaltenes content (%)
RA1 binder	16.3	64.9	-3	0.836	21.30
RA1 + 6% Add.	47	54.1	-12	0.590	-
RA1 + 7.5% Add.	**60**	**51.4**	**-14**	**0.495**	**18.15**
RA1 + 8% Add.	64	50.1	-17	0.479	-
VB 50/70	64	47.6	-8	0.281	15.76

From the results it can be seen that if the percentage of additive increases, the penetration value increases while the SP, the Fraass breaking point and the viscosity decrease. These are the effects of the additive leading to a softer material. Whereas for the penetration and SP it is possible to reach the target level, for the other properties some considerations can be made:

- The Fraass breaking point values are lower than the value of the virgin bitumen, meaning that the rejuvenated binder is less brittle and behaves better at low temperatures, leading to an improvement in the thermal cracking resistance.

- Rotational viscosity results (Figure 73) present an improvement after the rejuvenation, but not enough to reach the reference bitumen. This property could be reached if a dosage methodology based on viscosity was adopted, and consequently, a higher amount of the additive would be required. In contrast, permanent deformation problems could occur due to a softer binder, limited here by the softening point results.

- The asphaltenes fraction tends to rise with ageing, while the contribution of the additive helped to reduce the asphaltenes amount. Despite this decrease, the percentages used in this study were not able to reduce asphaltene content to the target level. The addition of 7.5% of added agent caused the initial difference (RA1 and VB) to be reduced by about 50%. Values here are used only as a comparison with the VB, since chemical components can exhibit entirely different proportions in materials from different origins. Similar outcomes were found by Lehtimäki

(2012), while the fundamental properties were recovered by the rejuvenator used, the chemical properties were distant from the target.

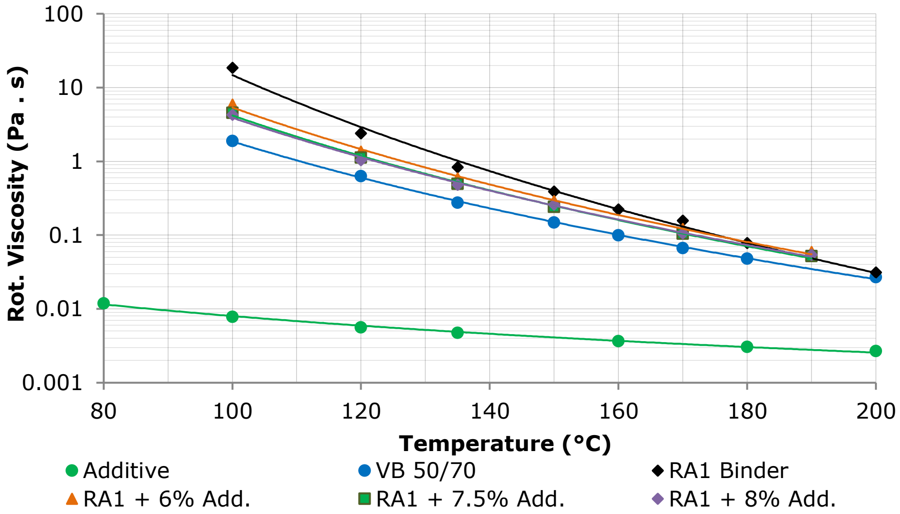


Figure 73: Additive dosage – Final viscosities

According to the DoBA results and the calculated percentages of released binder for the present material RA1, four values were chosen for the sequence of the research activities. These values were equal to 100%DoBA′, 75% DoBA′, 50% DoBA′ and 15%DoBA″. All the binders were prepared in the laboratory and their properties tested with results presented in the subsequent sections. The recycled binders are:

- **RA1+7.5% Add$_{\mathbf{(100\%DoBA')}}$**: RA1 binder + 7.5% Add by weight of RA1 binder active;

- **RA1+7.5% Add$_{\mathbf{(75\%DoBA')}}$**: RA1 binder + 7.5% Add by weight of RA1 binder active + Virgin Binder. This binder consists of 75% RA binder treated with the additive and 25% of virgin binder VB 50/70.

- **RA1+7.5% Add$_{\mathbf{(50\%DoBA')}}$**: RA1 binder + 7.5% Add by weight of RA1 binder active + Virgin Binder. This binder consists of 50% RA binder treated with the additive and 50% of virgin binder VB 50/70.

- **RA1+7.5% Add$_{\mathbf{(15\%DoBA'')}}$**: RA1 binder + 7.5% Add by weight of RA1 binder active + Virgin Binder. This binder consists of 15% RA binder treated with the additive and 85% of virgin binder VB 50/70.

5.2.3. Conventional properties

The conventional properties of the binders are presented in Table 24. Taking the results of the VB 50/70 penetration grade bitumen as a reference for a standard virgin binder in asphalt mixtures, it can be observed that the extracted RA binder comes from an aged source showing low penetration, high softening point and high Fraass breaking point. On the other hand, the recycled binder with 7.5% of additive (100%DoBA) has its conventional properties recovered to values similar to the VB 50/70 or even improved, such as for Fraass Breaking Point where the cracking temperature is considerably reduced.

Table 24: Binders conventional properties

Binder	Name	Penetration 25°C (dmm)	Softening Point (°C)	Fraass BP (°C)
RA1 + 7.5% Add. (100% DoBA’)	100%DoBA’	60.3	51.4	-14.0
RA1 + 7.5% Add. (75% DoBA’)	75%DoBA’	65.4	49.4	-13.0
RA1 + 7.5% Add. (50% DoBA’)	50%DoBA’	62.3	48.4	-12.0
RA1 + 7.5% Add. (15% DoBA”)	15%DoBA”	61.0	48.0	-9.0
Virgin Binder 50/70	VB 50/70	64.3	48.0	-8.0
RA1 Binder	RA1b	16.3	64.9	-3.0

Regarding the other recycled binders (75%DoBA’, 50%DoBA’ and 15%DoBA”), it is important to highlight that all three blends are different proportions of the RA1 binder + 7.5% Add. (100% DoBA’) mixed with the VB 50/70. In this sense, the conventional properties were expected to be between the values for those two binders, the boundaries. Therefore, it can be seen that the results are proportionally increasing or decreasing, depending on the property, to the closest value of the bitumen with 100% DoBA or the VB 50/70.

Figure 74 displays the results of the viscosity of the binders at different temperatures. RA1b shows higher viscosity when compared to VB 50/70 and recycled binders from 100°C up to 170°C after which the differences become less evident. In the case of the RA1b, the high viscosity has to be attributed to its aged state and hard consistency. As for the previous conventional properties, there is a gradual modification in viscosity from VB 50/70 up to the recycled binder 100%DoBA, with the three binders in intermediate positions. Moreover, it is important to highlight that the recycled binders meet the limits suggested by the Superpave specifications (AASHTO MP-1, 1998), with a maximum viscosity of 3 Pa.s at 135°C.

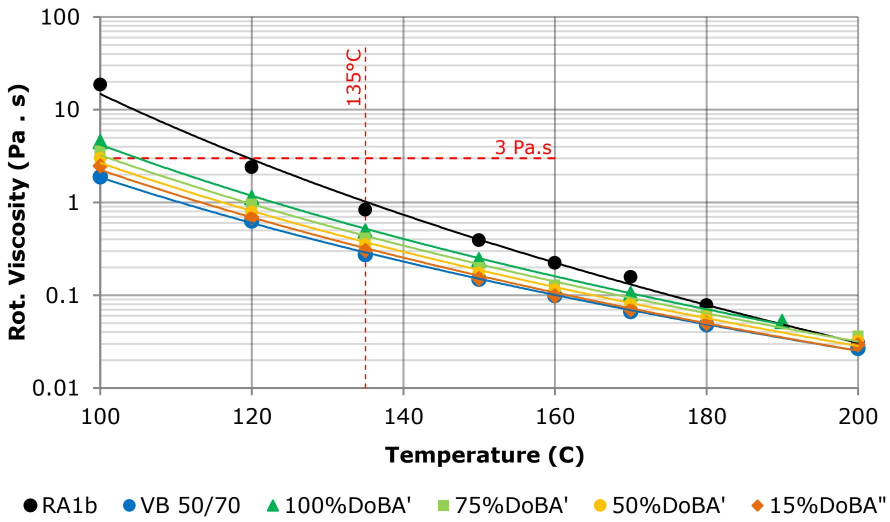


Figure 74: Recycled binders – Viscosities

5.2.4. Rheological properties

5.2.4.1. Dynamic Mechanical Analysis (DMA)

It is important to evaluate the rheology and viscoelastic properties of the final recycled binders under different loading times and temperatures. This has been achieved using DMA to define the stress-strain-time-temperature response of the binders. The rheological properties have been determined using both the DSR and the BBR. Master curves of complex shear modulus |G*| and phase angle δ at a reference temperature of 25°C were produced through manual adjustment of shift factors. Figure 75 shows the complex modulus master curves while Figure 76 shows the phase angle data.

RA1b shows the highest complex modulus from intermediate to high temperatures. Both RA1b and VB 50/70 bitumens present similar behaviour at low temperatures with slightly higher values for the RA1b. In particular, comparing recycled binders with the VB 50/70, it can be seen that at low temperatures (high frequencies) the binders have lower complex modulus and higher phase angle meaning that the material is less brittle and more viscous, so less susceptible to thermal cracking. This is similar to the Fraass BP results presented in Table 24 and is clearly effect of the rejuvenation due to the additive used. The rejuvenation effect can be observed in Figure 75 and the highlighted region, related to low temperatures, that shows the G* reduction from VB 50/70 to the 100% DoBA' binder (with the highest amount of additive in its composition).

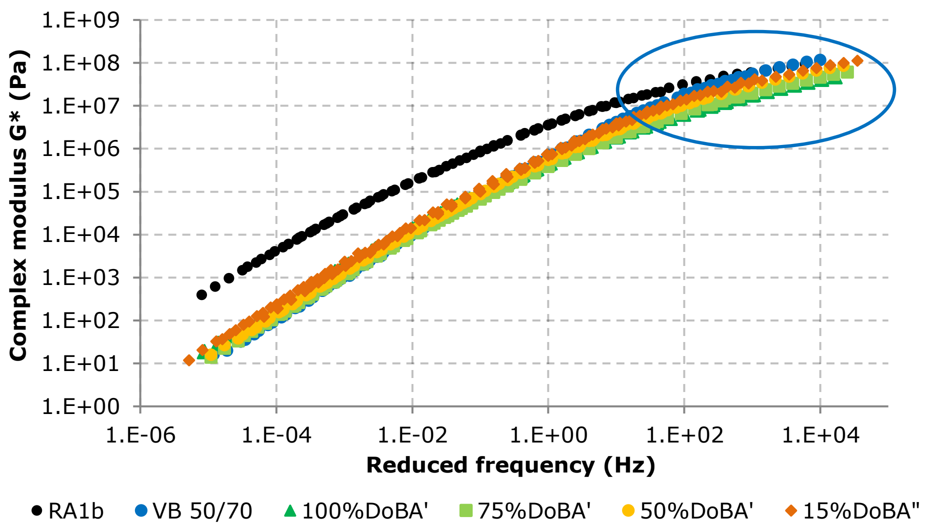


Figure 755: Recycled binders – |G*| master curves at 25°C

At high temperatures, the binders are slightly stiffer than the VB 50/70, although they do show the good rejuvenating capacity of the additive used. Moreover, at intermediate temperatures, the treated binders have lower stiffness than VB 50/70.

With the results, it can be said that besides recovering the RAb1, the additive has also changed the slope of the G* master curves, consequently improving the performance in the tested temperature range. Despite this, the great performance gain is evident at low temperatures, in much greater proportions than at intermediate and high temperatures.

However, considering phase angle in Figure 76, the elastic properties of the recycled bitumens increases with the increase of RA1b content in their composition. Also, the VB 50/70 presented higher viscous behaviour between all bitumens, and in contrast to the G* and binders stiffness, the additive was not capable of reducing the elasticity to the reference bitumen level (mainly at intermediate temperatures).

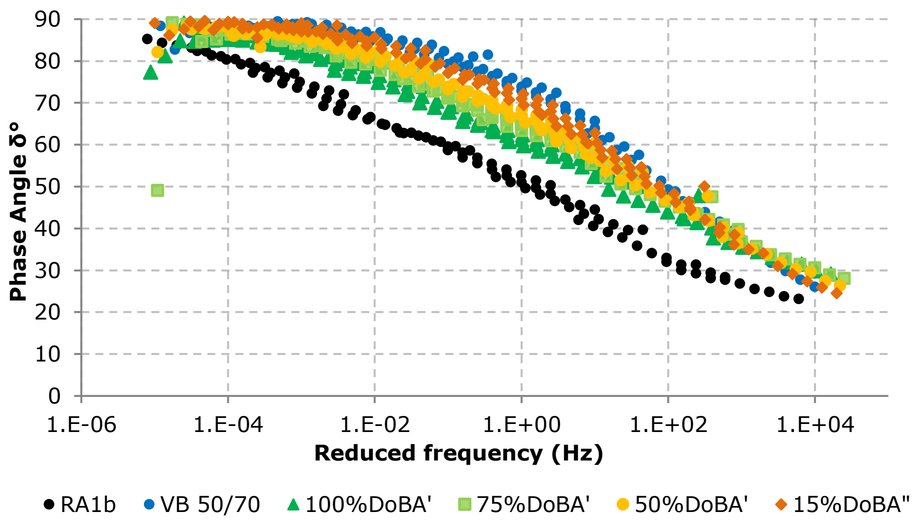


Figure 76: Recycled binders – Phase angle δ master curves at 25°C

Figure 77 shows the Black diagram of the binders. These diagrams can be used to show the relation between stiffness and viscoelasticity of materials without the need to apply shift factors to the raw data as required for master curves (Airey 2002). In the Black space diagram, high modulus values represent the results at low temperature and vice versa.

All binders exhibit thermorheologically simple behaviour over the range of frequencies and temperatures tested since their curves in the Black diagram smoothly overlap for the different temperatures and frequencies. A smooth curve in a Black diagram is a useful indicator of time-temperature equivalency, in this sense, the diagram indicates that all bitumens do not have any sign of modification by waxes or polymers as expected (at the range of temperatures tested).

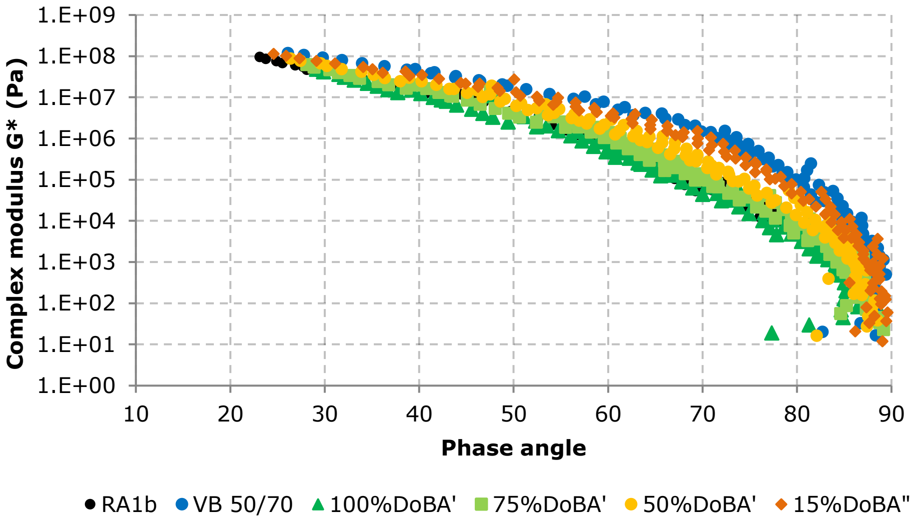


Figure 77: Recycled binders – Black space diagrams

5.2.4.2. Ageing

The results of the frequency and temperature sweeps of the RTFOT and RTFOT+PAV aged binders are shown in terms of master curves in the form of the G* and phase angle at a reference temperature of 25ºC in Figure 788, Figure 79, Figure 80 and Figure 81. As reported in Section 3.2.2, the ageing of non-modified binders produces an increase in their complex modulus and decrease of phase angle (Lu & Isacsson 2002). This behaviour is reflected in the subsequent figures.

The results show that the VB 50/70 and the recycled binders gradually change their rheology with progressive ageing becoming stiffer and more elastic. The most explicit method of observing this is by comparing the aged curves with the curves of the recycled binders and the RA1b. It is evident that the master curves are positioned closer to RA1b after ageing.

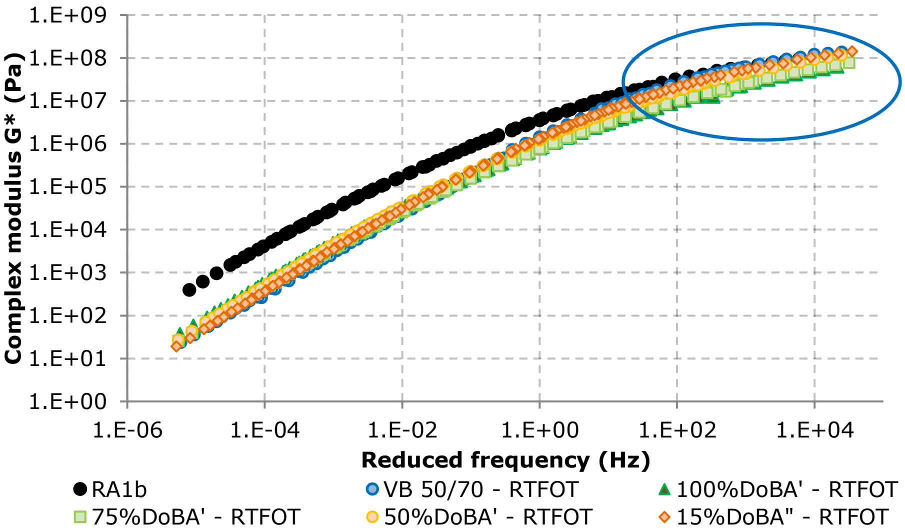


Figure 78: Recycled binders – RTFOT aged – |G*| master curves at 25°C

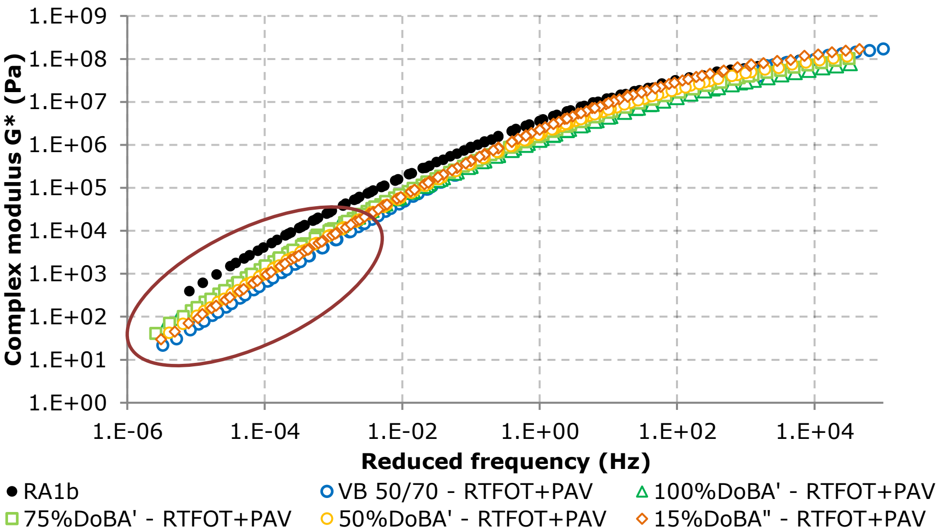


Figure 79: Recycled binders – RTFOT+PAV aged – |G*| master curves at 25°C

Regarding the performance at different temperature (low, intermediate and high), can be observed in Figure 788 the rejuvenation effect of the additive and the RA binders showing lower G* than VB 50/70 after ageing. However, in Figure 79 can be observed an effect not evident in unaged materials (Figure 75), the recycled binders are positioned closer to the RA1b than the VB 50/70, likely due to the temperature susceptibility of these binders with higher amounts of RA1b (consequently more additive) in their composition. Despite this and by the fact that recycled binders have the G* lower than RA1b, RA binders are still stiffer than VB 50/70 (translating into better performance under high temperatures).

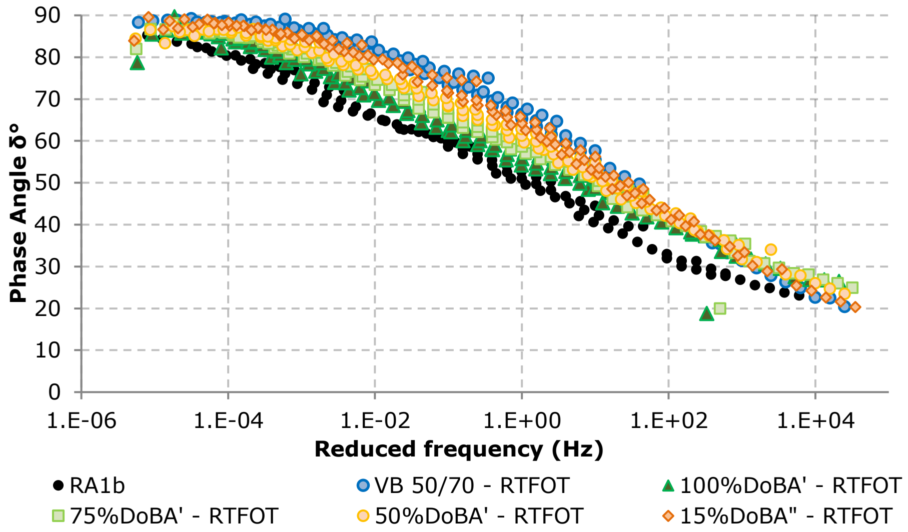


Figure 80: Recycled binders – RTFOT aged – δ° master curves at 25°C

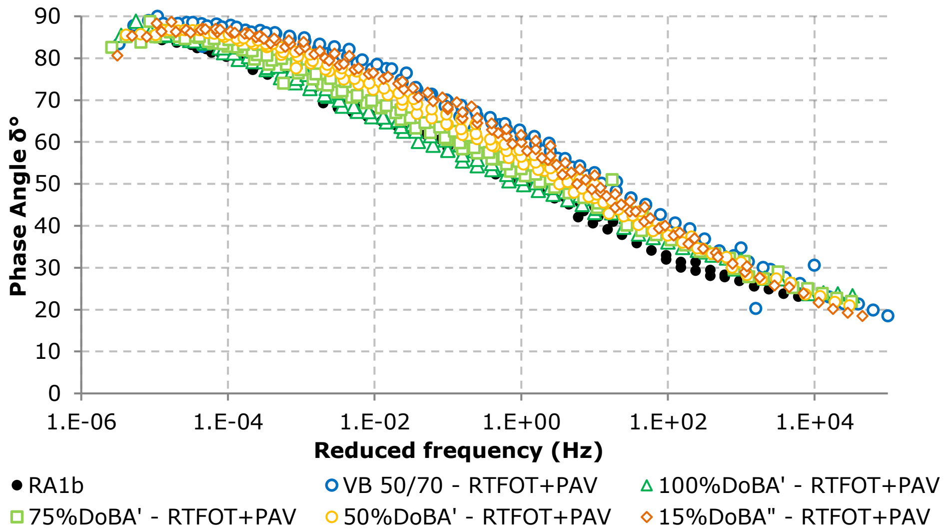


Figure 81: Recycled binders – RTFOT+PAV aged – δ° master curves at 25°C

In summary, although the recycled binders present slightly higher stiffness and less viscous properties, after ageing, this does not imply future issues in a pavement layer (if the materials were applied in field). Since without showing major weaknesses due to the ageing processes, recycled binders could provide satisfactory performance (considering G* and δ°) to the pavement.

5.2.4.3. Low temperature cracking - BBR

In addition to the DSR analysis, the BBR results (Figure 82) indicate the low temperatures behaviour of the recycled binders. The BBR results were used to obtain the low critical temperatures of stiffness ($Tc_{(Low)S}$) and m-value ($Tc_{(Low)m}$), calculated as presented in Section 2.3.1.2 with results in Table 25. The BBR test temperatures were selected based on the frequency sweep test results and the Standard specification for PG asphalt binder specification (ASTM D6373, 2016), conducted on the RTFOT+PAV aged binders.

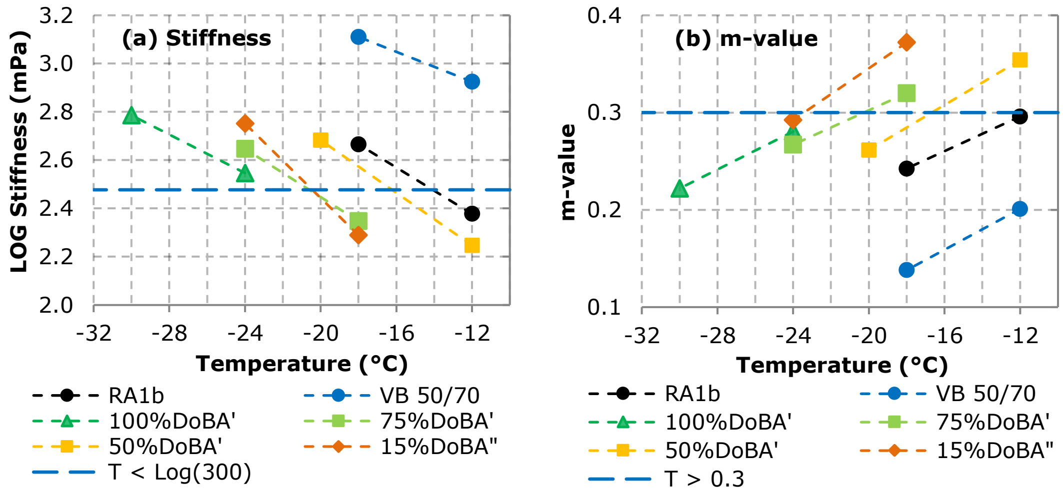


Figure 82: BBR – (a) Stiffness; (b) m-value

Table 25: Binders BBR results

Binder	Test temperature (°C)	Stiffness (MPa)	$Tc_{(Low)S}$ (C°)	m-value	$Tc_{(Low)m}$ (C°)
100%DoBA’	-24	352.0	-22.3	0.281	-22.0
	-30	610.5		0.222	
75%DoBA’	-18	222.5	-20.6	0.320	-20.3
	-24	443.5		0.267	
50%DoBA’	-12	176.5	-18.2	0.354	-18.7
	-20	481.5		0.262	
15%DoBA”	-18	194.5	-15.3	0.372	-19.2
	-24	564.5		0.292	
VB 50/70	-12	841.0	-9.5	0.201	-14.6
	-18	1290.0		0.138	
RA1b	-12	238.5	-14.1	0.296	-11.6
	-18	463.0		0.243	

5.2.5. Critical temperatures

Considering BBR and DSR results, the critical temperatures (high, intermediate and low) were calculated for the studied binders (Table 26):

Table 26: Designed binders critical temperatures

Binder	Tc_{High} (°C)	Tc_{Int} (°C)	Tc_{Low} (°C)	PG*
RA1b	80.7	24.1	-11.5	76-16
100%DoBA'	68.3	14.7	-22.0	64-28
75%DoBA'	66.2	17.3	-20.3	64-28
50%DoBA'	67.1	20.9	-18.2	64-28
15%DoBA"	65.0	21.3	-15.3	64-22
VB 50/70	66.8	19.4	-9.5	64-16

* ASTM D6373-16

The critical temperatures, plotted against the effects of ageing and rejuvenation on the binders, decrease with the additive (see Figure 83).

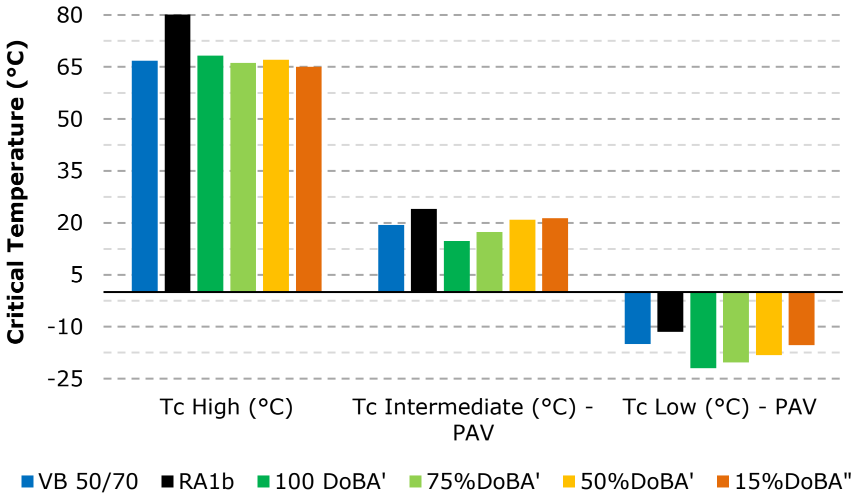


Figure 83: Designed critical temperatures

In summary, the RA1b, recycled binders and control binders have been characterised by means of conventional tests, rheology, ageing and critical temperatures. The RA1b has been shown to be a significantly aged binder with hard consistency, high stiffness and elastic components. It exhibits high values for the critical temperatures, which is interpreted as good resistance to rutting while poor fatigue and thermal cracking performance. These properties reveal the need for rejuvenation of this source of RA if it is to be used in high amounts in recycled asphalt mixtures.

Considering the critical temperatures, these values represent positive effects in intermediate and low temperatures, improving the fatigue and thermal cracking behaviour, however, reducing the high critical temperature weakens the rutting resistance. Furthermore, in comparison with the target VB 50/70, the additive efficiently restored the rheological properties by producing similar critical temperatures or even better than the control binder. In summary, these results mean better fatigue and thermal cracking resistance for the recycled binders. The rutting results are satisfactory compared to the VB 50/70 due to the stiffness of the RA1b binder. Translating the results in a ranking for the six binders according to the critical temperatures would be:

Tc High: RA1b > 100%DoBA’ > 50%DoBA’ > VB 50/70 > 75%DoBA’ > 15%DoBA”;

Tc Int.: 100%DoBA’ > 75%DoBA’ > VB 50/70 > 50%DoBA’ > 15%DoBA” > RA1b;

Tc Low: 100%DoBA’ > 75%DoBA’ > 50%DoBA’ > 15%DoBA” > RA1b > VB 50/70.

Thus, the percentages obtained by applying the penetration and SP laws in the additive dosage can also provide satisfactory results for the rheological properties. Further performance-related tests were carried out in order to understand the behaviour of those binders related to field applications.

5.2.6. Binders blend design and mixtures inputs

Once the conventional and rheological properties of the binders were obtained, the blend design for the RA1b and VB 50/70 binders was carried out. This section shows the process and results of the blend design, as the results are then used as inputs for the asphalt mixtures design. The first part of the blend design is the determination of the Replaced Virgin Binder (%RVB) for the recycled binders. Using the method shown in Section 3.2.5.3, the RVB (%) has been defined by assigning different values at each parameter for each recycled binder (and future mixtures).

To calculate the RVB (%), Equation 45 was used with the following variables:

- **Total percentage of RA to be added in the asphalt mixture ($RA_{in\ the\ mix.}$) (%):** Since the asphalt mixtures should have high RA content in order to investigate the DoBA, the RA content for the binders blend design and further asphalt mixtures is 100%;

- **Bitumen content in the RA ($RA_{bit.content}$) (%):** the RA1 characteristics obtained in Chapter 4 – Section 4.1.1 were applied. Therefore, the RA bitumen content to be used is 4.4%;

- **Bitumen content in the mixture ($MIX_{bit.content}$) (%):** as the binder content from the RA1 is key to the study, 4.4% was selected plus the amount of additive for the highest DoBA scenario (100%). In this sense, the 7.5% by weight of additive in the binder represents 0.33% of the total binder, giving a final 4.73% binder content;

- **DoBA (%):** given that the real percentage of RA binder that is reactivated in the mixtures is the aim in this thesis, and considering the results already presented in this chapter, the DoBA percentages investigated were 100, 75, 50 and 15%;

- ***Final bitumen content of the mixture – Full binder ($MIX_{FULLbit.content}$) (%):** as the investigated DoBA will be applied for the mixtures production, the remaining amount of RA bitumen is considered inactive. However, as this is a matter of DoBA investigation only and the black rock effect is another assumption, the $MIX_{FULLbit.content}$ is also presented for comparison considering the whole amount of bitumen from the RA1. In summary, this case consists of RA1b treated with the additive according to DoBA%, the VB 50/70 and RA1b considered inactive.

Table 27 presents the results:

Table 27: Binder content, moisture content and maximum density

	VB 50/70 - Control Virgin Mixture	**RA1b - Control 100% RA**	**100% DoBA′**	**75% DoBA′**	**50% DoBA′**	**15% DoBA″**
1) $RA_{in\ the\ mix.}$ (%)	0	100	100	100	100	100
2) $RA_{bit.content}$ (%)	0	4.4	4.4	4.4	4.4	4.4
3) $MIX_{bit.content}$ (%)	4.73	4.4	4.73	4.73	4.73	4.73
4) DoBA (%)	0	100	100	75	50	15
5) RVB (%)	0	100	100	75	50	15
6) Active RA_{bit} $^{(2 \times 4)}$ (%)	0	4.4	4.4	3.3	2.2	0.66
7) Inactive RA_{bit} $^{(2 - 6)}$ (%)	0	0	0	1.1	2.2	3.74
8) Active $RA_{bit+Add.}$ $_{(6 \times Add.dosage\ 7.5\%)}$ (%)	0	4.4	4.73	3.55	2.37	0.71
9) VB 50/70 added $^{(3 - 8)}$ (%)	4.73	0	0	1.18	2.37	4.02
10) $MIX_{FULLbit.content}$ $^{(7 + 8 + 9)}$ (%)	4.73	4.4	4.73	5.83	6.93	8.47

Having established these variables, it is important to analyse them. The three initials parameters were defined for mixture design purposes and could not be any different. In this sense, both the DoBA and RVB have the same values because the design was carried out considering 100% RA in the mixture. The virgin binder replaced by RA binder should be the maximum RA binder content. However, as the objective is to investigate the DoBA, it was necessary to reject the inactive bitumen portion in the calculation. Moreover, the final binder content in the mixture is similar to the total bitumen amount from the RA, thus, resulting in the same value for RVB and DoBA.

In addition to the necessary results for the binders blend design, the values (6 – 9 in Table 27) represent the details to be used in the mixtures, differentiating the bitumen contents from inactive to active bitumen treated with the additive. The last variable (10) represents the total amount of bitumen that the mixtures will have (basically adding the amount of inactive bitumen in the total weight). This shows that when the DoBA% decreases, the amount of VB 50/70 should be added in order to complete the amount of active binder in the mixture. Also, it is essential to highlight that the mixtures production will have differences in their production that affect the DoBA%, as already presented in Chapter 4. Therefore, the results from Table 27 show the link between binder design, mixture design and the DoBA. Further details of mixtures production are presented in the next Chapter 6.

5.3. Performance-related tests on binders

Further verification of the preliminary design consisted in subjecting the recycled binders to performance-related tests over the whole service temperature range of pavements. The performance-related properties have been investigated at each level of ageing (neat, RTFOT and PAV) for the "RA1b", recycled binders and the virgin bitumen. These tests were carried out to determine whether the preliminary design and the additive dosage were able to obtain binders showing similar potential in terms of rutting, fatigue and thermal cracking resistance.

5.3.1. Rutting-related properties

In order to evaluate rutting resistance of binders, Multiple Stress Creep Recovery (MSCR) tests have been carried out. This test (BS EN 16659:2015) assesses the binder's ability to recover deformation, and consequently, their potential resistance to rutting in the mixture.

The test has been carried out at two different stress levels (0.1 kPa and 3.2 kPa) at 60°C. The temperature selected was the nearest to the high critical temperatures of the studied binders (65°C on average). The test data outputs allow values of Recovery (%) and Non-Recoverable Creep Compliance (Jnr) to be determined.

The graphs shown in Figure 84 and Figure 85 show the accumulated strain for the binders during the test for RTFOT and RTFOT+PAV aged binders respectively. The results display a higher accumulated strain for the softer materials while the stiffer

RA1b can accumulate less strain. The graphs show that the 15%DoBA binder is actually softer than the VB 50/70 at both levels of ageing.

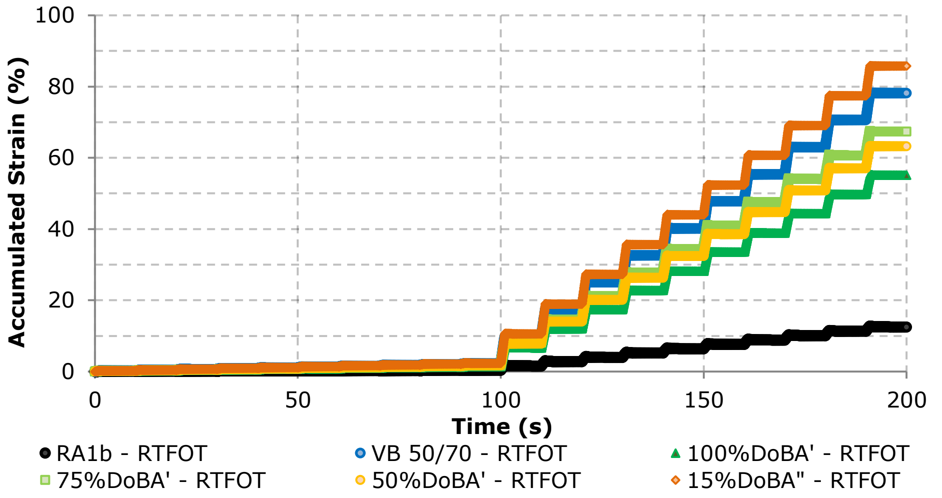


Figure 84: Accumulated strain during MSCRT at 60°C – RTFOT aged binders

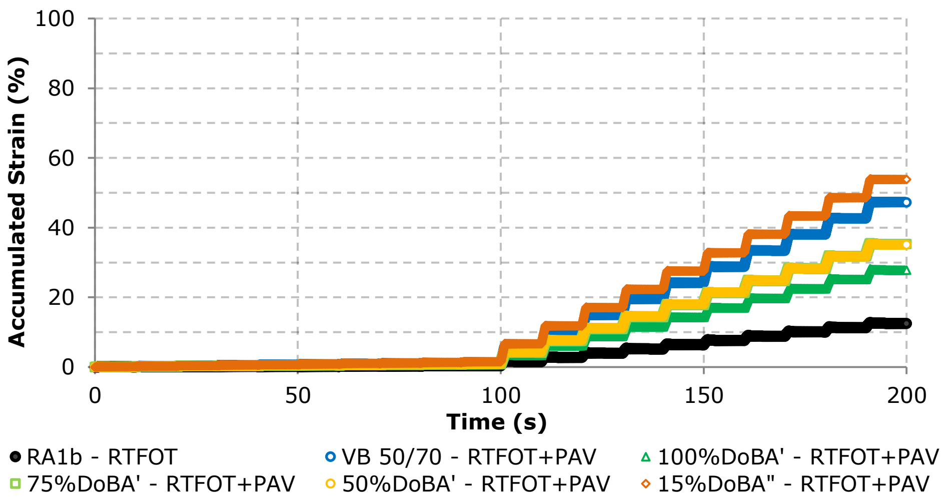


Figure 85: Accumulated strain during MSCRT at 60°C – RTFOT+PAV aged binders

The results above are confirmed by the two MSCR fundamental parameters: % Recovery (R(%)) and Non-recoverable compliance (Jnr). These two parameters are used to characterise the rutting resistance and have been defined following the procedure described in detail in Section 2.4.2.1. Figure 86 shows the R(%) for all the binders, and, Figure 87, the Jnr results at 3.2 kPa.

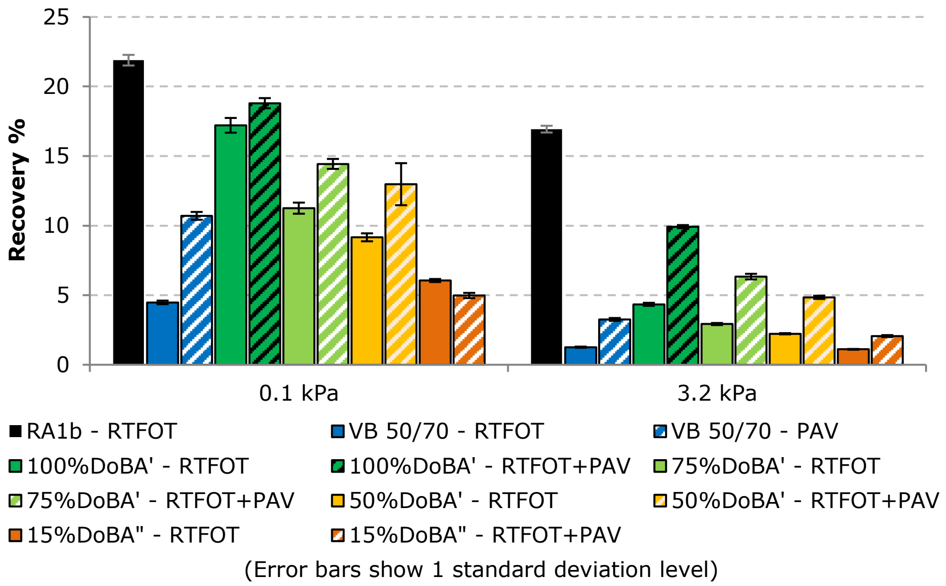


Figure 86: MSCR - 60°C – Recovery %

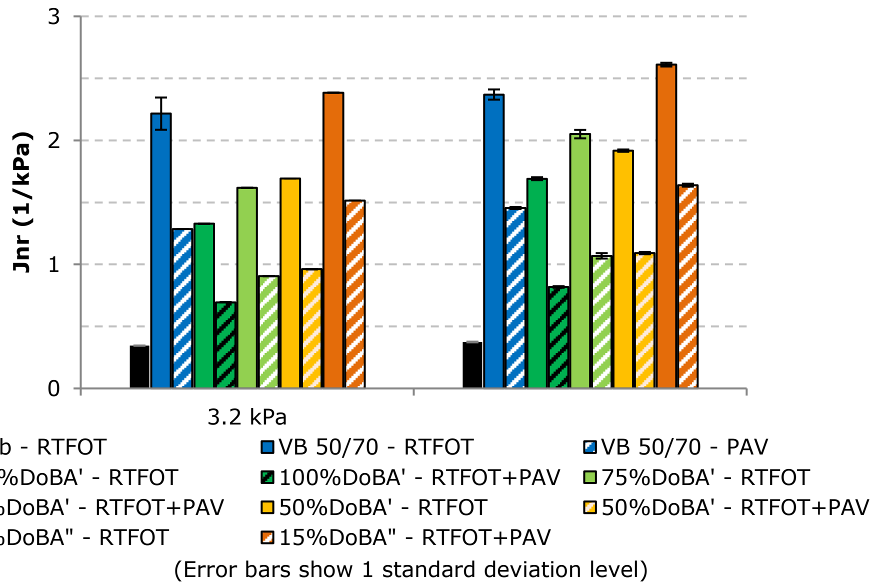


Figure 87: MSCR - 60°C – Jnr

The first noticeable result is the high R(%) and low Jnr that the RA1b exhibits in comparison to a conventional 50/70 penetration grade binder. This fact may lead to the conclusion that the RA1b is highly flexible and elastic. However, if the strain that the binders experience during the test is analysed (Figure 85), the significant difference in deformation between the RA1b and the VB 50/70 reveals the RA1b high recovery is because of the low strain that it experiences due to its hard consistency. Hence, a small amount of strain recovered is translated into a high R(%), on the contrary, the VB 50/70 presents high strains and is only able to recover 5%.

Translating into rutting resistance, RA1b indicates better performance, likely due to the higher stiffness commonly found in RA materials (Al-Qadi *et al.*, 2012; Zaumanis, Mallick and Frank, 2014b). However, the presence of additive decreases this resistance, thus, requiring attention when subjected to high temperatures. The analysis detailing critical temperatures had already shown this behaviour when the additive was used (Section 5.2.4). However, the MSCR test shows that the differences in critical temperatures are not as significant as their magnitude represents. Considering the R(%) results from recycled binders, the 15%DoBA presents the closest value to the VB 50/50 as already showed by the accumulated strain. The other recycled binders show reductions in their R(%) due to the presence of the additive together with the VB 50/70, where the R(%) experienced higher reductions in R(%) according to the higher amount of virgin bitumen.

In addition, the Jnr results in Figure 88 support the analysis that this parameter, obtained by dividing the non-recoverable strain by the applied stress, is a rutting potential indicator. The higher Jnr is, higher is the rutting susceptibility of the binder. According to D'Angelo (2010), the existing SHRP binder specification is based on measurements in the linear viscoelastic range and for most neat binders the Jnr results behave linearly usually up to the 3.2 kPa stress level. In this sense, and in accordance with the specifications, the Jnr at 3.2 kPa can provide a good correlation to the performance. Moreover, using the graph contained in the standard AASHTO T350 (2014) to evaluate the elastomeric response, further analysis can be made from Figure 88.

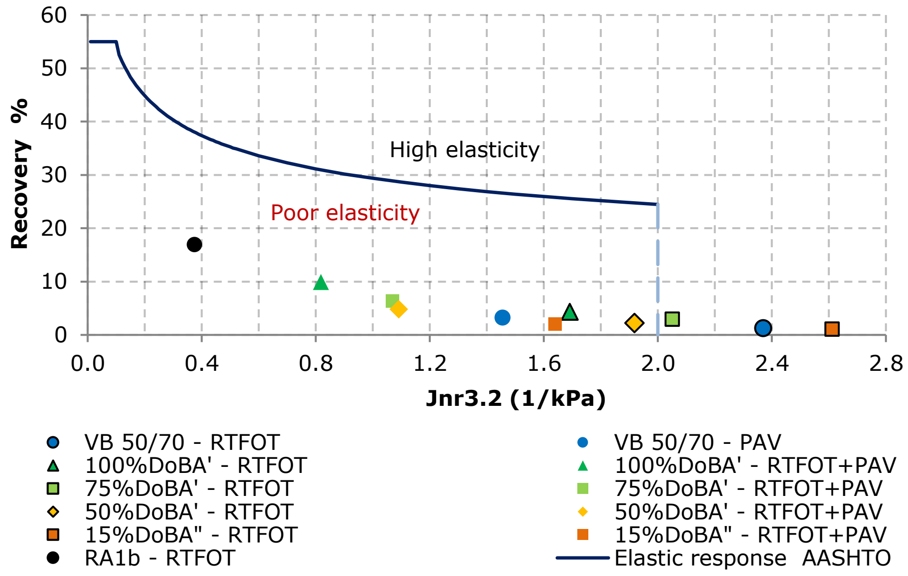


Figure 88: MSCR - 60°C – Elastic response $Jnr_{3.2kPa}$

None of the binders of the study could show satisfactory results regarding R(%), at least not at the level of a polymer modified bitumen. For this reason, all tested binders present poor elastic behaviour. However, Figure 88 is useful to show that the stiffer binder (RA1b) has higher recovery percentage and lower Jnr, while the recycled binders and the use of additive show the opposite. The simulated laboratory PAV ageing increases the R(%) and decreases the Jnr but does not change the recycled binders behaviour. Furthermore, in accordance with the suggestion by the standard AASHTO T350 (2014), the R(%) analysis is not a necessary requisite when the Jnr is higher than 2 kPa^{-1}, therefore, it can be seen that the VB 50/70 and the recycled binder (75%DoBA') do not need the elastic response analysis but still the rutting performance on mixtures should be investigated. Finally, the binders are ranked based on R(%) and Jnr at a test temperature of 60°C as follows:

MSCR: RA1b > 100%DoBA' > 75%DoBA' > 50%DoBA' > VB 50/70 > 15%DoBA"

5.3.2. Fatigue-related properties

Linear Amplitude Sweep (LAS) test has been carried out at 20°C following the standard AASHTO TP101 (2014) on RTFOT and RTFOT+PAV aged bitumens. The test was run, using the DSR, in two steps: a frequency sweep (from 0.1 Hz to 30 Hz with a strain level of 0.1%) followed by an amplitude sweep (at 10 Hz with a linear increase of strain from 0.1% to 30%). Then, the Viscoelastic Continuum Damage (VECD) analysis was carried out according to the procedures in Section 2.4.2.2 and, three parameters were taken into account to evaluate the fatigue resistance: Damage at failure, parameter α and the traffic volume indicator.

5.3.2.1. Damage at failure and Parameter Alpha (α)

The damage at failure (D_f) is defined as the level of damage accumulated before failure, and it is an indicator proposed by the standard AASHTO TP101 (2014). The results in Figure 89 (RTFOT binders) and Figure 90 (RTFOT+PAV binders) show the accumulated damage during the test against the loss modulus (G*.sin(δ)).

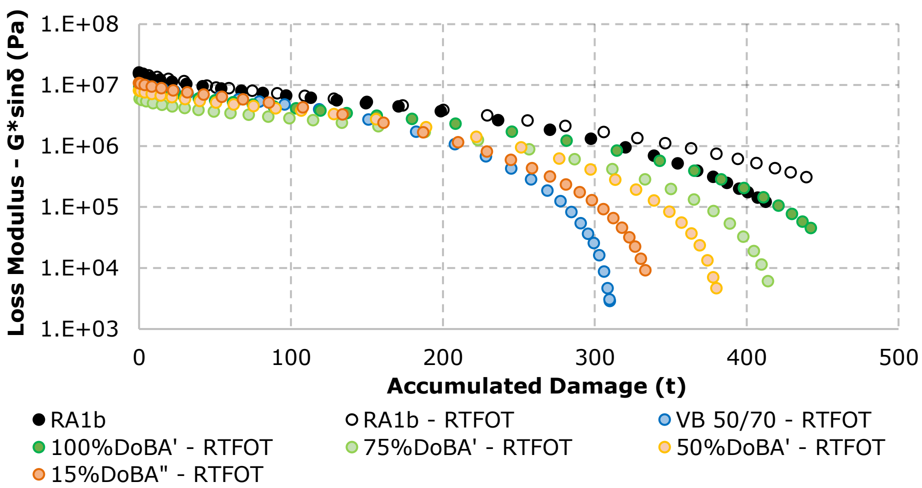


Figure 89: LAS - 20°C – Damage accumulation for RTFOT aged binders

From the results it can be seen that the RA1b binder can accumulate more damage than the VB 50/70. An opposite trend was expected because of aged RA binders are usually more susceptible to fatigue. On the other hand, the recycled binders

100%DoBA′ and 75%DoBA′ were able to accumulate more damage than RA1b, improving the performance according to those parameters. The binders 50%DoBA′ and 15%DoBA″ were also able to improve their resistance to fatigue in comparison to the control bitumen.

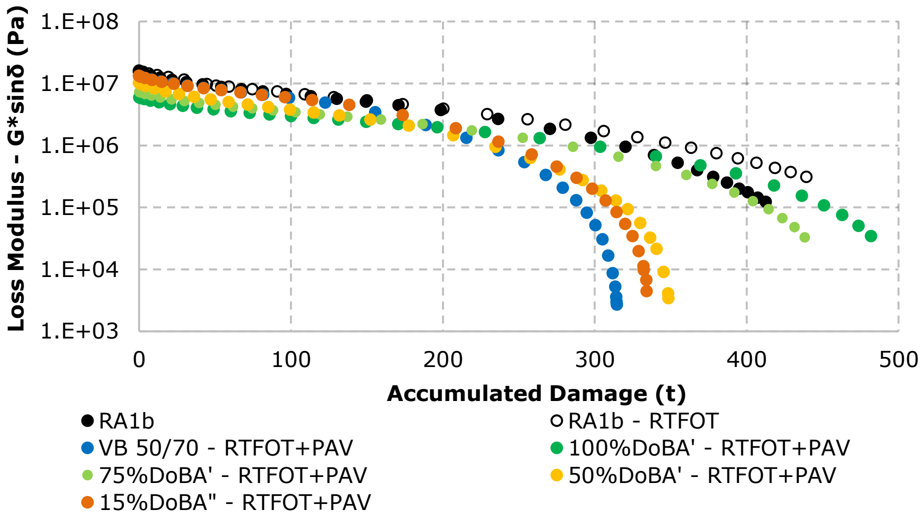


Figure 90: LAS - 20°C – Damage accumulation for RTFOT+PAV aged binders

Comparing both graphs (Figures 89 and 90) it is possible to analyse the binders susceptibility to ageing, where some of them (15%DoBA″ and 50%DoBA′) were able to accumulate more damage after RTFOT ageing, but, reduced their capacity after PAV. However, the 75%DoBA′ and 100%DoBA′ binders improved their resistance after PAV ageing, meaning that in this case, the RA1b was possibly leading the performance during the test.

Although the recycled binders seem to perform better than the VB 50/70, further analysis is required regarding the D_f and parameter α. In order to provide a better view of the phenomenon, Figure 91 was created.

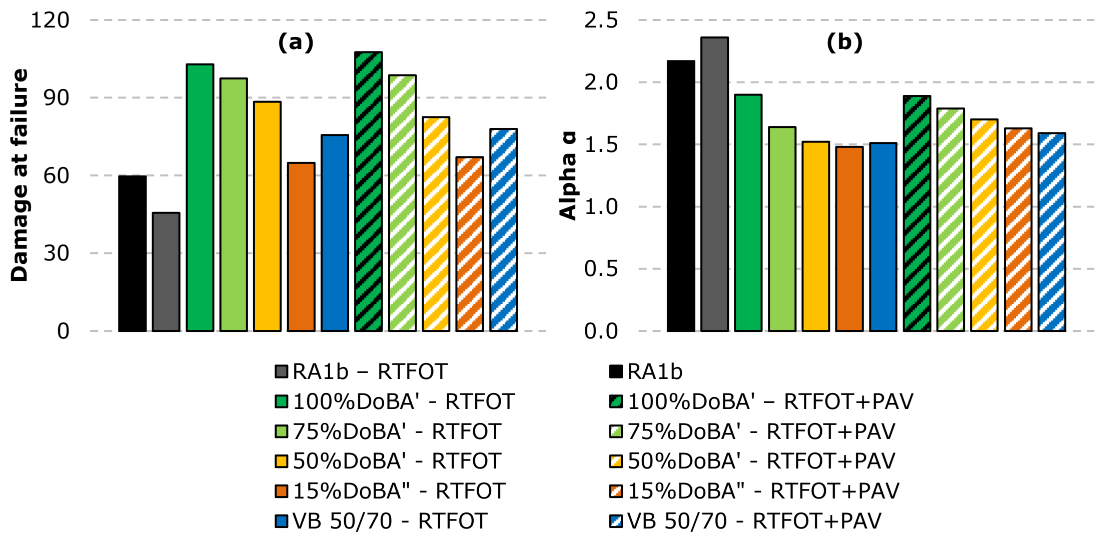


Figure 91: LAS – 20°C - Damage parameters

Considering each parameter, some notes can be made. The higher values of D_f mean that the material is capable of accumulating more damage before failure. The Parameter α is linked to the slope of the fatigue live curves and higher values represent less resistance to fatigue. Moreover, the primary analysis should be based on RTFOT+PAV aged bitumens.

- Damage at failure (Figure 90a): this parameter is in contrast to the results in Figure 90 where the RA1b showed high capacity to accumulate damage. This proves that the Df parameter is more reliable to use than the total damage accumulated, as it is based in the failure criteria (50% G* reduction). The best results were experienced by the recycled binders 100%DoBA' and 75%DoBA', where the fatigue resistance improved and showed higher capacity to accumulate damage. Thus, the ability to accumulate damage (before failure) is strongly linked to fatigue resistance, then, the total damage until the rupture means a longer fatigue life.

- Parameter α (Figure 91b): as an indicator of the slope of the fatigue life curves, the highest value (lower fatigue resistance) is in accordance to the D_f, the RA1b presents the lowest fatigue resistance. However, the RA1b is followed by the recycled binders and, lastly the VB 50/70. This would represent that VB 50/70 has the best fatigue performance, but this was not experienced when the D_f was analysed. In this sense, becomes essential to analyse the fatigue life curves in order to better understand the importance of their slope (parameter α). Therefore, further analysis is presented in the next section regarding the number of fatigue cycles and the traffic volume indicator.

5.3.2.2. Traffic volume indicator (Nf)

The N_f has been calculated using the procedures presented in Section 2.4.2.2. This parameter provides the number of equivalent single axes loads (ESALs) that a pavement can withstand depending on the applied strain. The results are presented in Figure 92 for the RTFOT aged binders and Figure 93 for the RTFOT+PAV aged.

The graphs in both figures show a particular trend for the RA1b binder, where it can support more ESALs than the other materials at low applied strain levels, but as the applied strain increases, the VB 50/70 and the recycled binders improve their performance. Comparing the recycled binders with the VB 50/70, the recycled ones are shown to support a higher number of axes load. For this, the slope and position of the curves are shifted to the right (because of the additive), maintaining the RA1 behaviour at low strain levels and increasing at higher strains. However, although there is a small difference between the recycled binders and the control, it is possible to affirm that the additive is able to restore the fatigue resistance properties up to the desired target level, including all the laboratory ageing procedures undertaken. The same trend for these curves is noticed after both the short and long-term ageing, emphasising that RTFOT+PAV aged binders are used to analyse fatigue resistance.

Also, the critical temperatures present a similar trend as those found from the LAS test. According to the Superpave methodology, the recycled binders have lower values of intermediate critical temperatures, meaning better fatigue performance than the VB 50/70 (see Figure 83). Therefore, the first conclusion is that both methods seem to be well correlated, however, the LAS test provides extra parameters and improve the investigation in a range of strains that result in fatigue laws able to show the bitumen's stress-strain sensibility.

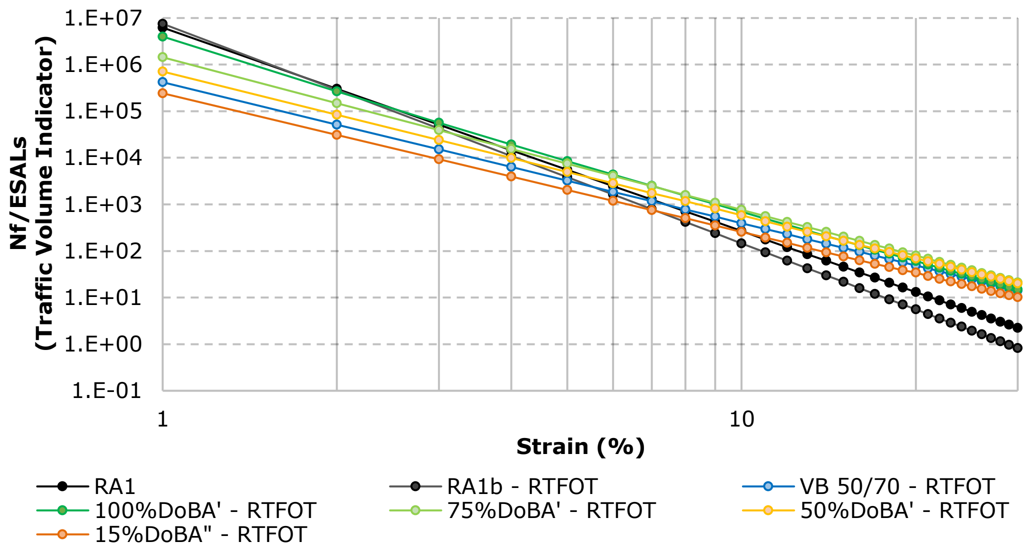


Figure 92: LAS - 20°C – N_f for RTFOT aged binders

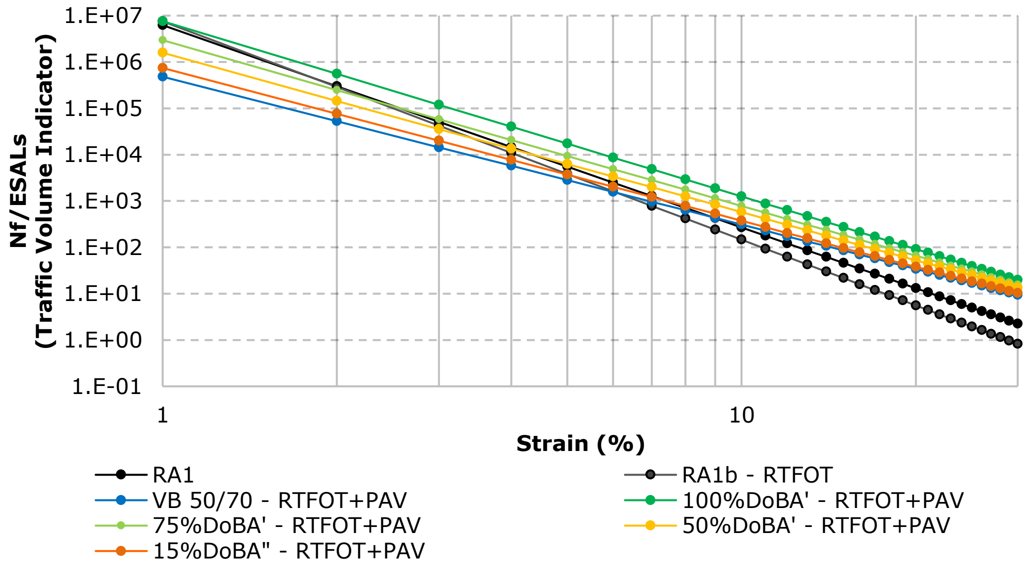


Figure 93: LAS - 20°C – N_f for RTFOT+PAV aged binders

The differences between the recycled binders, 100%DoBA' and 75%DoBA' for example, are relatively low and, in this sense, the mechanical testing on mixtures becomes even more important in order to identify more significant differences.

Therefore, the conditioning of the RA1 and the manufacturing process of mixtures should be performed in a way to support the DoBA understanding. Finally, the binders are ranked based on LAS parameters at a test temperature of 20°C as follows:

LAS: 100%DoBA’ > 75%DoBA’ > 50%DoBA’ > VB 50/70 > 15%DoBA” > RA1b

5.3.3. Thermal cracking-related properties

In order to characterise the thermal cracking resistance of binders at low temperature, the flexural creep stiffness (S, in MPa) and relaxation capacity (m-value) were obtained using the BBR as presented previously. The low critical temperatures were obtained by linear extrapolation following the procedure presented in Section 2.3.1.2 using the results from Section 5.2.4 with ΔT_c being calculated according to Equation (29) in Section 2.4.2.4.

When analysing the test results, if the creep stiffness is too high, the bitumen will behave in a brittle manner and is more likely to be susceptible to cracking. To prevent this cracking, the ΔT_c can also be analysed according to Anderson *et al.* (2011) using the differential between both critical temperatures determined using the Stiffness and m-value from BBR results. The results are shown in Figure 94.

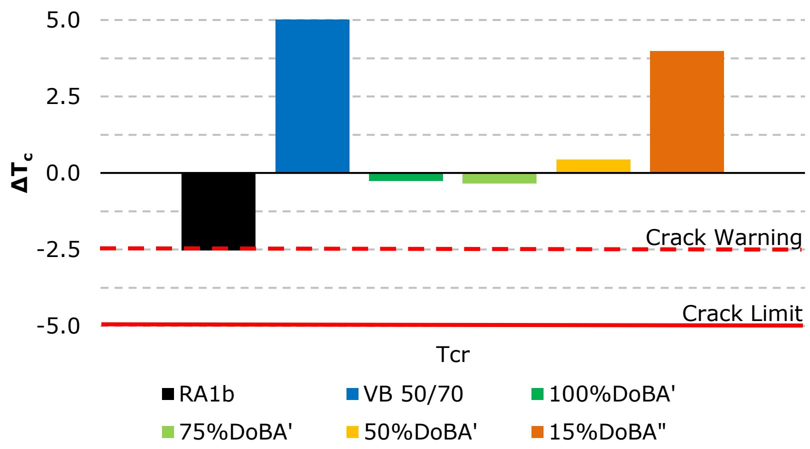


Figure 94: ΔTc of binders and cracking limits

The ΔT_c results show the poor resistance to thermal cracking of the aged RA1b. This is in accordance to what researchers have previously shown (Anderson, 2016; Hanz *et al.*, 2017) that as a bitumen ages, ΔT_c value becomes negative representing a loss of relaxation properties. This indicates that ΔT_c is an important parameter related to the bitumen durability. The results in Figure 94 show that for the recycled binders, the influence of the additive causes the values to be close to zero. This means that there is a better balance between S and m-value dependency for these binders. The higher ΔT_c values indicate improved relaxation capacity. Although the differential is not enough to reach the capacity presented by VB 50/70, except for the recycled binder 15%DoBA”, the values close to zero for the other bitumens is desirable. These results represent an equilibrium between the properties with the applied additive dosage. In the case of 15%DoBA” can be said that the significant amount of VB 50/70 dominated the material during the test. Therefore, the additive dosage together with the high capacity of the VB 50/70, in terms of thermal cracking resistance, have delivered great improvements on recycled binders, strengthening the material property. Lastly, the binders are ranked based on thermal cracking parameters as follows:

ΔT_c: VB 50/70 > 15%DoBA” > 50%DoBA’ = 75%DoBA’ = 100%DoBA’ > RA1b

5.3.4. Cracking and ageing parameters

5.3.4.1. Rheological Index and Crossover Frequency

As already presented, the Rheological Index (R-value) is a useful indicator of the rheology type of binders, observed through complex modulus master curves. As defined by Rowe *et al.* (2016), the ageing of binders produces a change in the shape of their master curves that can be translated into the change of their R-value and crossover frequency. The R-value decreases if the master curve becomes flatter, the main effect of the ageing, and the crossover frequency also decreases with the increase of ageing. In this regard, ageing reduces crossover frequencies and increases R-values while rejuvenation should have the opposite effect**.** In order to put together the results of all the materials, their rheological data were simplified in terms of R-values and crossover frequencies and plotted in Figure 95 to show the relative ageing and rejuvenating effect of the binders.

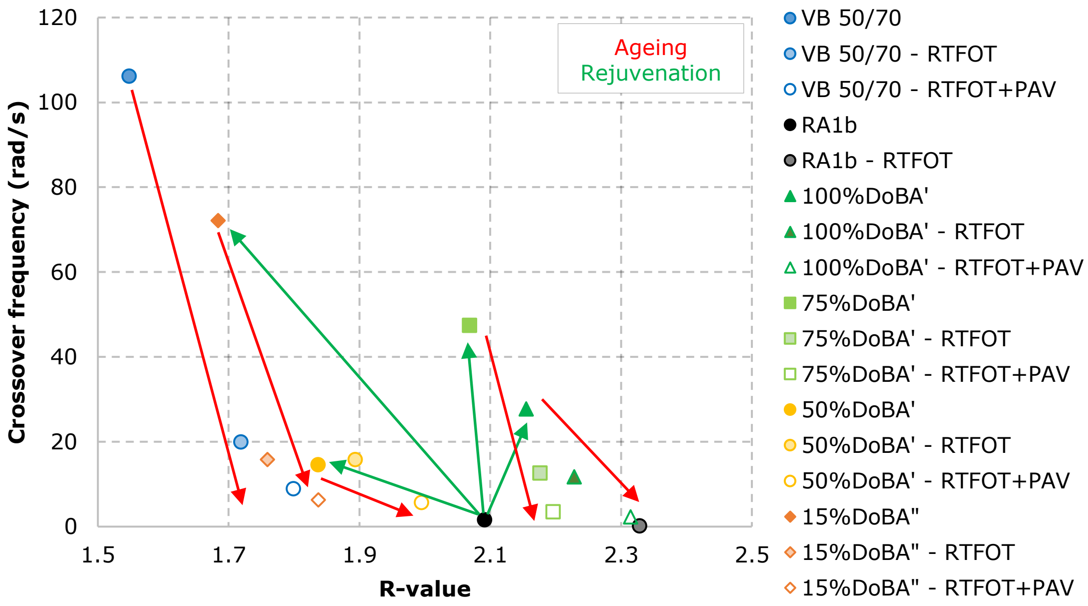


Figure 95: R-value vs Crossover frequency of recycled binders

According to the results, can be seen that the RA1b binder acts as the most aged binder being in the lowest and furthest right position. The effect of the blend of the RA1b binder with the additive is interpreted as rejuvenation and was previously shown by other parameters and properties. After the ageing of the recycled binders, all materials move down and to the right. However, the rejuvenating effect of the additive on 100%DoBA’ and 75%DoBA’ could only increase the crossover frequency, without affecting the R-value.

As suggested by Mensching *et al.* (2016) and Rowe *et al.* (2016), aging should result in an increase in R-value, reflecting a wider relaxation range, a flattened G* master curve, and a decrease in crossover frequency, meaning that a higher portion of the master curve is in the elastic behaviour region (delay of viscous behaviour). Work on rejuvenated binders show the opposite trend. In this sense, it can be said that the rheological parameters were not fully recovered by the additive as previously analysed by the master curves. The remaining two recycled binders (50%DoBA′ and 15%DoBA″) presented results relative to the amount of VB 50/70 in their composition, with lower R-value and higher crossover frequency likely because of the virgin binder properties dominating the binders during the analysis. According to Mensching *et al.* (2016), it is believed that changes in rheological properties can be directly attributed to changes in cracking resistance. However, it is currently difficult to understand how a minor numerical difference in R-value translates to field performance other than an apparent degradation due to a change in relaxation response of the bitumen.

In summary, the recycled binders presented improvements regarding R-value and crossover frequency by showing the rejuvenating effect because of both the additive and VB 50/70 in their composition. However, with higher amounts of RA1b treated with the additive, the rejuvenation process does not seem to recover the entire rheological properties. Moreover, it is essential to mention the ageing affecting these binders, presenting a rheological index inferior to the original RA1b, for example in the case of 100%DoBA′ RTFOT+PAV delivering the lowest crossover frequency and highest R-value. Therefore, 100%DoBA′ and 75%DoBA′ binders require higher attention regarding ageing and consequently cracking, where through the rheological index analysis those binders presented properties which can be decisive in the pavement life cycle.

5.3.4.2. Glover-Rowe

The Glover–Rowe (G-R) parameter can be used to assess the cracking resistance of bitumens as already explained in Section 2.4.2.3. The cracking potential of the recycled binders was analysed using the G-R parameter to further understand the influence that ageing could have on their performance-related properties. Regarding this, researchers (Mensching *et al.*, 2016; Rahbar-Rastegar *et al.*, 2017) have shown that the ageing of bitumens tends to move the initial point towards the cracking limits. In the same way, rejuvenation should produce the opposite effect. These movements are shown and indicated with red and green arrows in Figure 96.

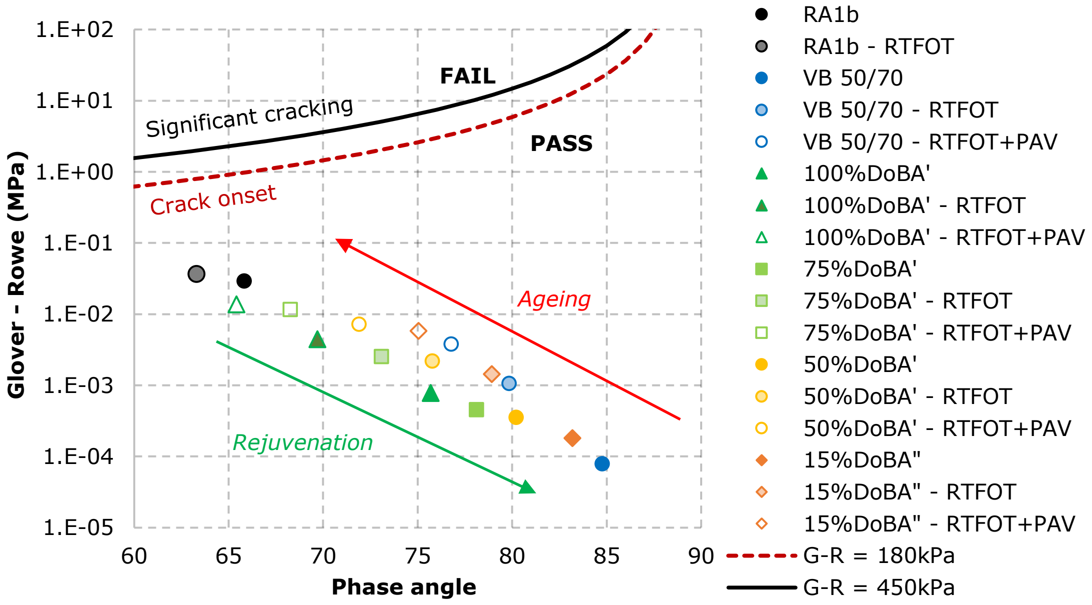


Figure 96: Glover - Rowe parameter of recycled binders

Figure 96 shows the G* and phase angle values for the recycled binders at a frequency of 0.005 rad/s at 15°C, plotted in Black Space along with the G-R curves that correspond to the G-R value of 180 kPa (onset of cracking zone) and the solid line corresponds to the G-R value of 450 kPa (significant cracking zone). From the results can be seen that all binders would have no cracking related issues according to this analysis. Also, recycled binders and VB 50/70 showed expected behaviour with ageing, moving towards the cracking criteria but without reaching the thresholds. As presented for other properties, the additive effect on the RA1b binder and the VB 50/70 in the recycled binder’s composition have affected the rejuvenation in different proportions. The control binder shows more viscous behaviour with the results positioned to the right towards higher phase angles. The opposite is true for RA1b which shows the lowest phase angle that means more elastic behaviour. In this sense, the recycled binders were positioned between these two extremes (RA1b and VB 50/70). Therefore, the cracking potential of the recycled binders was assessed by means of the G-R parameter with all of them in the “pass” area even after long-term ageing.

5.4. Summary and conclusions

According to the DoBA results presented and the estimate percentages of released binder for the RA1, four values were chosen for the investigation, these values equal to 100%, 75% and 50% DoBA’ and 15%DoBA”. All the binders were prepared in the laboratory and their properties tested with the following conclusions:

The optimal additive dosage carried out was based on penetration and softening point tests. Due to the differences found in the final properties and the similarity with the control bitumen VB 50/70, the content of 7.5% of additive was chosen to recover the RA1 binder properties. Then, the four recycled binders had their conventional and rheological properties determined, showing satisfactory results in all cases within limits indicated by the European standards for bitumen specification. Particularly considering rheological properties, the recycling process and the additive dosage were able to deliver final bitumens with considerable improvements by reducing stiffness and elasticity.

Critical temperatures were determined using the rheological measurements from DSR and the BBR, after short and long-term ageing. These temperatures were developed to connect the properties with the performance of the bitumens, relating them with the most common types of pavement failures (rutting, fatigue and thermal cracking). The results showed that recycled binders were able to provide similar or better performance than the control binder VB 50/70. In summary, similar rutting resistance, similar to better fatigue resistance (depending on the DoBA%) and better thermal cracking resistance in all recycled cases.

In order to support the conventional and rheological properties, performance-related tests were also run. Different from the critical temperatures analysis, the recycled binders presented variations when submitted to those performance tests. The DoBA% was affected by the results, but not necessarily because of the RA1 binder, but due to the amount of VB 50/70 present in each recycled binder. Translating the results into rankings, no trend was observed in the performance of the binders as it was detected in conventional properties, possibly because of the amount of virgin binder and its characteristics dominating some of the tests. However, the general performances of the binders were positive and promising, supporting the good binder design carried out as well as the additive dosage adopted. In this sense, the performance-related tests showed to be necessary for bitumen analysis, when some properties and behaviours cannot be identified through conventional tests.

Finally, the binders blend design was performed focusing on recycled asphalt mixtures. The DoBA% was considered together with the RA content in the future mixtures, equal to 100% RA. The necessity to use 100% RA is because of the low number of variables to be considered during the mixtures production, such as virgin aggregates and their interaction with the RA1b and VB 50/70. Thus, with the binders properties determined as well as their design linked to the 100% RA mixtures, this chapter's outcomes provide the necessary inputs for the mixtures production and analysis for the final step in the DoBA investigation.

6. RECYCLED MIXTURES

6.1. Introduction

One of the issues related to using RA from the industry is the variability which includes not only the gradation of RA but also the binder content and material origin. Whenever RA is acquired from industry, its properties may be different and for this reason it is important to have all the RA characteristics for the mixture design.

Recycled mixtures have been successfully used in HMA in addition to other applications such as Warm and Cold mixtures. Regarding HMA, the RA has been incorporated by various highway agencies and researchers in different mixture designs such as dense graded, open graded and gap graded as presented in the literature review. Due to the high content of RA chosen in this research, 100% RA, the mixture design is dependent on the RA grading curve to be classified according to the mixture grade types. In this sense, the results of the RA characterisation are significant for the sequence of the study.

Regarding asphalt mixtures, the laboratory investigations are important to understanding and then reliably predicting the performance of recycled mixtures in full-scale field applications. Once the blend design was performed for the recycled binders and the final binder content was defined, mixtures production and testing began.

Regarding the recycled mixtures, according to the DoBA study (Chapter 4) and results adopted for the binders investigation (Chapter 5), four recycled mixtures were produced, assuming 100%DoBA′, 75%DoBA′, 50%DoBA′ and 15%DoBA″. All the four mixtures were produced containing 100%RA, with the DoBA% related to the binders design and the active RA binder according to the conditioning processes (temperature and mixing time). Therefore, part of the RA binder was assumed be black rock (for DoBA% less than 100%) and was compensated in the mixtures by adding the virgin binder (to replace the part of the RA binder that was assumed to be inactive). In this sense, some mixtures have high amounts of bitumen although part of this bitumen is supposed to be inactive. Two control mixtures were also produced for comparison: Control virgin and Control 100% RA. Therefore, the present chapter includes the following investigation according to Figure 97.

This chapter aims to present a brief description of materials and the mixtures design procedure associated with manufacturing recycled asphalt samples. It also presents the mixtures compactability, volumetric properties and mechanical properties regarding rutting, fatigue, stiffness and moisture damage. Finally, relations were identified and established between binders and mixtures using the tests outcomes, in this sense, the verification of the DoBA study is carried out.

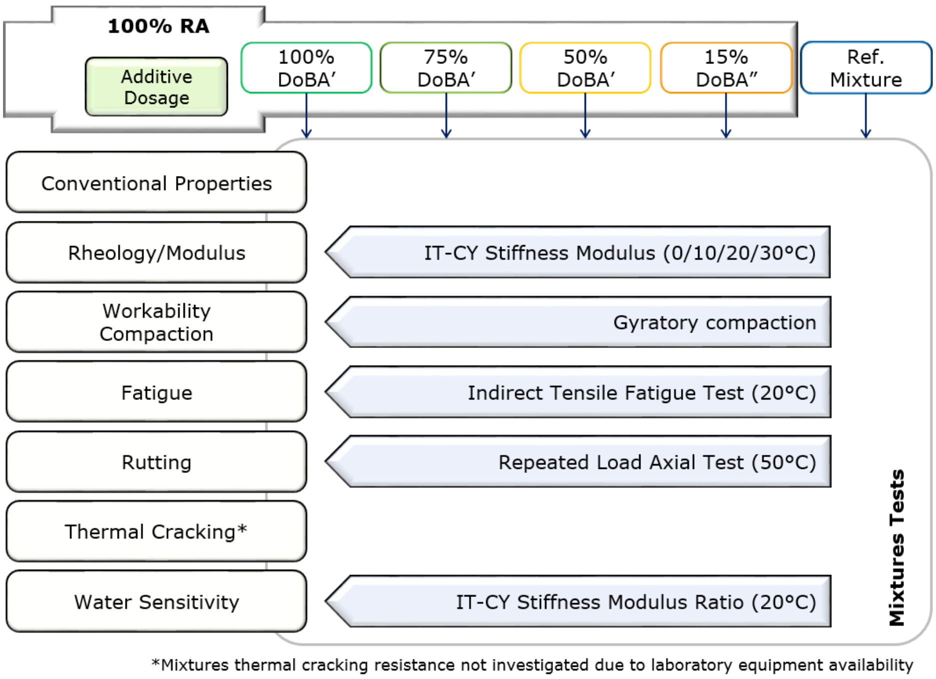


Figure 97: Mixtures – Experimental programme

6.2. Mixture design and production

6.2.1. Asphalt mixture gradation

The first step before the manufacturing of the asphalt mixtures was to define the gradation. As already mentioned in the previous chapter, the RA gradation is crucial because of the 100% RA content in the recycled mixtures. Considering the results presented in Section 4.1.1 with the RA1 material, the RA grading curve is shown in Table 28 and the final gradation is displayed in Figure 988. The control reference mixture produced with the VB 50/70 binder, using virgin aggregates detailed in Section 4.1.3 and the grading envelope for a standard Asphalt Concrete 20mm – AC 20 (EN 13108-1, 2016), 0/20mm dense binder (BS 4987-1, 2005) and the Curve DNIT B (DNIT 031 ES, 2006), are also presented.

Table 28: Mixtures – Grading curves

Sieve (mm)	Control Virgin Mixture	RA – Black Curve	RA – White Curve
31.5	100	100	100
20	99.65	99.64	100
16	92.98	93.87	91.64
14	87.72	87.74	87.31
10	77.29	67.22	74.04
8	64.92	53.72	63.78
6.3	54.40	41.92	52.89
4	41.28	26.85	41.95
2.8	37.64	20.72	36.67
2	34.88	16.03	32.75
1	26.44	9.80	27.97
0.5	19.37	4.72	23.26
0.25	14.77	1.13	15.31
0.125	11.89	0.31	10.77
0.063	10.27	0.12	8.42

When analysing the RA grading curves, the difference between the RA white and black curves can be seen. The white curve is important because it represents the original curve of the asphalt mixture, which in this case can be taken as an AC 20, and in addition could be Curve B – DNIT for surface/binder courses if the material was from Brazil. Thus, the six mixtures produced in this research used the RA White curve as a reference for the mixture design.

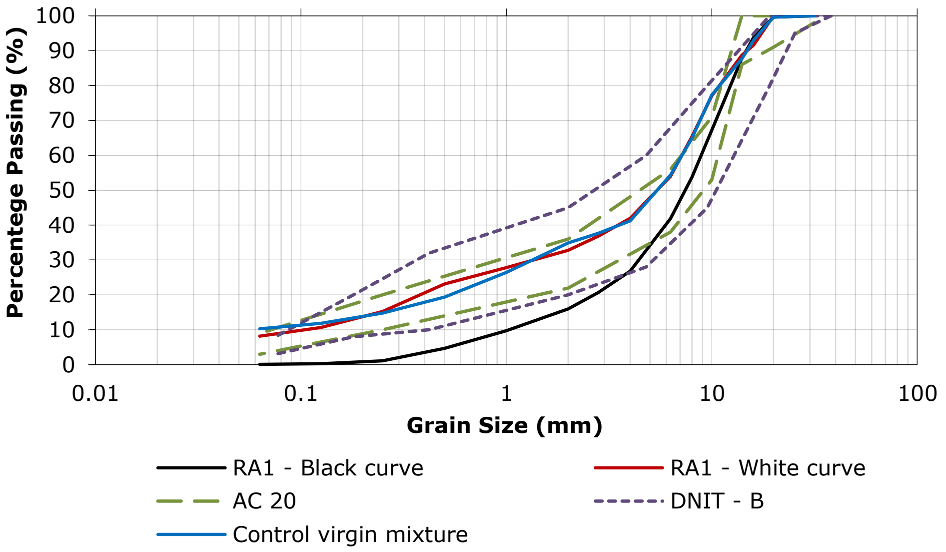


Figure 98: Mixtures - Grading curves

Although it is known that there may be differences in the effective grading curve to be considered, being positioned between RA white and RA black curves due to the active bitumen (or the black rock portion), in this research it was necessary to choose the RA white curve to reproduce the control virgin mixture. This choice was made because using the RA white curve allows the bitumen activation to be evaluated with the reference mixture representing the full mechanical properties in relation to the amount of binder. Thus, when comparing the recycled mixtures with the control one (with the RA white curve), it provides an assessment with less variability in the mechanical properties, and also, because the actual grading curve of the recycled mixtures cannot be defined with 100% accuracy.

6.2.2. Binder content

Following the previous results presented in Section 5.2.5 with the binder blend design and mixture inputs, Table 29 presents the summary of the mixtures binder contents (not considering the “inactive” bitumen).

Table 29: Mixtures – Binder content

Mixtures	$RA_{bitumen\ content}$ (%)	$RA_{in\ the\ mixture}$ (%)	Active RA_{bit} (%)	Active $RA_{bit+Add.}$ (%)	VB 50/70 (%)	$MIX_{bitumen\ content}$ (%)
Control Virgin Mixture	0	0	0	0	4.73	4.73
Control 100% RA1	4.4	100	4.40	4.40	0	4.40
100%DoBA’	4.4	100	4.40	4.73	0	4.73
75%DoBA’	4.4	100	3.30	3.55	1.18	4.73
50%DoBA’	4.4	100	2.20	2.37	2.37	4.73
15%DoBA”	4.4	100	0.66	0.71	4.02	4.73

6.2.3. Mixtures production

In order to begin the specimen production, some details were defined regarding the materials’ conditioning and mixing times. These details were based on the DoBA results presented in Chapter 4, where the RA conditioning and mixing times are critical parameters to achieve the desired and estimated DoBA for the recycled mixtures. In this sense, the manufacturing details and procedures are as follow:

RA conditioning: 4 hours in oven at 170°C, for control RA1,
4 hours in oven at 170°C, for 100%DoBA’ mixture,
4 hours in oven at 140°C, for 75%DoBA’ mixture,
4 hours in oven at 70°C, for 50%DoBA’ mixture,
4 hours in oven at 70°C, for 15% DoBA” mixtures;

Virgin aggregate conditioning: 8 hours in oven at 165°C;

Virgin binder conditioning: 3 hours in oven at 165°C;

Additive conditioning: room temperature (20°C);

Mixing temperature: 170°C, for control RA1 and 100%DoBA’ mixtures,
140°C, for 75%DoBA’ mixture,
70°C, for 50%DoBA’ and 15%DoBA” mixtures,
165°C, for control virgin mixture;

Mixing times: see Table 30;

Table 30: Mixtures – Mixing times

Mixtures	Only RA (s)	Additive added (s)	VB 50/70 added (s)	Total (s)
Control Virgin Mixture	-	-	180	180
Control 100% RA1	180	-	-	180
100%DoBA’	120	60	-	180
75%DoBA’	60	60	60	180
50%DoBA’	60	60	60	180
15%DoBA”	60	60	60	180

With the mixtures production temperatures and mixing times defined, the next procedure carried out was the determination of the maximum density of the mixtures. Considering the maximum density, the European Standard EN 12697-5 (2009) was used. The summary of the results are presented in Table 31.

Table 31: Mixtures – Maximum density

Mixtures	Maximum density (g/cm^3)
Control Virgin Mixture	2.569
Control 100% RA1	2.475
100%DoBA’	2.484
75%DoBA’	2.461
50%DoBA’	2.407
15%DoBA”	2.360

The maximum density results were determined to be used in the specimen production to target volumetric properties. The differences between each mixture can be seen in Table 31 with the Control virgin mixture having the highest max density likely due to the origin of the virgin aggregates. Analysing the recycled mixtures, compared with the Control 100% RA1, it can be observed that the density decreases with decreasing DoBA%, because of the higher amount of VB 50/70 bitumen added in the mixtures. This outcome was expected because of the inactive RA binder present in the mixtures resulting in increasing amounts of binder (inactive RAb + active RAb + VB). The low density of the binder with larger amounts to the mixtures should result in lower maximum densities as seen in Table 30.

Having the maximum density of the mixtures, the next stages undertaken were the determination of the mixtures compactability and the specimens manufacturing.

6.2.4. Gyratory compaction and compactability

As the gyratory compaction method is widely used, and due to the high productivity of test specimens, the method was selected for the compactability investigation and specimen production. The European Standard EN 12697-31 (2007) was followed for the specimen preparation using the gyratory compactor, applying 600 kPa in compaction pressure, 1.25° inclination angle and 30 gyrations per minute.

The compactability of asphalt mixture in the laboratory is one crucial factor that influences the mixtures. The way in which the mixture is compacted in the field significantly impacts the level of compaction, which is associated with the construction quality of the asphalt pavement. As already showed in Section 3.3.2.2, the compactability is used for evaluating how easy it is to compact a mixture in the field and is one aspect representing the mixtures workability. The European Standard EN 12697-10 (2002) was followed to measure the compactability of the recycled mixtures. The procedure details are given in Section 3.3.2.2 and three replicates of each mixture were used in the investigation.

The mixtures production followed the same procedures explained in the previous section. In addition, after finishing the mixing process, the mixtures were placed in gyratory moulds and into an oven at 140°C for 1 hour for temperature stabilisation. It was known that this could modify the original RA temperature, e.g. for the 50%DoBA′ and 15%DoBA″ recycled mixtures that were produced with RA at 70°C. It was understood that the conditioning at 140°C for 1 hour would not severely affect the results, since the added VB 50/70 bitumen was already at a higher temperature (165°C) and also has begun to diffuse with the RA binder at 70°C during the mixing. In the case of the other mixtures (Control mixtures, 100%DoBA′ and 75%DoBA′), no issues can be addressed as the conditioning temperatures are at a minimum the same (140°C). Therefore, choices had to be made so that they would not affect the results significantly and thereby jeopardise the DoBA investigation. At the same time a method of easy reproduction needed to be provided.

The compactability results are presented in Figure 99 with the k index determined according to the procedure showed in Section 3.3.2.2.

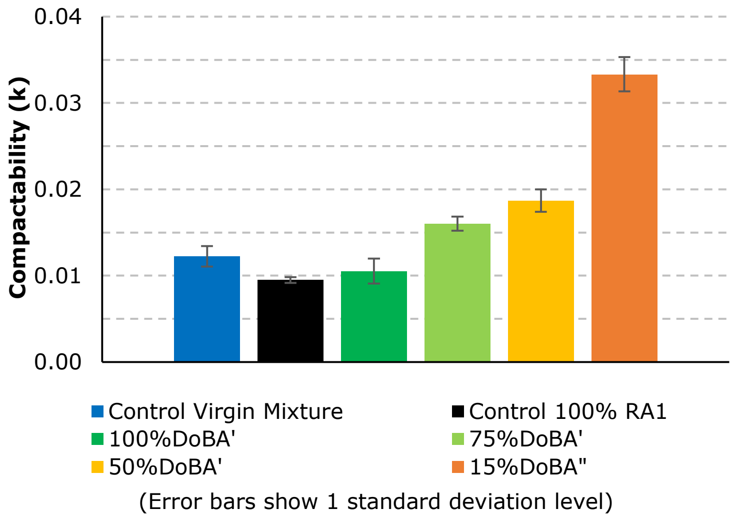


Figure 99: Mixtures - Compactability

The compaction procedure was carried out for the six mixtures and the parameter k was determined. The k parameter represents the resistance to compaction of the mixtures with the lower the value of k, the higher the resistance to compaction (the less compactability). When analysing the results, it can be seen that the Control 100% RA1 mixture presents the lowest compactability requiring a higher number of gyrations to reach the densities required to obtain the k. The values found for the Control virgin mixture and 100% DoBA were quite close to the value of the Control RA1. The other recycled mixtures (75%DoBA’, 50%DoBA’ and 15%DoBA”) present a gradual increase in the compactability with the decrease of the DoBA% due to the effect of the amount of VB 50/70 bitumen added (lower DoBA% translates into more virgin bitumen and higher compactability). Consequently, considering that the grading curves are the same and influence compactability (Micaelo, 2008; Hu *et al.*, 2017), the higher amount of bitumen provides greater impact as it decreases the contact between aggregates resulting in faster compaction. Therefore, the compactability results may not completely cooperate with the DoBA analysis, although it suggests that the Black rock theory behaves different in distinct occasions during the manufacturing process (e.g. the inactive binder may be a black rock during the mixing process, but stills acts as bitumen during compaction).

6.2.5. Specimens manufacturing

The asphalt mixtures were manufactured at NTEC following the same conditions for heating temperatures and mixing times presented in previous sections. The required quantities of RA1, virgin aggregate and binder were pre-heated at the specified mixing temperature to distribute heat uniformly. The RA1, or the virgin aggregates, were placed and mixed in a hand mixer (with temperature control), at the specified mixing temperature and times. The mixture was then placed in a preheated gyro mould (100 mm ø), aged 1 hour in oven at 140°C and compacted in the gyratory compactor until the desired final height of the sample was achieved.

The mixtures were manufactured targeting the final height, using the maximum density determined for each mixture and consequently the desired bulk density. The air voids content selected was equal to 5%, commonly used in asphalt mixtures and already in accordance with the specifications described in Section 3.3.1. However, the mixtures were manufactured targeting 70mm thickness and higher air voids content (6%-7.5%). The cylindrical specimens were then trimmed on both faces (10 mm) in order to reach 50 mm as the final height and to obtain the 5% air voids. Trials were attempted before manufacturing the final specimens for testing in order to get the desirable air voids and improve the samples' homogeneity as suggested by researchers (Lo Presti *et al.*, 2013, 2015). The final produced specimens, trimmed on both faces and with a final height of 50 mm are showed in Figure 100.

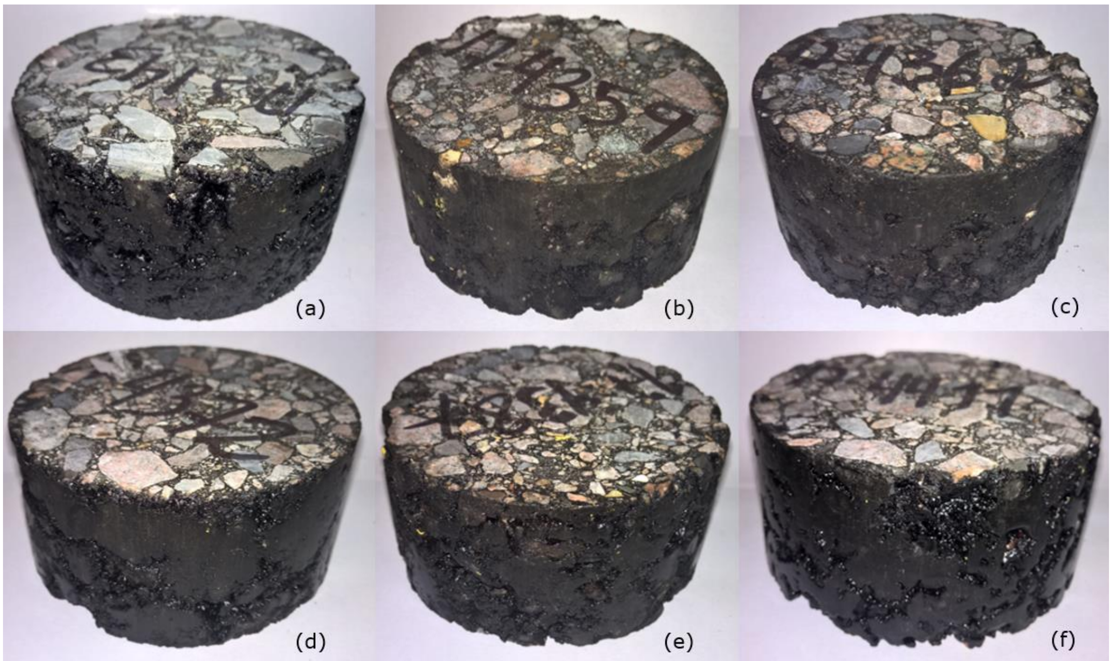


Figure 100: Produced specimens – (a) Virgin control mixture; (b) Control 100% RA1; (c) 100%DoBA'; (d) 75%DoBA'; (e) 50%DoBA'; (f) 15%DoBA"

6.2.6. Specimens - Volumetric properties

The maximum densities of the mixtures (previously determined) were used to calculate other properties of each specimen such as the air voids. In order to do this, the bulk density of the specimens was determined following the European Standard EN 12697-6 (2012) using the sealed specimen procedure. Equation 51 was used to calculate the bulk density:

$$\rho_{bsea} = \frac{m_1}{\left(\frac{m_2 - m_3}{\rho_w}\right) - \left(\frac{m_2 - m_1}{\rho_{sm}}\right)} \qquad \text{(Equation 51)}$$

Where,

ρ_{bsea} = bulk density sealed, in g/cm^3;
m_1 = mass of the dry specimen, in grams (g);
m_2 = mass of the sealed specimen dry, in grams (g);
m_3 = mass of the sealed specimen in water, in grams (g);
ρ_w = density of the water at the test temperature, in g/cm^3;
ρ_{sm} = density of the sealing material at the test temperature, in g/cm^3.

The measured air voids for each specimen was then calculated from the following Equation 52:

$$v_v = \frac{\rho_m - \rho_{bsea}}{\rho_m} \times 100 \qquad \text{(Equation 52)}$$

Where,

v_v = air voids;

ρ_{bsea} = bulk density, in g/cm^3;

ρ_m = maximum density of the mixture, in g/cm^3.

The findings shown in Figure 101, represent the average values of 15 samples for each mixture. The error bars represent the standard deviation values of bulk density and air voids (columns) for these samples.

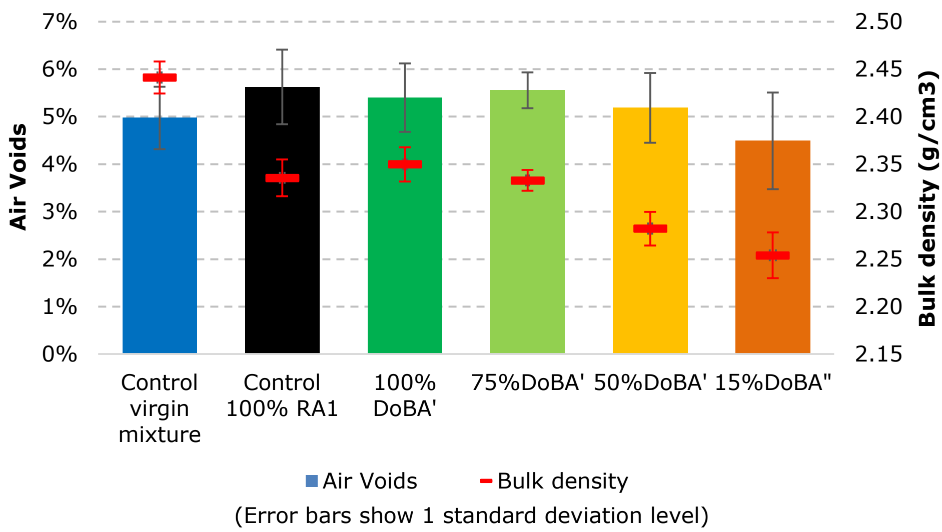


Figure 101: Produced cylindrical specimens – Air voids and bulk densities

The air voids (columns in Figure 101) are in accordance with the targeted 5%, although there is a variation between the minimum and maximum values. This variation could be attributed to the distribution of air voids and the orientation of aggregates within the specimen. However, this range is still within the allowable limit in accordance with the standard EN 13108-1 (2016). It can be also seen that the mixtures with lower DoBA% (higher binder amount, considering the inactive RA1 binder) resulted in relatively lower air voids. The better compactability of those mixtures could be responsible for this decrease, although it might be result of the excess of bitumen and compromised arrangement of aggregates as well.

In summary, the most important outcome is the similar air voids between all the mixtures, which makes them comparable for the mechanical tests. In this sense, eventual differences in mechanical test results may be the effect of the bitumen presence in the mixtures, active and inactive.

6.3. Mixtures mechanical performance

6.3.1. Stiffness modulus

6.3.1.1. Indirect Tensile Stiffness Modulus (IT-CY)

The IT-CY method is described in the European Standard EN 12697-26 (2012) and was performed on cylindrical specimens measuring 100mm diameter by 50mm. The procedures undertaken were described in detail in Section 3.3.3. The temperature commonly used to conduct the IT-CY test is 20°C, and all test specimens were subjected to 24 hours conditioning at the test temperature. In addition, three temperatures were also selected to observe the stiffness variation between the mixtures (0°C, 10°C and 30°C). The results are presented in Figure 102 with the mean of 5 samples taken as the final value:

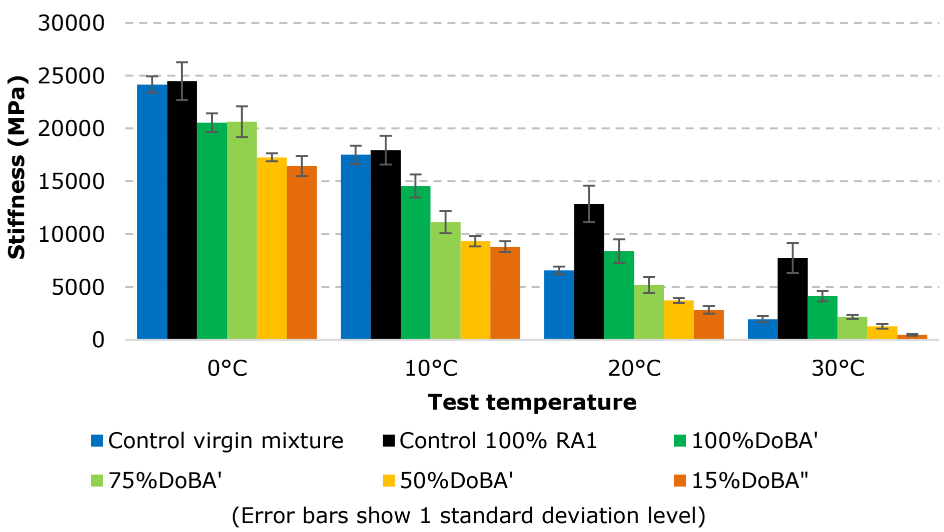


Figure 102: IT-CY results

At the standard test temperature of 20°C there is considerable variation of the values obtained for the recycled mixtures. Firstly, the Control 100% RA1 mixture showed the highest stiffness. This was expected as the aged material is stiffer than any other material with a value around 12500 MPa. The Control virgin mixture, as the target, had about 6000 MPa in stiffness modulus at 20°C. In this sense, the recycled mixtures were expected to have values between these references as the binder blend design targeted the VB 50/70 properties. However, the amount of VB 50/70 binder added (in 75%DoBA', 50%DoBA' and 15%DoBA" mixtures) has probably changed the internal arrangements of aggregates, affecting the stiffness of the specimens due to the increased bitumen presence. The average values obtained are summarised in Table 32:

Table 32: IT-CY results at 0°C, 10°C, 20°C and 30°C

	Stiffness Modulus			
Mixtures	**0°C**	**10°C**	**20°C**	**30°C**
Control Virgin Mixture	**24163**	**17518**	**6562**	**1940**
Control 100% RAP	**24490**	**17947**	12862	7740
100% RAP (100%DoBA' 170°C)	20551	14554	**8380**	4131
100% RAP (75%DoBA' 140°C)	20643	11143	**5192**	**2161**
100% RAP (50%DoBA' 70°C)	17257	9313	3707	1269
100% RAP (15%DoBA" 70°C)	16458	8816	2820	466

The results similar to the Control virgin mixture for each temperature are highlighted (in bold) in the table. Firstly, is important to mention that due to the many variables (such as binder design, mixture design and total binder content) involved in the mixtures it is difficult to confirm which material (e.g. active/inactive binder or virgin binder) or property dominates the test results. Due to this, it is assumed that the DoBA for each mixture affects the results because of the amount of added VB 50/70. This can be seen when the stiffness of the mixtures decreases when less active binder from the reclaimed asphalt RA1 is assumed (75%DoBA'>50%DoBA'>15%DoBA"). This seems to indicate that the Black Rock theory usually considered for RA materials during production (where the inactive binder material is supposed to act as an aggregate) may not be valid. The observation was already evident from the compactability k results.

The results in Table 32 show that at lower temperatures (0 and 10°C) both the control virgin and the control 100% RA mixtures have the same stiffness values. Although this would appear strange due to the harder nature of the RA binder compared to the virgin binder, similar results (complex shear modulus) were found for the binder properties (Section 4.2.2) at low temperatures for RA1 binder and VB 50/70. For the recycled mixtures with added VB 50/70 (75%DoBA’, 50%DoBA’ and 15%DoBA”), the stiffness is lower at low temperatures (0 and 10°C) in comparison to both control mixtures. This is probably due to the higher bitumen content (considering the inactive binder) in these mixtures.

However, looking at the highest test temperature of 30°C (Table 31), the stiffness is similar for the Control virgin and 75%DoBA’ mixtures. From the results highlighted in Table 32, it seems that the Control virgin mixture behaves differently to the others, with a different slope comparing the temperatures, e.g. if the test were carried out at 40°C, the stiffness would possibly be similar for Control virgin and 50%DoBA’ mixtures.

Therefore, even though there are some variability in the analyses, which can mainly be attributed to the addition of VB 50/70, the trend for all temperatures is that the stiffness reduces when more bitumen is added in the mixtures. Moreover, is critical to say that the inactive binder still seems to be contributing with some viscoelasticity to the test and not simply act as a black rock. In this sense, the final analysis will compare the stiffness modulus and the complex shear modulus from the binders in order to find any similarities between those results.

6.3.1.2. Stiffness: Mixtures x binders

In order to better understand the effect of the DoBA on the IT-CY, the magnitude of the complex shear modulus of RTFOT aged binders measured at approximately similar conditions (temperature and loading frequency) of IT-CY testing were used. Figure 103 and Figure 104 were drawn with the Stiffness and Complex modulus, respectively, showing the isochrones at 8 Hz frequency.

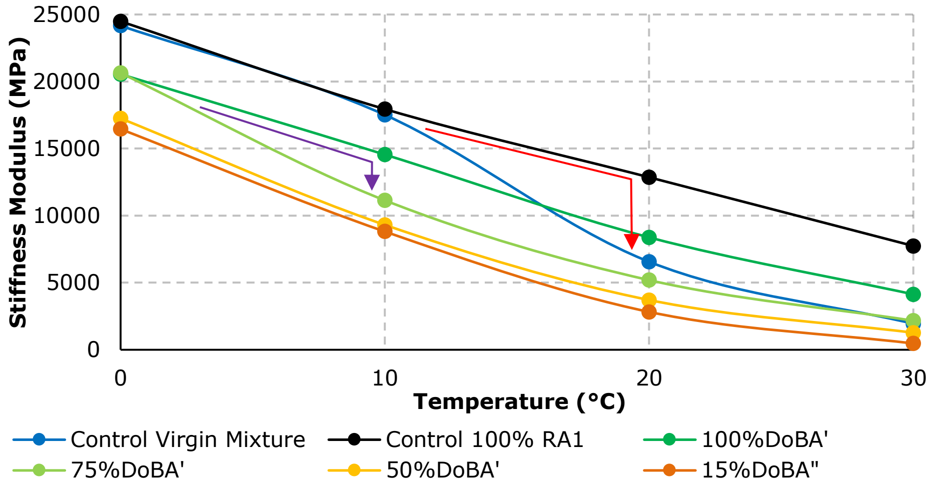


Figure 103: Mixtures – Isochrones (8 Hz)

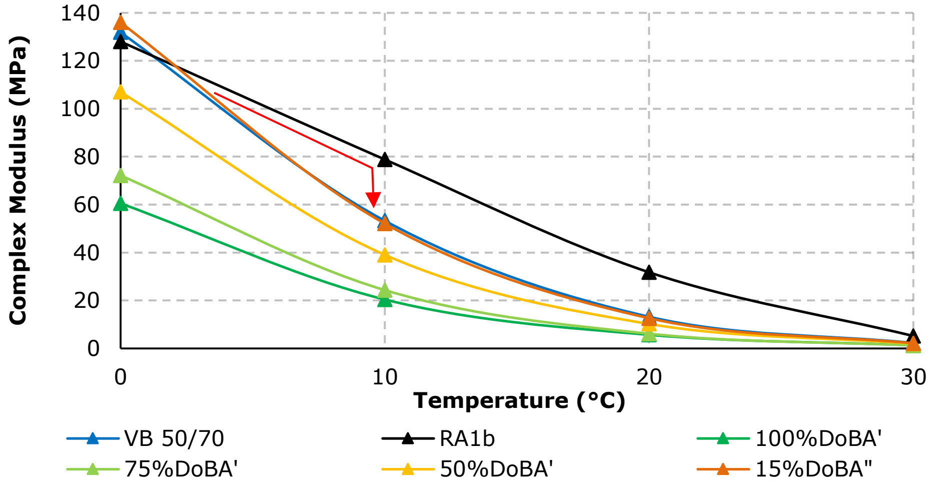


Figure 104: Binders – Isochrones (8 Hz)

The first analysis that can be made is the difference in the shape of most curves. The mixture RA1, as well as the bitumen RA1b, are the only ones that present the same trend and the same position in both graphs (stiffer materials). Among the other materials, some similarities can be observed for the blue curves (VB 50/70 and Control virgin mixture) and also in the case of 75%DoBA′. However, the position in the graphs (if we list a stiffness ranking) changes.

The recycled binders 100%DoBA′ and 75%DoBA′ were the less stiff (Figure 102), but the opposite was found in the mixtures, being shifted in relation to the other binders 50%DoBA′ and 15%DoBA″ (likely because of the larger amounts of virgin bitumen affecting these mixtures).

Regarding VB 50/70 bitumen and Control virgin mixture, the change of stiffness as a function of the variation of temperatures can be observed. In Figure 103, the stiffness decreases between 10 and 20°C, from a position similar to the Control 100% RA1 mixture to a value between the recycled mixtures 100%DoBA′ and 75%DoBA′ (indicated by the red arrow). A similar situation can be seen in the complex modulus (red arrow in Figure 104), where VB 50/70 at 0°C has a stiffness value close to that of the RA1b binder, but this decreases considerably when tested at 10°C.

Similar behaviour can be seen in the recycled mixture 75%DoBA′, but in this case from 0°C to 10°C (indicated by the purple arrow, Figure 103), probably because of the addition of the VB 50/70 bitumen. It is challenging to analyse the temperature susceptibility of the materials behaviour and therefore the link between the bitumens and the mixtures. Despite this, it is evident that the bitumens' temperature susceptibility affects the results of the mixtures.

The mixture 75%DoBA′ which has similar stiffness to 100%DoBA′ mixture at 0°C shows a difference between the two mixtures at 10°C due to the extra presence of virgin binder in its composition. Moreover, the presence of the VB 50/70 in the recycled mixtures makes the analysis more complex due to the excess of softer material, which interferes with the IT-CY results. Unlike this, the DSR tests were carried out on bitumens with their proportions (RAb and VB 50/70) totally controlled. In this sense, the graph in Figure 105 was created in order to compare all the materials and how much the stiffness changed between the temperatures, presenting the percentages of reduction of Stiffness/Complex modulus of each material.

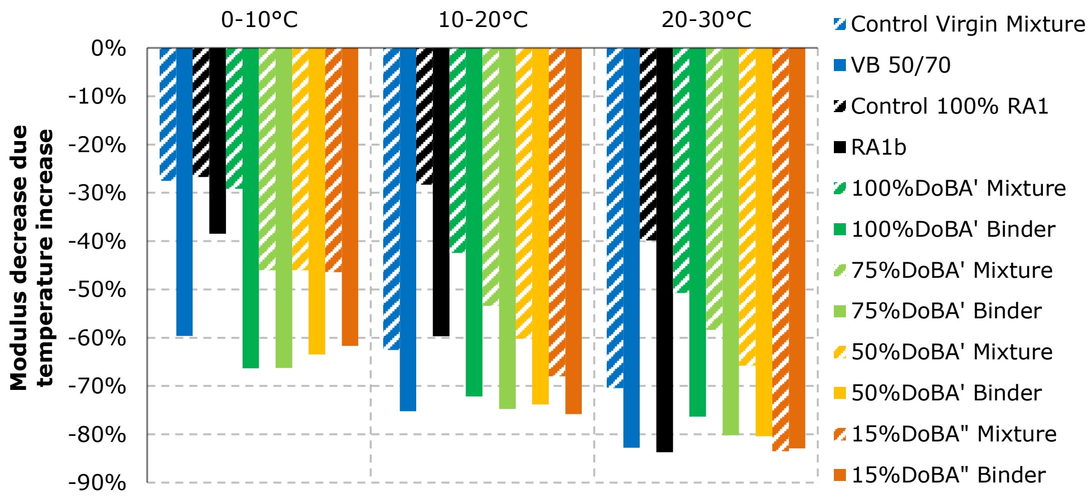


Figure 105: Mixtures/Binders – Stiffness/Complex modulus reductions

Firstly, it is important to observe the results for complex modulus only (solid fill columns), where except for the RA1b binder, all the binders showed a similar reduction in G* with the gradual increase of temperature (individually analysing each temperature change). This consistency can be attributed to the accuracy of the binder blend design and the tests performed on bitumens with precise DoBA, which consequently provided the balance between all binders. Using this consistent binder trend, it is possible to analyse the mixtures since the decrease in the stiffness has changed the shape of the curves shown in Figure 103 and Figure 104.

Based on the two references, Control 100% RA1 mixture presents a constant reduction between 0-10°C and 10-20°C, but a larger change for 20-30°C. In the case of the Control virgin mixture this larger change occurs for 20-30°C, although the changing in the material behaviour seems that happens at 10-20°C.

When analysing the recycled mixtures in Figure 105, firstly for the 0-10°C case, two groups can be perceived: 100%DoBA’ together with the reference mixtures, and the second group with 75%DoBA’, 50%DoBA’ and 15%DoBA” mixtures. In this temperature variation, the differences were lower because the VB 50/70 and RA1b bitumens have similar stiffness modulus at these temperatures, as already mentioned (Figure 103). Similarly, in the second group at 0-10°C, the mixtures have binders (VB 50/70 and RA1b) in their composition which are not severely affected by the low temperature. From this, together with the increase in temperature, up to 20°C and 30°C, the VB 50/70 binder presented the highest variation as well as the Control virgin mixture, directly affecting the results of all the other recycled mixtures.

With these results, it was possible to see the variation in the materials behaviour due to the temperature change. Despite the complexity of doing an exact analysis of the DoBA adopted through the IT-CY test, it was possible to see how temperatures affect and are important in this type of research with RA. In conclusion, the most promising results were found for the 100%DoBA’ mixture which has similar stiffness to reference mixtures at low temperatures; and, 75% DoBA’ recycled mixture, which also shows similar results although the increase in temperature was influenced by the VB 50/70 binder (similar variation between isochrones VB 50/70 and 75% DoBA’ mixture). The other mixtures (50%DoBA” and 15%DoBA’) presented high variations, probably as a result of the excess bitumen in the mixtures as a consequence of the mixture design approach that was used in this study. At the same time, it cannot be affirmed that the DoBA is incorrect, because for the mixing process and investigation of the DoBA itself (Chapter 4), these values seem to be practical. A key consideration is the fact that the RA1b inactive portion (DoBA values less than 100%) might led to errors when assuming that the inactive binder acts as a Black rock when it may still be contributing to the actual binder volume.

6.3.2. Rutting related properties

6.3.2.1. Repeated Load Axial Test (RLAT)

The rutting resistance of the asphalt mixtures was characterised by means of RLAT at 50°C. This test provides as the main outcome the permanent axial deformation as a function of loading cycles. The test method is described in the British Standard BS DD 226 (1996) and was performed on cylindrical specimens measuring 100mm diameter by 50mm. The procedures undertaken were detailed in Section 3.3.3. The typical results obtained from the RLAT for the different mixtures are shown in Figure 106, where permanent axial strain is plotted against load cycles.

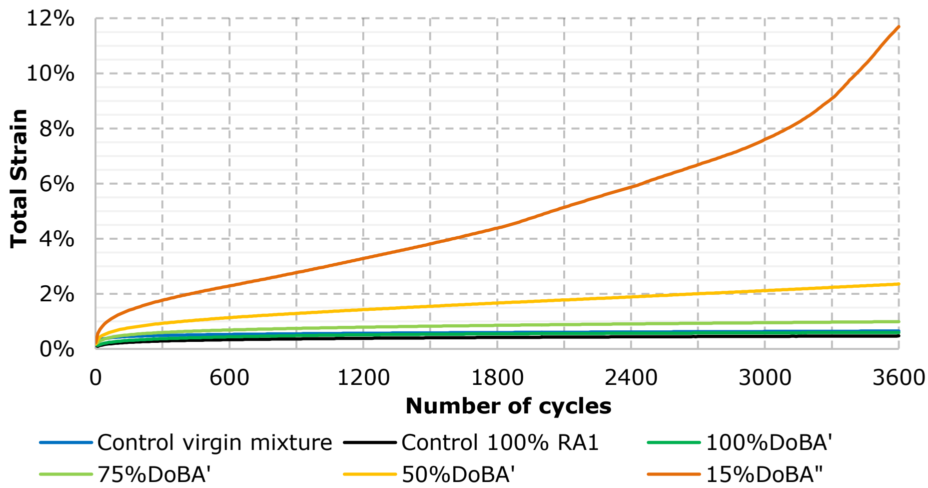
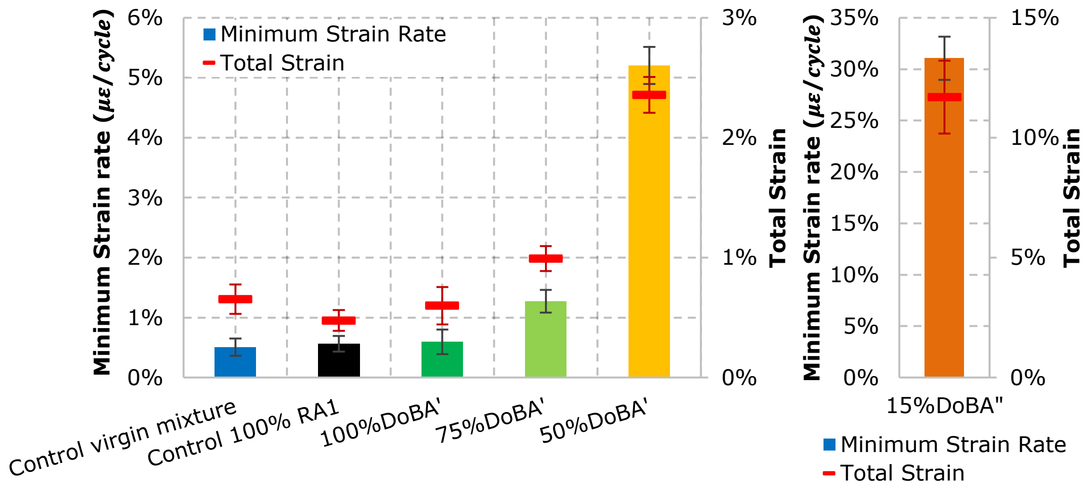


Figure 106: RLAT results at 50°C

As can be seen, the recycled mixtures 50%DoBA’ and 15%DoBA” present the highest strain values throughout the cycles, especially the 15%DoBA” mixtures with very high strains. Regarding the other four mixtures (Controls, 100%DoBA’ and 75%DoBA’), the results were very close, presenting similar permanent deformation resistance from the beginning until the end of the cycles, with a slightly greater deformation for 75%DoBA mixture. The permanent deformation results in terms of the total accumulated strain at the end of 3600 pulses, and in terms of the minimum strain rate (columns), are presented in Figure 107. The error bars represent the standard deviation values for three replicates.



(Error bars show 1 standard deviation level)

Figure 107: RLAT results in terms of the minimum strain rate and total strain

The results confirm the similar rutting performance of the 100%DoBA' recycled mixture and the Control mixtures (virgin and RA1). In particular, these mixtures show the best rutting performance among the other mixtures. According to Widyatmoko (2008), a standard asphalt mixture is considered to have a satisfactory rutting resistance if its axial deformation is lower than 2% on specimens tested at 40°C. The control mixtures as well as the 100%DoBA' and 75%DoBA' have met this indication at 50°C. The 50%DoBA' mixture showed a final total strain between 2-2.5% at 50°C, a test at 40°C would possibly achieve the maximum 2% strain suggested; and, the 15%DoBA" was the only one which presented a total discrepancy in the results, clearly affected by the excess amount of bitumen.

The influence of binder content on permanent deformation for the mixtures 50%DoBA' and 15%DoBA" show an unexpected trend if it is assumed that portion of the RA binder are inactive (Black rock situation). However, as the previous results have already suggested, the inactive RA1b binder may not be acting entirely as a black rock and therefore this extra amount of binder has a significant effect on the RLAT results. It can be seen that increasing the binder content has increased the total strain and significantly changed the minimum strain rate (e.g. comparing

75%DoBA’ to 15%DoBA”). Increasing the binder content can make the asphalt mixture more susceptible to permanent deformation as the binder film becomes thicker between aggregate particles (Khanzada, 2000; Tayfur et al., 2007). Figure 108 shows the tested samples where the effect of the bitumen amount in those mixtures can be seen and where the total axial strain was high in comparison to the control mixtures (specimens clearly damaged). The mixtures not included in Figure 106 showed no signs of damage.

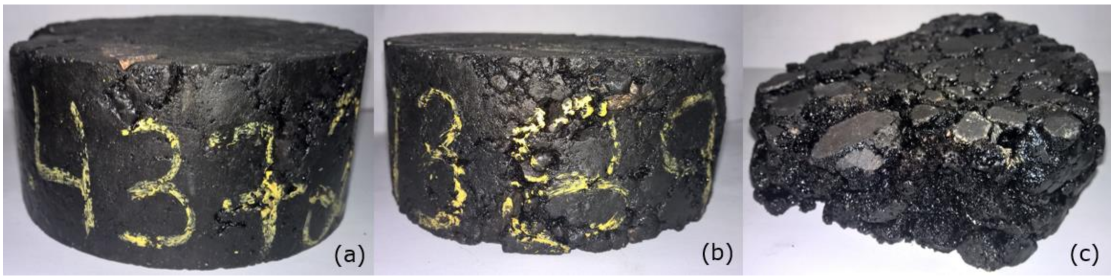


Figure 108: RLAT specimens - (a) 75%DoBA’; (b) 50%DoBA’; (c) 15%DoBA”

Therefore, as observed for the IT-CY results, the RA1b might be still active in the recycled mixtures and not behaving entirely as a mineral aggregate. Moreover, similar to the results found on stiffness, the recycled mixtures 100%DoBA’ and 75%DoBA’ are the ones with closer values to the control virgin mixture. This means that the assumed DoBA for these mixtures can provide satisfactory results in this type of analysis. In this sense, the final analysis compares the RLAT parameters with the MSCR test of the binders in order to find any similarities between these results.

6.3.2.2. Rutting: Mixtures x binders

In order to obtain a better understanding of the effect of the DoBA on the RLAT, the magnitude of the non-recoverable compliance (Jnr) of RTFOT aged binders measured at the same temperature (50°C) of the RLAT testing were used.

Regarding the mixture parameters, the total strain is considered highly affected by the initial strain, and, the initial strain is more sensitive to the initial conditioning of the test apparatus as well as the different orientation of the aggregates within the specimen. With this in mind, the minimum strain rate was considered more fundamental to consistently characterise the rutting resistance of the mixtures according to Subhy (2016). Due to these reasons, and to create a relationship between binder properties and mixture, the rutting parameter of binders are correlated with the minimum strain rate of mixtures. Based on the results in Figure 107, the mixtures are ranked based on the minimum strain rate at a test temperature of 50°C as follows:

RLAT: Control virgin mixture= Control 100% RA1 = 100%DoBA′ > 75%DoBA′ > 50%DoBA′ > 15%DoBA″

The binders were also ranked based on MSCR test results at 50°C:

MSCR: RA1b > 100%DoBA′ > 75%DoBA′ > 50%DoBA′ > VB 50/70 > 15%DoBA″

From the rankings, it can be seen that only one material is in a different position, namely the VB 50/70 or the Control virgin mixture. This results may be the result of the volumetric properties as well as the final binder content. It can therefore be concluded that the DoBA methodology adopted provides a good correlation between binders and mixtures in accordance with their rankings. However, the DoBA percentages cannot be confirmed as the parameters of binders and mixtures are not correlated through the ranking classification.

In order to evaluate statistically how these parameters are associated with the rutting performance of the mixtures, a linear regression analysis was applied to the values of Jnr binder parameters and the minimum strain rate of mixtures, as shown in Figure 109.

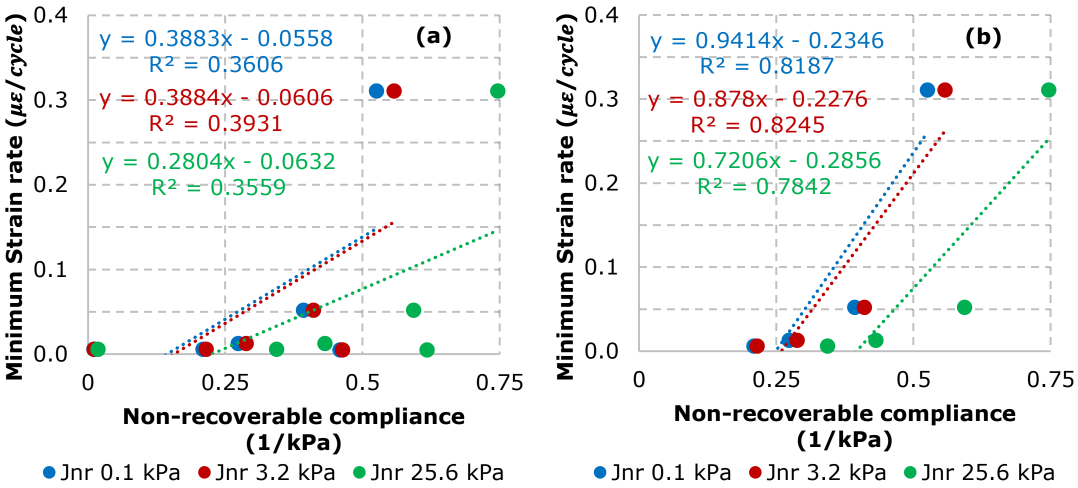


Figure 109: Mixtures and binders rutting correlation – (a) All mixtures; (b) Recycled mixtures

It can be seen that the rutting parameters obtained, Figure 109a, have a very poor correlation with the mixture property characterised by the minimum strain rate. On the other hand, to directly analyse the DoBA, the Jnr parameter obtained from MSCR at stress levels of 0.1 kPa, 3.2 kPa and 25.6 kPa (suggested by Subhy (2016)) was correlated with minimum strain rate but only for the recycled mixtures in Figure 109b. It can be seen that the correlation improves significantly if the control materials are excluded. Removing the control mixtures and binders from the analysis provides a better understanding of how the recycled mixtures behave with regard to DoBA. Despite knowing the characteristics of the control mixtures, the results are quite different from a trend that can be found for the recycled materials. In the case of Control virgin mixture, this may be because of the fresh properties of the binder, and for Control 100% RA1, this may be simply because of the high resistance to rutting commonly found in aged materials.

Another analysis is presented in Figure 110, considering only three recycled mixtures in each graph, (a) with 50% DoBA' and (b) with 15% DoBA". Because the two mixtures were produced with the same RA1 conditioning process (70°C), the only difference between them is the amount of virgin binder added to replace the supposedly Black rock portion. In this sense, it was understood that one of these could provide a better trend for the real DoBA of the RA1 conditioned and produced at 70°C.

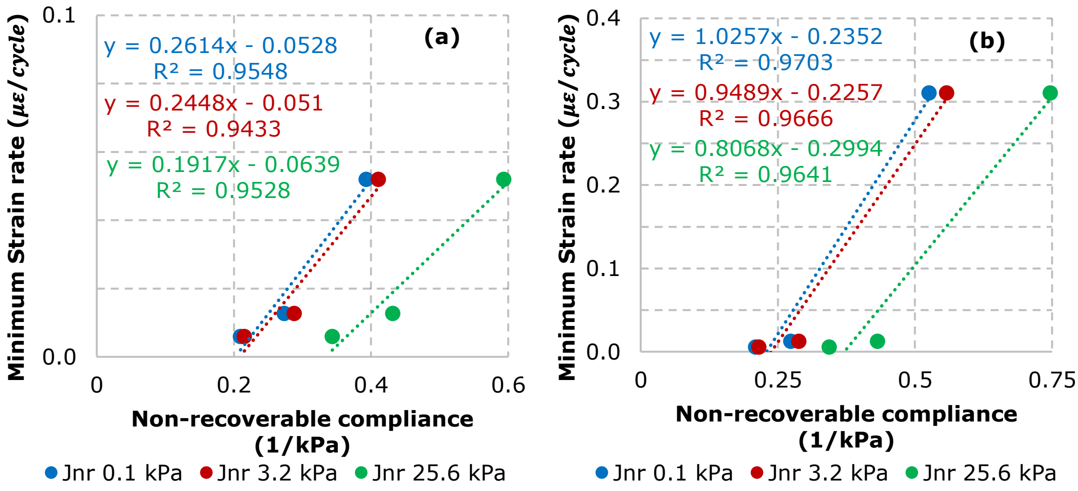


Figure 110: Mixtures and binders rutting correlation – (a) Recycled mixtures with 50%DoBA'; (b) Recycled mixtures with 15%DoBA"

Figure 110 shows that ignoring one of the values of DoBA, the correlation coefficient improves from about R^2=0.8 (Figure 109b) up to values higher than 0.95 in most of the cases. First, it was known that these values would increase due to the fact that the analysis only has three points for correlation. Also, it is evident that in the correlation between binders and mixtures, there are no major differences between 50%DoBA' and 15%DoBA", since linear regression in both cases presented high R^2. With this, the RLAT results of the mixtures can be analysed directly, and, the higher resistance of the 50%DoBA' was identified. Therefore, with better results in the 50% DoBA' mixture, a satisfactory correlation between binders and mixtures, it can be considered that the RA1 conditioning and production at 70°C should have a DoBA value closer to 50% than 15%. For the other recycled mixtures (100%DoBA' and 75%DoBA'), the results of resistance to rutting are as good as the Control virgin mixture. Not even the 75%DoBA' mixture (with virgin binder added) presented negative effects in terms of permanent deformation performance of the mixture.

6.3.3. Fatigue cracking related properties

6.3.3.1. Indirect Tensile Fatigue Test (ITFT)

The fatigue resistance of the asphalt mixtures was characterised by means of ITFT at 20°C. This test provides as the main outcome the cycles to failure of the asphalt mixture after the loading cycles. The test method is described in the standard BS DD ABF (2003) and was performed on cylindrical specimens measuring 100mm diameter by 50mm. The procedures undertaken were described in detail in Section 3.3.3. The fatigue laws, together with the raw fatigue data obtained from the ITFT for the different mixtures are shown in Figure 111.

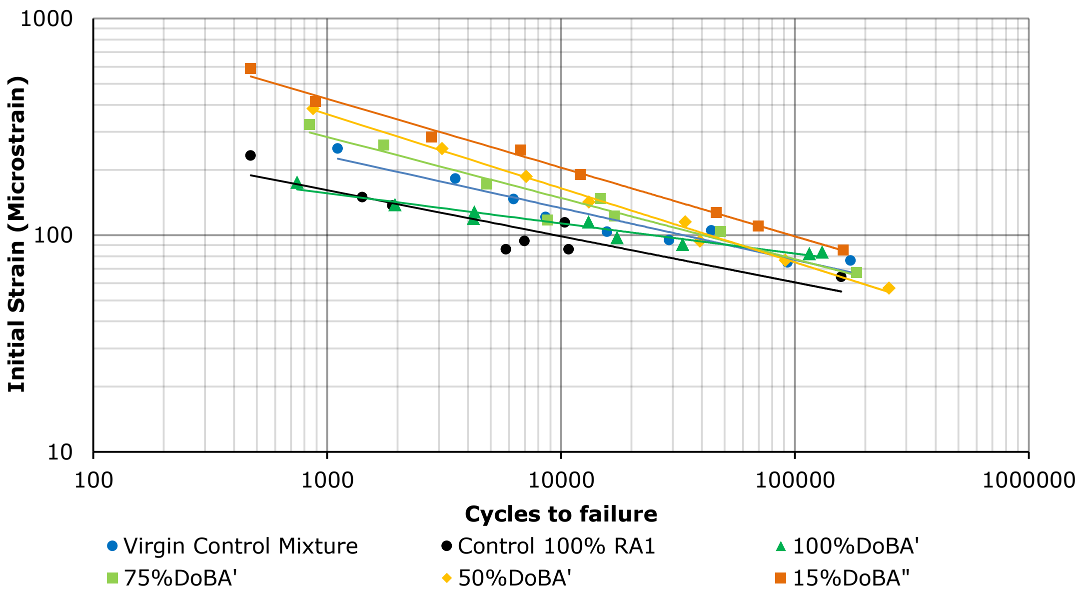


Figure 111: ITFT results at 20°C

It can be seen that the recycled mixture 15%DoBA” had the best fatigue life. The worst performance was obtained for the Control 100% RA1, likely due to the aged properties of the bitumen without any rejuvenation treatment. From this, can be observed that by using the additive in the 100%DoBA’ mixture, the fatigue law is shifted to the right (from the Control RA1) and the slope of the curve is also modified.

Regarding the control virgin mixture, can be seen to have a better performance than the Control 100% RA1 and 100%DoBA but similar to the other two recycled mixtures (75% and 50%DoBA'). Although it can be clearly seen which mixtures had the best and worst performance (15%DoBA" and Control 100% RA1, respectively), the other mixtures present different slopes and their performance should be analysed differently as is not simple to distinguish them. Thus, in order to support the fatigue laws, the fatigue parameters obtained are presented in Table 35, together with the fatigue functions and their R^2 values.

Table 35: ITFT - Fatigue relationships for asphalt mixtures

	Fatigue relationships		Fatigue parameters	
Mixtures	**Fatigue function**	**R^2**	**$\varepsilon 10^6$ $(\mu\varepsilon)$**	**$N_f^{100\mu\varepsilon}$**
Control Virgin Mixture	$1.830 \times 10^{12}e^{-3.88}$	0.93	44	31,800
Control 100% RA1	$6.198 \times 10^{11}e^{-3.93}$	0.84	37	8,500
100%DoBA'	$1.072 \times 10^{18}e^{-6.83}$	0.95	60	23,400
75%DoBA'	$1.650 \times 10^{11}e^{-3.32}$	0.94	41	37,800
50%DoBA'	$2.800 \times 10^{10}e^{-2.91}$	0.99	34	42,400
15%DoBA"	$1.702 \times 10^{11}e^{-3.13}$	0.99	47	93,600

Except for the Control 100% RA1 asphalt mixture, the fatigue functions have been determined with a relatively high degree of accuracy (large R^2 correlation coefficients) for all the other five mixtures, with the fatigue functions being established from a fatigue data set with a minimum of eight points (specimens) as shown in Figure 111.

Therefore, as observed from the IT-CY and RLAT results, the RA bitumen might still be having an influence on the mixtures and not acting only as a black rock. From the results in Table 35, it can be seen that the effect of virgin bitumen in the recycled mixtures is to increase the number of cycles at 100 microstrain. Similar effects were previously investigated by researchers (Baaj *et al.*, 2005; Tomlinson, 2012; Boriack *et al.*, 2014) where the fatigue performance of the mixtures was improved with the higher binder content for mixtures also including RA. However, the parameter of microstrain at 1 million cycles presented differences and did not follow the sequence expected by the DoBA percentages. Despite this, it is evident that the change in the slope of the curves directly affects the parameters under analysis, since the fatigue relationships from Table 35 are derived from different points of each fatigue law. Thus, in the same way as the stiffness and rutting investigations presented previously, a final fatigue analysis is presented where the parameters of the ITFT tests were compared with the results obtained from the LAS test on binders.

6.3.3.2. Fatigue cracking: Mixtures x binders

In order to further analyse the relationship between recycled binders and asphalt mixtures, the ITFT parameters have been related to the binders parameters from LAS tests (Section 5.3.2) for several combinations to find a reliable linear relationship. The analysis adopted the RTFOT aged binders for analysis for the best correlation with the lab produced mixtures. The investigations included the curve slope, microstrain at 1 million cycles and N_f^{100ue} parameter as characteristics from the mixtures. From the binders, the Glover-Rowe parameter, R-value, Alfa, slope of the curve (Nf x strain) and $Nf^{x\%e}$ from LAS tests were used.

Figure 112 (a-h) presents the charts displaying the relation between binder parameters (x-axis) and mixture parameters (y-axis). It is important to mention that in the same way as the rutting correlations, the values of the reference mixtures and binders (Virgin and RA) were also withdrawn.

The results presented in Figure 112 show that the weaker correlations are those where the microstrain at 1 million cycles from mixtures is used (Figure 112 a, c, e). This shows that the extrapolation of the results up to 1 million cycles is affected by other properties of the mixture, since low strain values are generally not used in the cracking parameters analysis for bitumens. Therefore, it is possible to observe that the correlations using N_f at 100 microstrains presented higher R^2

coefficients (Figure 112 b, d, f), more noticeable when related to the R-value of the recycled binders (Figure 112d) with R^2=0.75.

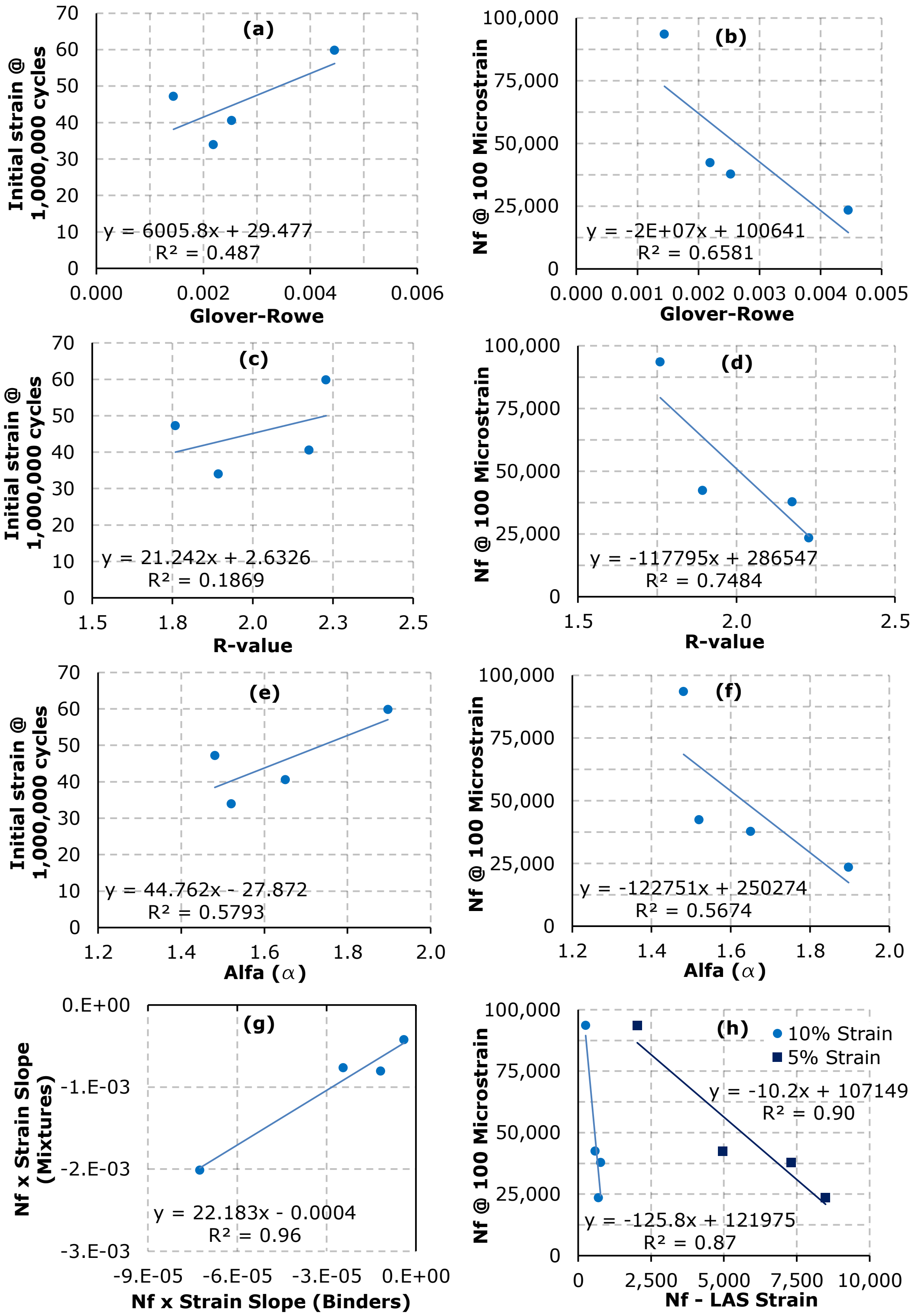


Figure 112: Mixtures and binders fatigue correlation

The two best correlations were found when similar properties are related to each other. Figure 112g shows the fatigue law slopes from each material, with the highest R^2 obtained (0.96). Figure 112h presents the correlation made between cycles to failure at 100 microstrains from mixtures and the cycles to failure at 10% and 5% strains from the binders, also with a high R^2 values of 0.87 and 0.9 respectively. The LAS strains selected were chosen based on similar analysis made by researchers (Ameri *et al.*, 2015; Foroutan Mirhosseini *et al.*, 2017; Sabouri *et al.*, 2018). From these results, it can be concluded that any analysis carried out for the DoBA may provide a more reliable outcome once the results are well correlated from similar test conditions (though different magnitudes). These results are in accordance with similar findings from other researchers (Jiménez del Barco Carrión, 2017; Hasaninia and Haddadi, 2018; Sabouri *et al.*, 2018).

Considering the slopes of the fatigue lives, it is important to highlight the need to analyse these slopes as they affect the materials behaviour and especially their stress-strain sensitivity when using RA. In this sense, the good correlation between the fatigue laws slopes leads to the conclusion that despite the amount of virgin bitumen added in the recycled mixtures, the DoBA% assumed might have positively affected the results due to the good correlation with the binders.

Similarly to what was found for stiffness and rutting, fatigue is also affected by the extra amount of virgin binder, but here providing better fatigue resistance. Therefore it becomes difficult to conclude if the fatigue resistance is partially or entirely affected by the assumed DoBA or if the main outcomes are related to the amount of binder in each mixture. Regardless of the observations and accuracy of the DOBA% analysis, it is evident that the RA1 binder has been reactivated, partially acting in the recycled mixtures regardless of the conditioning temperature.

6.3.4. Water sensitivity

Because the recycled mixtures were designed for single courses and there is a concern about inadequate aggregate coating and adhesion (between RA1b and VB 50/70), it is important to evaluate the water sensitivity. However, this property was investigated only for design purposes, because it is not possible to use it for the DoBA verification.

The water sensitivity was characterised by determining the ratio of conditioned (wet) to unconditioned (dry) stiffness modulus (IT-CY) value at 20°C, performed on cylindrical specimens measuring 100mm diameter by 50mm. The procedures undertaken were detailed in Section 3.3.3.

Regarding water damage, given that RA aggregates are already covered with bitumen, the water sensitivity resistance is likely to increase in mixtures with high percentages of RA (Karlsson and Isacsson, 2006; Mogawer *et al.*, 2012; Tran *et al.*, 2012). Table 36 presents the IT-CY values for the Control and recycled mixtures before and after being exposed to successive water immersion cycles. Figure 113 shows the IT-CY ratio after three conditioning cycles.

Table 36: IT-CY Water sensitivity

	Stiffness Modulus (MPa)			
Mixtures	**Unconditioned**	**Cycle 1**	**Cycle 2**	**Cycle 3**
Control Virgin Mixture	6217	8878	7547	6983
Control 100% RA1	13056	15379	14120	12816
100%DoBA’	7877	10156	8488	8780
75%DoBA’	5088	6160	6159	5328
50%DoBA’	3577	4421	3826	3513
15%DoBA”	2920	4037	3914	3170

The average value of IT-CY for three specimens is reported in Table 36. The crucial benefit of running the IT-CY test after each conditioning cycle is that it gives more information about the behaviour of the mixtures. However, contrary to what could be expected, the results in Table 36 indicate that the immersion regime after the first cycle led to an increase in stiffness compared to its unconditioned value for all mixtures. The initial increase in stiffness could be attributed to the binder ageing during conditioning, most probably after immersing the specimens in the hot water bath at 60°C for 6 hours. The effect of binder ageing on IT-CY was possibly dominant over the water damage. Similar results were found by other researchers (Widyatmoko, 2008; Heneash, 2013; Lancaster and Lancaster, 2016; Subhy, 2016).

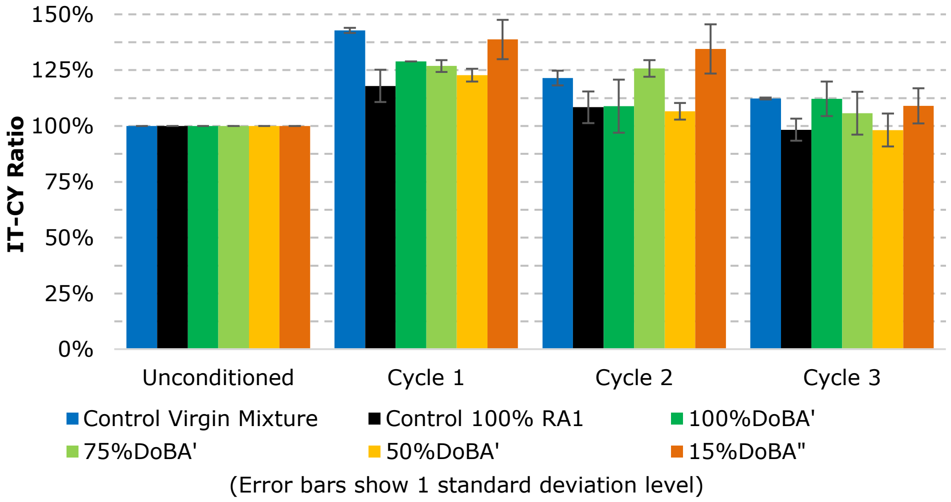


Figure 113: Water sensitivity – IT-CY ratio after immersion cycles

By looking at the retained stiffness ratio after the Cycle 1, it can be noticed that a significant increase of 42% took place for Control virgin mixture, while 18% occurred for the Control 100% RA1, with the recycled mixtures positioned between these two. Heneash (2013) hypothesises that the temperature of the hot water might have accelerated the diffusion of the virgin bitumen through the RA binder, leading to more blending between the binders or possibly acting as a curing period.

In order to better evaluate the moisture damage on mixtures, Figure 114 presents the IT-CY Ratios using the Cycle 1 as the reference. As the conditioning Cycle 1 affected the stiffness of the mixtures because of the possible ageing during the conditioning, the remaining two cycles were compared with the first.

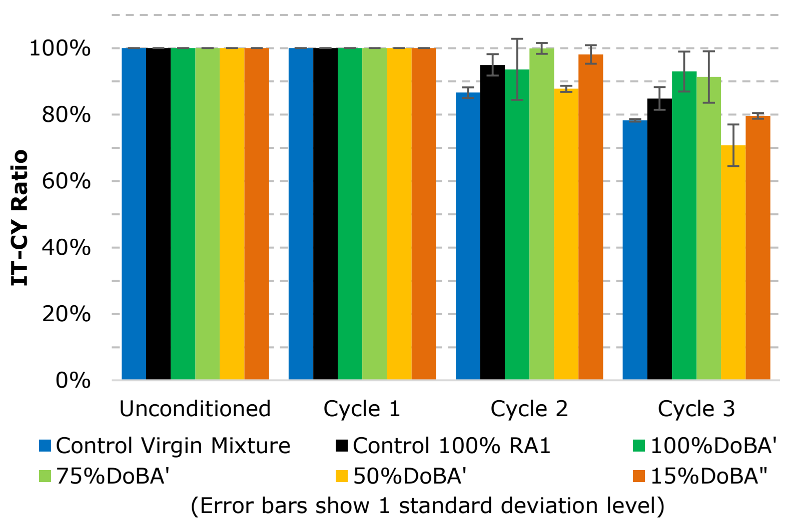


Figure 114: Water sensitivity – IT-CY ratio Cycle 1 reference

A minimum retained stiffness ratio of 70% has been set to safeguard against stripping (EN 13108-1, 2016). It can be seen that all mixtures passed the minimum limit indicating good moisture damage resistance after the third conditioning cycle. Comparing the results, the recycled mixtures presented higher resistance than the Virgin control mixture, only the 50%DoBA showed lower values.

The moisture damage mechanism occurs by infiltration of moisture through binder or mastic film. The moisture then reaches the aggregate-binder interface and shifts the binder from the aggregate, leading to a reduction of the adhesive bond between the materials. Consequently, separation of aggregate particles is prone to take place. As the RA is a material which has been exposed to ageing, consequently RA binder becomes stiffer. Thus, the bond between the aggregate and the RA binder becomes stronger. This strong bond makes the invasion of moisture difficult to reach the aggregate-binder interface (Caro *et al.*, 2008). It has also been reported that the RA binder tends to stick and coats the RA aggregates. This reduces water absorption when using RA materials, therefore, the resistance of recycled mixtures to water sensitivity increases (Karlsson and Isacsson, 2006; Guthrie *et al.*, 2007; Schaertl and Edil, 2009; Heneash, 2013).

6.4. Summary and conclusions

According to the DoBA results presented in Chapter 4 and the estimated percentages of released binder for the RA1, four values were chosen for the binders (Chapter 5) and mixtures investigation. All the mixtures were prepared in the laboratory and their properties tested. From these a summary of the conclusions can be made:

A typical Asphalt Concrete 0/20mm was selected from the European specification EN 13108-1 (2016) for manufacturing the recycled and control mixtures. The selection was based on the original white grading curve of the RA material. The bitumen content in these mixtures was targeted as 4.73%, although some portion of the RA1b was disregarded in some mixtures due to the DoBA% investigated (inactive bitumen).

The mixing and compaction process of the recycled mixtures were accomplished by considering different RA conditioning and mixing temperatures (70, 100, 140 and 170°C). Also, an additive was considered to rejuvenate the RA1 bitumen properties. The maximum density results were determined for each mixture and used in the specimen production, targeting a final specimen with 5% air voids.

The first procedure carried out for analysis was the compaction where the mixtures compactability was evaluated. The main outcome was that the compactability results do not fully collaborate with the DoBA verification, but suggests that the Black rock theory (inactive bitumen) is not entirely true with part of the inactive binder not acting as black rock.

Regarding the mechanical performance of the mixtures, the IT-CY test was performed at 0, 10, 20 and 30°C. Even though there are some variations in the stiffness analysis, the trends are the same for all investigated temperatures as the stiffness reduces when more virgin bitumen is added to the mixtures. Furthermore, it seems that even for lower assumed values of DoBA (less than 100%), the RA1b binder still contributes to the viscoelasticity of the test results and does not simply act as part of the mineral aggregate (black rock portion).

The rutting resistance of the asphalt mixtures was characterised using RLAT at 50°C. The mixture produced assuming 100%DoBA presented the best rutting resistance amongst the recycled mixtures similar to the control virgin and RA1 mixtures. Similar to the findings from the stiffness analysis, the inactive RA1b binder also influences the RLAT results, where the influence of binder content for 50%DoBA′ and 15%DoBA″ mixtures showed an unexpected trend and lower rutting resistance. Moreover, the recycled mixtures 100%DoBA′ and 75%DoBA′ are the ones with closer values to the control virgin mixture, meaning that the assumed DoBA for these mixtures is probably correct.

The fatigue resistance of the asphalt mixtures was characterised by means of ITFT at 20°C. The recycled mixture 15%DoBA″ delivered the best fatigue performance while the Control 100% RA1 was the worst. The Control 100% RA1 also showed considerable stress-strain sensitivity. The control virgin mixture presented similar resistance to that found for the 75%DoBA′ and 50%DoBA′ recycled mixtures. The different slopes of the fatigue lives are affected by the RA1 and its aged properties becoming difficult to distinguish some mixtures. Therefore, as observed in the IT-CY and RLAT results, the RA1 inactive bitumen is possibly influencing the mixtures. Moreover, the added virgin bitumen significantly affected the results.

The water sensitivity was also characterised by determining the IT-CY ratio (dry and wet samples) at 20°C. Different to what was expected, the results showed that the conditioning cycle led to an increase in stiffness. This can be attributed to the binder ageing during the conditioning in hot water. Despite this, all mixtures indicated good moisture damage resistance. In summary, the recycled mixtures presented higher resistance than the Virgin control mixture.

Regarding the DoBA, the main findings from the mechanical tests (stiffness, rutting and fatigue) were correlated with the designed binders parameters obtained in the previous chapter.

Stiffness and Complex Modulus: Despite the complexity of doing an exact analysis of the DoBA, it was possible to see how temperatures affected the research using RA and how important it is. The most promising results were found for the 100%DoBA' case which had similar stiffness to the reference at low temperatures and the 75% DoBA' recycled mixture which also presenting similar results but was affected by the VB 50/70 binder portion. However, it cannot be affirmed that the DoBA is incorrect as the RA1b inactive portion might have led to an error when considering it partially as a Black rock.

Rutting: In order to evaluate the rutting, a linear regression analysis was applied to the values of Jnr binder parameters and the minimum strain rate of mixtures. A good linear correlation was found between both parameters, especially for 100%DoBA' and 75%DoBA' together with 50%DoBA'. Therefore, with good results with the 50% DoBA' mixtures, it can be considered that the RA1 conditioning and specimens produced at 70°C should have a DoBA value closer to 50% than 15% and the other recycled mixtures possibly similar to what was assumed.

Fatigue: the ITFT parameters were correlated with the binders parameters from the LAS test at several combinations to find a reliable linear relationship. The best correlation was found from the fatigue live curves slope from each material (binder versus mixture). Also, a good correlation was found between cycles to failure at 100 microstrains from mixtures and the cycles to failure at 10%/5% strains from the binders. This lead to the conclusion that despite the amount of virgin bitumen added in some mixtures, the assumed DoBA% may have affected the fatigue lives. Similarly to what was found for stiffness and rutting, the fatigue is also affected by the virgin binder which resulted in a better fatigue resistance. Therefore, it is difficult to conclude if the fatigue resistance is affected entirely by the DoBA% or if the major consequences are connected to the amount of binder in each mixture. Regardless of the comments related to the DoBA% analysis, it can be concluded that part of the RA1 binder has been reactivated and has attributed to the fatigue results.

Therefore, the final mechanical tests proved that they can be well correlated with the designed binders. Regarding DoBA%, the three properties analysed in mixtures (stiffness, rutting and fatigue) can be analysed together to verify the DoBA% found from the methodology proposed in Chapter 3 and the results in Chapter 4. In this sense, stiffness and fatigue results were found to be more sensitive to binder content present in the mixtures, whereas the rutting analysis is more affected when the bitumen amount is too high. Every property analysed brings slightly different conclusions, but the most important outcome is related to the assumed inactive bitumen.

From the results presented, is evident that the bitumen disregarded during the DoBA design is still potentially active in the mixtures. It can also be said that different RA materials may act more like a Black rock than the RA1 under investigation in this research due to its own characteristics, although this could only be proved by undertaking an extense experimental programme.

7. CONCLUSIONS AND RECOMMENDATIONS

7.1. Summary

The main objective of the work described in this thesis was to propose an innovative and practical methodology to estimate the Degree of Binder Activation (DoBA) for selected RA's as a function of mixture manufacturing processes (mixing temperature and time). In this thesis, the DoBA was defined as the proportion of RA binder that is activated in the RA and can be considered a unique property of the RA. Moreover, active binder is the amount of aged binder that does not need replacement with a new agent. In order to estimate the DoBA for each temperature and material, the parameters investigated were obtained through the Indirect Tensile Test (ITT), with the DoBA determined as a mechanical relationship of the selected test. For this purpose, procedures to better understand the RA as a material within new mixtures were assessed, connecting the manufacturing process and its outcomes with binder and mixture design approaches. Furthermore, the study evaluated the feasibility of using an asphalt mixture which incorporated 100% RA.

The research has also focused on the characterisation and design of recycled binders through the study of their conventional, rheological and performance-related properties. These properties have been used as an input in the mixture design stage of the research using specific DoBA levels, determined in the previous stage of the research. Thus, four recycled mixtures containing 100% RA were produced and their performance evaluated in terms of stiffness, rutting, fatigue and water sensitivity. The relationships between recycled binders and mixtures were studied in order to verify the DoBA methodology.

7.2. Conclusions

In the following sections, a detailed review of the main findings and conclusions are presented after an in-depth investigation and analysis conducted in this study.

7.2.1. The Degree of Binder Activation methodology

A methodology was proposed which connected the DoBA, binder design and recycled asphalt mixture design. The selected RA's were first characterised in order to gather information, the Cohesion test framework was tailored and the outcomes analysed to correlate the mechanical test results with the DoBA. This DoBA information was then used to develop the blend design for the binders and asphalt mixtures with the results from all three stages (DoBA, binder and mixture design) finally correlated together.

The DoBA investigation was carried out to understand and measure the bitumen activation from RA for new asphalt mixtures. The outcomes can be used as a tool to better understand the RA in order to improve the binder and mixture design. Mixture design methodologies naturally aim at estimating the optimum binder content of a given asphalt mixture, hence the risk is to over or under-dose the bitumen for the new mixtures, which can be directly affected by the DoBA assumed. The active binder content of the RA can affect the field performance of the new mixture where too much bitumen (underestimated DoBA% = more virgin bitumen to be added) can result in bleeding, lowered skid resistance and lowered resistance to permanent deformation while low bitumen content (overestimated DoBA% = less virgin bitumen to be added) may compromise fatigue resistance and result in problems with raveling and stripping.

In order to achieve the objective of measuring the RA's DoBA, the procedure adopted to age a virgin bitumen to create an artificial RA binder proved to be efficient and the artificial bitumens were obtained with similar properties to their respective RA binders. The Penetration, Softening Point, Viscosity (Brookfield) and Complex Shear Modulus results from the bitumen's were investigated and can be applied as comparison methods to achieve the desired binder properties.

The tests undertaken to estimate the DoBA showed that variations in conditioning and manufacturing temperatures are a crucial factor when considering 100% RA. DoBA increased as temperature increased resulting in higher indirect tensile strength values. The same tendency was not found by increasing mixing times. The range of temperatures investigated was from 70°C to 170°C, and mixing times from 30 seconds (asphalt plant reality) up to 3 minutes (lab reality). Regarding the specimens volumetrics, between the intermediate temperatures (100-140°C), the RA bitumen began to be activated in larger amounts thereby improving the workability.

The DoBA was determined using different parameters (indirect tensile strength, total energy, pre and post peak energies). The indirect tensile strength proved to be an efficient tool where the RA material showed similar behaviour to Artificial RA, making the comparison feasible. The Energy Pre-peak region parameter also proved to be an essential extra tool of analysis. These parameters considered other aspects of the Indirect Tensile Test such as the load-displacement and the energy absorption during the whole test. The flexibility index, Total Energy and Energy Post-peak parameters did not present satisfactory correlation with the temperatures as expected, indicating that these parameters may not be appropriate to be used in this methodology.

The DoBA index provided an innovative idea of how to estimate the amount of active RA binder available in recycled mixtures. The DoBA analysis was performed on RA samples tested without any binder extraction and therefore directly simulated the real mixing process that RA materials are subjected to in recycled asphalt mixtures. This is opposite to the approach generally described in literature where DoBA is assessed through binder results. An alternative DoBA index (using the ITT results from specimens of RA produced at 170°C as reference, instead of using the Artificial RA results) was also presented with a good correlation with the manufacturing temperatures. However, the alternative DoBA is relative to the highest temperature tested, therefore this method requires further investigation because of the manufacturing limitations, e.g. production at 70°C and 100°C result in specimens with higher air voids content than 170°C and might affect the ITT.

An additive was used as a rejuvenator in one source of RA and tested to obtain the effect on the DoBA. The DoBA analysis from the additive application was in accordance with the DoBA obtained previously (without the additive) and presented similar trends with temperature variation. Moreover, it was also observed that the additive can improve the RA bitumen mobilisation. In this sense, with the DoBA index obtained through different temperatures, four DoBA percentages were successfully selected according to the DoBA study. These DoBA % were 100% DoBA at 170°C, 75% DoBA at 140°C, 50% DoBA at 70°C and 15% DoBA at 70°C.

7.2.2. Binders: design and performance-related properties

A binder blend design was carried together with the optimal additive dosage in order to determine the recycled binders to be investigated in the research. Then, four recycled binders (100%DoBA, 75%DoBA, 50%DoBA and 15%DoBA) had their conventional and rheological properties determined. These properties were found to be similar to the control virgin binder and in accordance with the limits indicated by the European standards for bitumen specification. Also, the critical temperatures were determined using the rheological measurements and developed to connect the properties with the performance of the bitumens. The results showed that recycled binders were able to provide similar or better performance than the control binder VB 50/70.

Performance-related tests (Multiple Stress Creep Recovery, Linear Amplitude Sweep and BBR) were performed on the recycled binders to support the conventional and rheological properties and for the DoBA verification (comparing with the mixtures performance-related tests). The results from the performance-related tests of the binders showed that the DoBA% was affected by the amount of VB 50/70 added to replace the inactive RA binder. This because no such trend was observed in the performance of the binders (with VB dominating some tests), showing the differences experienced by the recycled binders and virgin bitumen. However, the overall performances of the binders were positive and promising, supporting the good binder design carried out as well as the additive dosage adopted.

7.2.3. Mixtures: design, performance-related tests and correlation with binders

A control virgin mixture, control RA mixture (no treatment) and four recycled mixtures were designed and produced meeting the European specification for a typical Asphalt Concrete 0/20mm. The selection of the aggregate grading and binder content was based on the original white grading curve of the RA material and its binder content. These grading requirements were adopted due to the fact that when 100% RA was used in the final recycled mixture, the aggregate grading and binder content would be that of the RA material.

The mixtures were produced at different RA conditioning temperatures to reproduce the DoBA investigation. The first analysis carried out addressed mixture compactability with the results suggesting that the assumption that the inactive RA bitumen (based on DoBA) only acts as Black rock and does not contribute to the final asphalt mixture is possibly not wholly accurate. Part of the inactive RA binder does appear to play an active role in the asphalt mixture during compaction.

The mechanical properties of the mixtures were also evaluated. The stiffness was determined using the IT-CY test with some variations in the results due to the amount of virgin binder added to compensate for the inactive RA binder portion. The results suggest that the RA binder still contributes to the viscoelastic response of the mixture during the test and does not simply act as part of the mineral aggregate. Correlating the Stiffness Modulus with the Complex Shear Modulus of the bitumens, it was possible to see how temperature affects the research results. The most promising results were found for the 100%DoBA case which had similar stiffness to the reference mixture at low temperatures and the 75% DoBA recycled mixture which also presented similar results but was slightly affected by the VB 50/70 binder portion (because of its temperature susceptibility).

The rutting resistance of the mixtures was evaluated through the RLAT test. The best performance was experienced by the recycled mixture 100%DoBA, similar to the control mixtures. In the same way as the stiffness test, the inactive RA binder also influenced the rutting analysis, where the influence of bitumen content for 50% and 15% DoBA mixtures showed low rutting resistance. The rutting parameters from mixtures were correlated with the MSCR test from the binders. A good linear correlation was found between both parameters (non-recoverable compliance of binders and minimum strain rate of mixtures).

The fatigue resistance of the mixtures was assessed with the ITFT. The best performance was experienced by the 15%DoBA mixture due to the effect of the high amount of binder in the mixture composition. The control virgin mixture presented similar fatigue resistance to the 75%DoBA and 50%DoBA recycled mixtures. The different slopes of the fatigue lives are affected by the RA, with considerable stress-strain sensitivity for some mixtures. The ITFT parameters were correlated with the binders parameters from the LAS test and the best correlation was found between the fatigue law slope from each material (binder versus mixture). The correlation between cycles to failure 100 microstrains from mixtures and the cycles to failure at 10% and 5% strain from the binders were also good. Similarly to what was found for stiffness and rutting, fatigue results lead to the conclusion that the amount of virgin bitumen added in some mixtures and the assumed DoBA% may have affected the results (with better performance for those mixtures with higher total bitumen content, usually observed in fatigue tests).

The water sensitivity of the mixtures was also evaluated. All mixtures indicated good moisture damage resistance, with the recycled mixtures presenting higher resistance than the Virgin control mixture.

7.2.4. The DoBA of the investigated scenarios

Regarding the assumed DoBA%, the following conclusions regarding the DoBA can be made:

- **100%DoBA':** the only case where the virgin bitumen could not affect the analysis. It has also provided the best comparisons with the control virgin mixture. Thus, even if it is not possible to confirm the 100% activation of the bitumen, from the tests performed in this research and according to the methodology adopted, it is hypothesised that the RA1 conditioned and produced at 170°C has activated an amount of bitumen close to 100% in this study. Translating the results into road performance implications, using the principle of designing mixtures with the optimum binder content, the durability of the pavement is guaranteed by the adequate dosage (disregarding the quality of the materials).

- **75%DoBA':** first recycling mixture with virgin bitumen added. Here the results could be directly affected by the fresh bitumen and the higher amount of binder. However, the mixture produced at 140°C presented promising results regarding DoBA% as well as in its performance compared to a 100% RA mixture. In this case, it can be concluded that the small amount of virgin binder added has provided more benefits in terms of performance than negatively affecting the mixture through excess bitumen. Thus, having positive results and being comparable with the control virgin mixture, the hypothesised 75%DoBA’ has also proved to be near to real DoBA of the material at these manufacturing conditions.

- **50%DoBA’** and **15%DoBA”:** the mixtures produced with RA1 conditioned at 70°C should be analysed together. In both cases, the amount of virgin bitumen added has highly affected the mechanical properties of the mixtures. Considering the mechanical tests, the stiffness presented lower values when more virgin bitumen was added; fatigue was improved with the addition of virgin bitumen; and, rutting was also affected by the bitumen, presenting low rutting resistance in the 15%DoBA” case. Analysing all the results together, it is clear that the 50%DoBA’ provides more equilibrium between all the properties, with good fatigue and rutting resistance as well as satisfactory stiffness. When correlating with the binders, the analysis does not provide too many answers because both materials were well correlated. In this sense, analysing the results, it is evident the DoBA of 15% was underestimated and it could be concluded that 50%DoBA is the most reasonable value. Translating hypothetically into field performance, underestimated DoBA% requires more virgin bitumen to reach the desired optimum binder content, thus, the pavement can experience problems such as bleeding, low skid resistance and low rutting resistance.

7.2.5. Practical implications

According to the research shown, it is evident that RA bitumen activation changes, and the final properties during the mixing procedures change according to various factors such as temperature, mixing time and types of materials. In this sense, in order to maximise the incorporation of RA material in new mixtures, it becomes important to have a reasonable assumption of the DoBA of a selected RA for a specific manufacturing process. In fact, one of the reasons for moving forward with this property (DoBA) and its methodology, is that during the mixture design phase in the lab, it is relatively simple to recover the RA binders and determine the properties, but during construction in the field, the time available for quality control does not permit all essential tests to be performed. Furthermore, characterisation of RA should be performed on the RA itself directly, then this material can be considered in new mixture design as a component on its own, cooperating with the other components.

Regarding high-content RA asphalt mixtures, correctly predicting the real amount RA binder available is understood to be decisive to obtain asphalt mixtures which meet with specific design standards. If the RA binder does not blend with the virgin binder as predicted, pavement performance could be compromised. Therefore, the activation of the RA binder can play an important role in the new mixture, where correctly predicting its value will govern the amount of new components to be considered during the mixture design.

On this basis, the present research brings the potential of the methodology to be a wide-spread method to characterise RA with the need of having a label for the DoBA of RA at different manufacturing conditions. In this sense, it is believed that identifying and estimating the DoBA can support the maximisation of RA content in new asphalt mixtures.

7.3. Recommendations for future research

The following points are recommended for future research work:

- In order to improve the DoBA methodology, different sources of RA should be investigated. RA's derived from three distinct types of asphalt mixtures could be considered such as dense grade (e.g., asphalt concrete), gap graded (e.g., stone matrix asphalt) and open-graded (e.g., porous asphalt). Moreover, two sub-types of RA containing pure bitumen and PMB as well as divided into two sub-types on the basis of the different stages of ageing, such as short (e.g. binder penetration >20 dmm) and long-term aged (e.g. binder penetration < 10 dmm) could be included. This will give a total of 12 different RA sources in this scenario. Different sources of RA could provide different approaches for the further binder and mixture design.

- Using the DoBA methodology, characterise the RA using the indirect tensile strength test, and develop a procedure based on the bitumen Penetration (most common bitumen property to identify the RA). This could be done by undertaking ITS tests to find out the bitumen tenacity and performing fatigue, rutting and stiffness tests on the RA (samples) to find correlations between e.g. Bitumen penetration x ITS x fatigue. With several sources of RA encompassing different bitumen penetration (possibly distinguishing neat and modified binders), a large database can be obtained from ITS and mechanical properties. With this, the correlation could result in final guidelines to use the DoBA from any RA, based on mechanical properties (but estimated through the ITS).

- Different conditioning temperatures of the RA could be used to improve the accuracy of the analysis. The effect of mixing temperatures higher than 170°C on RA sources with neat and modified bitumens could lead to different results if the assumption that mixing at 170°C results in 100% DoBA is incorrect. This assumption could be checked by seeing if ITS continues to increase with temperature at temperatures greater than 170°C. Including more intermediate temperatures and those lower than 70°C will also increase the reliability of the methodology.

- Although the increase of mixing time did not show significant differences in samples with 100% RA, further studies of mixing times would be valuable to investigate the time effect when additives are used. This fact could result in different perspectives depending on the type of additive, where higher DoBA (and also DOB) could be achieved if the additive was able to reach internal layers of RA bitumen (closer to the aggregate).

- The development of an improved methodology that considers the DoBA and the degree of blending of RA binder and virgin binders or additives occurring in RA mixtures. This would be a step forward for the mixture design, with a DoBA method able to define the blending between RA binders and other materials based on the properties of the RA. Therefore, the binders and mixtures could be better correlated and the mechanical properties would lead to the investigation of which bitumen (RA or virgin) may be dominating the outcomes of the mixtures tests (in contrary to what is observed in the binders, where the portion of RA and virgin binder can be controlled).

- The investigation of other binder combinations, including several DoBA% (e.g., from 0%, increasing 10% up to 100%), would also deliver a large amount of data to be analysed. Combining the binder tests with the same values of DoBA% for the mixtures would provide enough data to allow the binders and mixtures to be correlated in terms of conventional and performance-related properties. Furthermore, the RA conditioning temperatures for mixture production also requires variations such as producing mixtures at the same temperatures but considering different DoBA%. However, this would require an extensive experimental programme to provide enough data to validate the DoBA methodology and its optimisation.

- An investigation of the DoBA measurement from the laboratory practice to the field reality would be a step towards the increase of RA content in new asphalt mixtures. The procedures described on the present thesis focus in the laboratory determination of the DoBA for the selected RA’s, however, it is still necessary to investigate if the same conditioning and manufacturing procedures will provide the same DoBA in an asphalt plant to that found in the lab. The mechanical tests execution may provide similar outcomes, despite this, the materials conditioning, mixing time and temperatures are likely to require adaptation.

- Investigate the DoBA through different mechanical tests. Although the results with ITT are promising and with good statistical correlation, it is not possible to affirm that the values of DoBA estimated by the ITT method would be the same (or similar) as the values obtained by another mechanical test applied in the same methodology. In addition to a verification of the proposed method, it could also provide answers on the need to investigate the DoBA through different mixture properties (such as fatigue, rutting and stiffness).

- Undertake an experimental campaign to establish precision limits for the DoBA measurement. Although mechanical test results usually have their own specifications regarding precision in the standards (e.g. ITS values are acceptable if the difference in indirect tensile strength on the individual test specimens is not greater than 17% of the mean value), an investigation to establish precision and bias limits for the repeatability and reproducibility of the tests for the DoBA (as a mechanical relationship measurement) would provide higher accuracy to the methodology.

- Substitute the laboratory experimentation for modelling and simulation. Firstly, a mathematical model containing all the parameters and variables affecting the DoBA (those variables already presented in this thesis) should be built in order to represent the model in a virtual form, and finally, simulations could be performed. In this way, the experimental work which is extremely time consuming could be avoided or at least reduced.

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APPENDICES

Appendix 1 – Europe binder specifications (EN 12591, 2009)

Property	Test method	Unit	20/30	30/45	35/50	40/60	50/70	70/100	100/150	160/220
Penetration at 25 °C	EN 1426	0,1 mm	20 – 30	30 – 45	35 – 50	40 – 60	50 – 70	70 – 100	100 – 150	160 – 220
Softening point	EN 1427	°C	55 – 63	52 – 60	50 – 58	48 – 56	46 – 54	43 – 51	39 – 47	35 – 43
Resistance to hardening at 163 °C	EN 12607-1									
Retained penetration		%	≥ 55	≥ 53	≥ 53	≥ 50	≥ 50	≥ 46	≥ 43	≥ 37
Increase in softening point, - *Severity 1*		°C	≤ 8	≤ 8	≤ 8	≤ 9	≤ 9	≤ 9	≤ 10	≤ 11
or			*or*	*or*	*or*	*or*	*or*	*or*	*or*	*or*
Increase in softening point, - *Severity 2* [a]		°C	≤ 10	≤ 11	≤ 11	≤ 11	≤ 11	≤ 11	≤ 12	≤ 12
Change of mass [b] (absolute value)		%	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,8	≤ 0,8	≤ 1,0
Flash point	EN ISO 2592	°C	≥ 240	≥ 240	≥ 240	≥ 230	≥ 230	≥ 230	≥ 230	≥ 220
Solubility	EN 12592	%	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0

a When Severity 2 is selected it shall be associated with the requirement for Fraass breaking point or penetration index or both measured on the unaged binder (see Table 1B)

b Change in mass can be either positive or negative.

Appendix 2 – SUPERPAVE binder specifications

Performance Grade	PG 46			PG 52							PG 58					PG 64					
	-34	-40	-46	-10	-16	-22	-28	-34	-40	-46	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40
Average 7-day Maximum Pavement Design Temperature, °C [a]	<46			<52							<58					<64					
Minimum Pavement Design Temperature, °C [a]	>-34	>-40	>-46	>-10	>-16	>-22	>-28	>-34	>-40	>-46	>-16	>-22	>-28	>-34	>-40	>-10	>-16	>-22	>-28	>-34	>-40
Original Binder																					
Flash Point Temp, T48: Minimum °C	230																				
Viscosity, ASTM D 4402: [b] Maximum, 3 Pa•s (3000 cP), Test Temp, °C	135																				
Dynamic Shear, TP5: [c] G*/sin δ, Minimum, 1.00 kPa Test Temperature @ 10 rad/s, °C	46			52							58					64					
Rolling Thin Film Oven (T 240) or Thin Film Oven (T 179) Residue																					
Mass Loss, Maximum, %	1.00																				
Dynamic Shear, TP5: G*/sin δ, Minimum, 2.20 kPa Test Temp @ 10 rad/sec, °C	46			52							58					64					
Pressure Aging Vessel Residue (PP1)																					
PAV Aging Temperature, °C [d]	90			90							100					100					
Dynamic Shear, TP5: G*sin δ, Maximum, 5000 kPa Test Temp @ 10 rad/sec, °C	10	7	4	25	22	19	16	13	10	7	25	22	19	16	13	31	28	25	22	19	16
Physical Hardening [e]	Report																				
Creep Stiffness, TP1: [f] S, Maximum, 300 MPa m-value, Minimum, 0.300 Test Temp, @ 60 sec, °C	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30
Direct Tension, TP3: [f] Failure Strain, Minimum, 1.0% Test Temp @ 1.0 mm/min, °C	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30

Performance Graded Asphalt Binder Specification 1/2 (AASHTO, 1998)

Performance Grade	PG 70						PG 76					PG 82				
	-10	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-10	-16	-22	-28	-34
Average 7-day Maximum Pavement Design Temperature, °C [a]	<70						<76					<82				
Minimum Pavement Design Temperature, °C [a]	>-10	>-16	>-22	>-28	>-34	>-40	>-10	>-16	>-22	>-28	>-34	>-10	>-16	>-22	>-28	>-34
Original Binder																
Flash Point Temp, T48: Minimum °C	230															
Viscosity, ASTM D 4402: [b] Maximum, 3 Pa·s (3000 cP), Test Temp, °C	135															
Dynamic Shear, TP5: [c] $G^*/\sin\delta$, Minimum, 1.00 kPa Test Temperature @ 10 rad/s, °C	70						76					82				
Rolling Thin Film Oven (T 240) or Thin Film Oven (T 179) Residue																
Mass Loss, Maximum, %	1.00															
Dynamic Shear, TP5: $G^*/\sin\delta$, Minimum, 2.20 kPa Test Temp @ 10 rad/sec, °C	70						76					82				
Pressure Aging Vessel Residue (PP1)																
PAV Aging Temperature, °C [d]	100(110)						100(110)					100(110)				
Dynamic Shear, TP5: $G^*\sin\delta$, Maximum, 5000 kPa Test Temp @ 10 rad/sec, °C	34	31	28	25	22	19	37	34	31	28	22	40	37	34	31	28
Physical Hardening [e]	Report															
Creep Stiffness, TP1: [f] S, Maximum, 300 MPa m-value, Minimum, 0.300 Test Temp, @ 60 sec, °C	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24
Direct Tension, TP3: [f] Failure Strain, Minimum, 1.0% Test Temp @ 1.0 mm/min, °C	0	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	0	-6	-12	-18	-24

Performance Graded Asphalt Binder Specification 2/2 (AASHTO, 1998)

Appendix 3 – RA1b and VB HSM 36h rheology comparison

		LOG Complex Modulus |G*| (Pa)				Phase Angle (°)			
Temp. (°C)	Freq. (Hz)	RA1b	VB HSM 36h	Absolute Differ.	Relative Differ.	RA1b	VB HSM 36h	Absolute Differ.	Relative Differ.
0	0.1	7.401	7.532	0.132	1.8%	34.05	35.09	1.040	3.1%
0	0.158	7.497	7.611	0.113	1.5%	32	33.6	1.600	5.0%
0	0.251	7.553	7.684	0.131	1.7%	30.05	32.01	1.960	6.5%
0	0.398	7.613	7.753	0.140	1.8%	29.27	30.31	1.040	3.6%
0	0.631	7.662	7.817	0.154	2.0%	28.1	28.81	0.710	2.5%
0	1	7.737	7.879	0.142	1.8%	27.74	27.18	0.560	-2.0%
0	1.58	7.775	7.937	0.162	2.1%	26.87	25.78	1.090	-4.1%
0	2.51	7.826	7.989	0.163	2.1%	25.54	24.28	1.260	-4.9%
0	3.98	7.873	8.038	0.165	2.1%	24.88	23.08	1.800	-7.2%
0	6.31	7.927	8.086	0.159	2.0%	23.77	21.77	2.000	-8.4%
0	10	7.973	8.127	0.153	1.9%	23.13	20.85	2.280	-9.9%
10	0.1	6.985	7.152	0.167	2.4%	42.07	42.4	0.330	0.8%
10	0.158	7.074	7.240	0.166	2.3%	40.62	40.8	0.180	0.4%
10	0.251	7.161	7.325	0.164	2.3%	39.14	39.21	0.070	0.2%
10	0.398	7.242	7.407	0.164	2.3%	37.83	37.45	0.380	-1.0%
10	0.631	7.317	7.488	0.171	2.3%	35.87	36.01	0.140	0.4%
10	1	7.421	7.563	0.141	1.9%	34.12	34.38	0.260	0.8%
10	1.58	7.493	7.636	0.143	1.9%	32.96	32.86	0.100	-0.3%
10	2.51	7.567	7.706	0.139	1.8%	31.38	31.29	0.090	-0.3%
10	3.98	7.599	7.773	0.173	2.3%	31.32	29.88	1.440	-4.6%
10	6.31	7.696	7.840	0.143	1.9%	29.43	28.47	0.960	-3.3%
10	10	7.743	7.895	0.152	2.0%	28.33	27.39	0.940	-3.3%
20	0.1	6.361	6.437	0.076	1.2%	52.27	53.69	1.420	2.7%
20	0.158	6.472	6.560	0.088	1.4%	51.04	52.77	1.730	3.4%
20	0.251	6.586	6.677	0.091	1.4%	49.59	51.18	1.590	3.2%
20	0.398	6.694	6.788	0.094	1.4%	48.08	49.53	1.450	3.0%
20	0.631	6.800	6.897	0.097	1.4%	46.54	47.82	1.280	2.8%
20	1	6.900	6.999	0.099	1.4%	45.14	46.04	0.900	2.0%
20	1.58	6.997	7.100	0.102	1.5%	43.51	44.47	0.960	2.2%
20	2.51	7.091	7.195	0.105	1.5%	42.19	42.85	0.660	1.6%
20	3.98	7.180	7.287	0.108	1.5%	40.96	41.26	0.300	0.7%
20	6.31	7.263	7.375	0.113	1.5%	39.59	39.79	0.200	0.5%
20	10	7.325	7.453	0.128	1.8%	39.65	38.61	1.040	-2.6%
25	0.1	5.949	5.997	0.049	0.8%	58.68	60.51	1.830	3.1%
25	0.158	6.075	6.134	0.059	1.0%	56.93	58.92	1.990	3.5%
25	0.251	6.198	6.263	0.065	1.1%	55.56	57.49	1.930	3.5%
25	0.398	6.320	6.388	0.068	1.1%	54.05	55.85	1.800	3.3%
25	0.631	6.439	6.510	0.072	1.1%	52.57	54.34	1.770	3.4%
25	1	6.554	6.629	0.075	1.1%	51.02	52.7	1.680	3.3%
25	1.58	6.665	6.745	0.080	1.2%	49.71	51.11	1.400	2.8%
25	2.51	6.773	6.856	0.083	1.2%	48.23	49.54	1.310	2.7%
25	3.98	6.876	6.962	0.086	1.3%	46.83	47.99	1.160	2.5%
25	6.31	6.977	7.066	0.089	1.3%	45.57	46.47	0.900	2.0%
25	10	7.067	7.158	0.091	1.3%	44.48	45.2	0.720	1.6%
30	0.1	5.506	5.552	0.046	0.8%	62.74	64.77	2.030	3.2%
30	0.158	5.651	5.704	0.053	0.9%	62.17	63.75	1.580	2.5%
30	0.251	5.790	5.841	0.051	0.9%	60.97	63.05	2.080	3.4%
30	0.398	5.924	5.974	0.050	0.8%	59.59	61.55	1.960	3.3%
30	0.631	6.058	6.109	0.051	0.8%	58.14	60.07	1.930	3.3%
30	1	6.186	6.241	0.055	0.9%	56.87	58.71	1.840	3.2%
30	1.58	6.309	6.368	0.059	0.9%	55.45	57.21	1.760	3.2%
30	2.51	6.428	6.491	0.063	1.0%	54.05	55.7	1.650	3.1%

		LOG Complex Modulus |G*| (Pa)				Phase Angle (°)			
Temp. (°C)	Freq. (Hz)	RA1b	VB HSM 36h	Absolute Differ.	Relative Differ.	RA1b	VB HSM 36h	Absolute Differ.	Relative Differ.
30	3.98	6.545	6.610	0.066	1.0%	52.7	54.29	1.590	3.0%
30	6.31	6.659	6.728	0.069	1.0%	51.42	52.85	1.430	2.8%
30	10	6.764	6.835	0.071	1.0%	50.32	51.58	1.260	2.5%
40	0.1	4.717	4.707	0.010	-0.2%	69.26	73.48	4.220	6.1%
40	0.158	4.868	4.860	0.008	-0.2%	68.12	73.16	5.040	7.4%
40	0.251	5.015	5.021	0.006	0.1%	67.05	71.29	4.240	6.3%
40	0.398	5.162	5.182	0.020	0.4%	66.07	69.98	3.910	5.9%
40	0.631	5.310	5.336	0.026	0.5%	65.01	68.75	3.740	5.8%
40	1	5.455	5.490	0.035	0.6%	63.91	67.51	3.600	5.6%
40	1.58	5.595	5.644	0.049	0.9%	62.82	66.33	3.510	5.6%
40	2.51	5.730	5.790	0.060	1.1%	61.79	65.15	3.360	5.4%
40	3.98	5.865	5.934	0.069	1.2%	60.67	63.95	3.280	5.4%
40	6.31	5.996	6.076	0.080	1.3%	59.66	62.9	3.240	5.4%
40	10	6.120	6.205	0.085	1.4%	58.65	61.84	3.190	5.4%
50	0.1	3.930	3.872	0.057	-1.5%	77.22	81.69	4.470	5.8%
50	0.158	4.092	4.070	0.022	-0.5%	76.05	79.32	3.270	4.3%
50	0.251	4.259	4.246	0.013	-0.3%	74.67	77.99	3.320	4.4%
50	0.398	4.427	4.418	0.009	-0.2%	73.63	76.78	3.150	4.3%
50	0.631	4.585	4.588	0.003	0.1%	72.23	75.39	3.160	4.4%
50	1	4.739	4.753	0.014	0.3%	71	73.95	2.950	4.2%
50	1.58	4.893	4.917	0.024	0.5%	69.63	72.39	2.760	4.0%
50	2.51	5.044	5.077	0.033	0.7%	68.18	70.87	2.690	3.9%
50	3.98	5.193	5.233	0.039	0.8%	66.56	69.21	2.650	4.0%
50	6.31	5.335	5.385	0.049	0.9%	64.74	67.42	2.680	4.1%
50	10	5.462	5.526	0.064	1.2%	62.93	65.53	2.600	4.1%
60	0.1	3.249	3.139	0.110	-3.4%	82.45	84.81	2.360	2.9%
60	0.158	3.431	3.328	0.103	-3.0%	81.33	83.76	2.430	3.0%
60	0.251	3.608	3.514	0.093	-2.6%	80.34	83.16	2.820	3.5%
60	0.398	3.784	3.700	0.084	-2.2%	79.21	82.27	3.060	3.9%
60	0.631	3.959	3.881	0.079	-2.0%	78.32	81.12	2.800	3.6%
60	1	4.130	4.059	0.072	-1.7%	77.17	80.05	2.880	3.7%
60	1.58	4.297	4.238	0.059	-1.4%	76.1	78.9	2.800	3.7%
60	2.51	4.464	4.413	0.051	-1.2%	75.01	77.62	2.610	3.5%
60	3.98	4.627	4.581	0.046	-1.0%	73.86	76.41	2.550	3.5%
60	6.31	4.787	4.750	0.037	-0.8%	72.91	75.31	2.400	3.3%
60	10	4.935	4.908	0.027	-0.6%	72.03	74.28	2.250	3.1%
70	0.1	2.596	2.494	0.102	-3.9%	85.24	85.51	0.270	0.3%
70	0.158	2.788	2.678	0.110	-3.9%	84.31	86.2	1.890	2.2%
70	0.251	2.983	2.877	0.106	-3.6%	83.72	85.42	1.700	2.0%
70	0.398	3.171	3.064	0.107	-3.4%	83.17	85.38	2.210	2.7%
70	0.631	3.352	3.256	0.097	-2.9%	82.23	84.47	2.240	2.7%
70	1	3.530	3.444	0.086	-2.4%	81.14	83.92	2.780	3.4%
70	1.58	3.710	3.633	0.077	-2.1%	80.41	83.1	2.690	3.3%
70	2.51	3.887	3.817	0.070	-1.8%	79.5	82.23	2.730	3.4%
70	3.98	4.061	3.998	0.063	-1.6%	78.53	81.35	2.820	3.6%
70	6.31	4.232	4.182	0.050	-1.2%	77.71	80.41	2.700	3.5%
70	10	4.395	4.350	0.046	-1.0%	76.95	79.62	2.670	3.5%

Appendix 4 – RA2b and VB HSM 36h rheology comparison

		LOG Complex Modulus |G*| (Pa)				Phase Angle (°)			
Temp. (°C)	Freq. (Hz)	RA2b	VB HSM 36h	Absolute Differ.	Relative Differ.	RA2b	VB HSM 36h	Absolute Differ.	Relative Differ.
0	0.1	7.553	7.532	0.020	-0.3%	32.84	35.09	2.250	6.9%
0	0.158	7.444	7.611	0.167	2.2%	29.37	33.6	4.232	14.4%
0	0.251	7.690	7.684	0.007	-0.1%	28.43	32.01	3.580	12.6%
0	0.398	7.751	7.753	0.002	0.0%	27.18	30.31	3.130	11.5%
0	0.631	7.811	7.817	0.006	0.1%	25.85	28.81	2.960	11.5%
0	1	7.837	7.879	0.042	0.5%	26.85	27.18	0.330	1.2%
0	1.58	7.923	7.937	0.014	0.2%	23.83	25.78	1.950	8.2%
0	2.51	7.969	7.989	0.020	0.3%	22.66	24.28	1.620	7.1%
0	3.98	8.016	8.038	0.022	0.3%	21.72	23.08	1.360	6.3%
0	6.31	8.060	8.086	0.026	0.3%	21.00	21.77	0.770	3.7%
0	10	8.102	8.127	0.025	0.3%	20.75	20.85	0.100	0.5%
10	0.1	6.940	7.152	0.212	3.1%	43.79	42.4	1.390	-3.2%
10	0.158	7.006	7.240	0.234	3.3%	41.21	40.8	0.410	-1.0%
10	0.251	7.132	7.325	0.194	2.7%	39.12	39.21	0.090	0.2%
10	0.398	7.211	7.407	0.196	2.7%	37.81	37.45	0.360	-1.0%
10	0.631	7.296	7.488	0.192	2.6%	36.60	36.01	0.590	-1.6%
10	1	7.376	7.563	0.186	2.5%	35.19	34.38	0.810	-2.3%
10	1.58	7.453	7.636	0.184	2.5%	34.18	32.86	1.320	-3.9%
10	2.51	7.531	7.706	0.175	2.3%	32.53	31.29	1.240	-3.8%
10	3.98	7.596	7.773	0.176	2.3%	31.72	29.88	1.840	-5.8%
10	6.31	7.665	7.840	0.174	2.3%	30.75	28.47	2.280	-7.4%
10	10	7.729	7.895	0.166	2.1%	29.96	27.39	2.570	-8.6%
20	0.1	6.186	6.437	0.251	4.1%	55.39	53.69	1.700	-3.1%
20	0.158	6.311	6.560	0.249	3.9%	52.52	52.77	0.250	0.5%
20	0.251	6.429	6.677	0.248	3.9%	50.99	51.18	0.190	0.4%
20	0.398	6.532	6.788	0.256	3.9%	49.86	49.53	0.330	-0.7%
20	0.631	6.643	6.897	0.253	3.8%	48.58	47.82	0.760	-1.6%
20	1	6.750	6.999	0.250	3.7%	47.06	46.04	1.020	-2.2%
20	1.58	6.853	7.100	0.247	3.6%	45.66	44.47	1.190	-2.6%
20	2.51	6.952	7.195	0.243	3.5%	44.31	42.85	1.460	-3.3%
20	3.98	7.049	7.287	0.238	3.4%	43.06	41.26	1.800	-4.2%
20	6.31	7.141	7.375	0.235	3.3%	41.95	39.79	2.160	-5.1%
20	10	7.230	7.453	0.224	3.1%	41.01	38.61	2.400	-5.9%
25	0.1	5.768	5.997	0.229	4.0%	60.59	60.51	0.080	-0.1%
25	0.158	5.900	6.134	0.234	4.0%	58.12	58.92	0.800	1.4%
25	0.251	6.027	6.263	0.236	3.9%	57.01	57.49	0.480	0.8%
25	0.398	6.152	6.388	0.236	3.8%	55.46	55.85	0.390	0.7%
25	0.631	6.276	6.510	0.234	3.7%	54.17	54.34	0.170	0.3%
25	1	6.396	6.629	0.232	3.6%	52.82	52.7	0.120	-0.2%
25	1.58	6.513	6.745	0.232	3.6%	51.44	51.11	0.330	-0.6%
25	2.51	6.626	6.856	0.230	3.5%	50.18	49.54	0.640	-1.3%
25	3.98	6.735	6.962	0.227	3.4%	48.87	47.99	0.880	-1.8%
25	6.31	6.841	7.066	0.225	3.3%	47.72	46.47	1.250	-2.6%
25	10	6.943	7.158	0.215	3.1%	46.65	45.2	1.450	-3.1%
30	0.1	5.333	5.552	0.219	4.1%	65.27	64.77	0.500	-0.8%
30	0.158	5.475	5.704	0.229	4.2%	63.00	63.75	0.750	1.2%
30	0.251	5.613	5.841	0.228	4.1%	61.98	63.05	1.070	1.7%
30	0.398	5.749	5.974	0.225	3.9%	60.63	61.55	0.920	1.5%
30	0.631	5.885	6.109	0.225	3.8%	59.40	60.07	0.670	1.1%
30	1	6.016	6.241	0.225	3.7%	58.24	58.71	0.470	0.8%
30	1.58	6.145	6.368	0.223	3.6%	56.89	57.21	0.320	0.6%
30	2.51	6.270	6.491	0.221	3.5%	55.71	55.7	0.010	0.0%
30	3.98	6.392	6.610	0.218	3.4%	54.50	54.29	0.210	-0.4%
30	6.31	6.510	6.728	0.217	3.3%	53.40	52.85	0.550	-1.0%

		LOG Complex Modulus |G*| (Pa)				Phase Angle (°)			
Temp. (°C)	Freq. (Hz)	RA2b	VB HSM 36h	Absolute Differ.	Relative Differ.	RA2b	VB HSM 36h	Absolute Differ.	Relative Differ.
30	10	6.624	6.835	0.210	3.2%	52.42	51.58	0.840	-1.6%
40	0.1	4.528	4.707	0.179	4.0%	72.89	73.48	0.590	0.8%
40	0.158	4.687	4.860	0.172	3.7%	70.89	73.16	2.270	3.2%
40	0.251	4.844	5.021	0.177	3.7%	69.80	71.29	1.490	2.1%
40	0.398	4.999	5.182	0.183	3.7%	68.62	69.98	1.360	2.0%
40	0.631	5.152	5.336	0.184	3.6%	67.43	68.75	1.320	2.0%
40	1	5.303	5.490	0.187	3.5%	66.32	67.51	1.190	1.8%
40	1.58	5.449	5.644	0.195	3.6%	65.19	66.33	1.140	1.7%
40	2.51	5.594	5.790	0.197	3.5%	64.15	65.15	1.000	1.6%
40	3.98	5.735	5.934	0.199	3.5%	63.13	63.95	0.820	1.3%
40	6.31	5.874	6.076	0.203	3.4%	62.09	62.9	0.810	1.3%
40	10	6.007	6.205	0.198	3.3%	61.13	61.84	0.710	1.2%
50	0.1	3.787	3.872	0.085	2.2%	78.10	81.69	3.590	4.6%
50	0.158	3.945	4.070	0.125	3.2%	77.28	79.32	2.040	2.6%
50	0.251	4.126	4.246	0.120	2.9%	75.77	77.99	2.220	2.9%
50	0.398	4.293	4.418	0.125	2.9%	74.39	76.78	2.390	3.2%
50	0.631	4.458	4.588	0.130	2.9%	73.32	75.39	2.070	2.8%
50	1	4.621	4.753	0.132	2.9%	71.87	73.95	2.080	2.9%
50	1.58	4.780	4.917	0.137	2.9%	70.59	72.39	1.800	2.5%
50	2.51	4.936	5.077	0.141	2.8%	69.38	70.87	1.490	2.1%
50	3.98	5.091	5.233	0.142	2.8%	68.11	69.21	1.100	1.6%
50	6.31	5.241	5.385	0.143	2.7%	66.85	67.42	0.570	0.9%
50	10	5.389	5.526	0.137	2.5%	65.48	65.53	0.050	0.1%
60	0.1	3.103	3.139	0.036	1.2%	83.25	84.81	1.560	1.9%
60	0.158	3.290	3.328	0.038	1.2%	81.48	83.76	2.280	2.8%
60	0.251	3.485	3.514	0.029	0.8%	79.39	83.16	3.770	4.7%
60	0.398	3.650	3.700	0.050	1.4%	79.54	82.27	2.730	3.4%
60	0.631	3.834	3.881	0.047	1.2%	78.58	81.12	2.540	3.2%
60	1	4.008	4.059	0.051	1.3%	77.59	80.05	2.460	3.2%
60	1.58	4.180	4.238	0.058	1.4%	76.38	78.9	2.520	3.3%
60	2.51	4.348	4.413	0.064	1.5%	75.37	77.62	2.250	3.0%
60	3.98	4.512	4.581	0.069	1.5%	74.36	76.41	2.050	2.8%
60	6.31	4.675	4.750	0.075	1.6%	73.46	75.31	1.850	2.5%
60	10	4.837	4.908	0.071	1.5%	72.67	74.28	1.610	2.2%
70	0.1	2.491	2.494	0.003	0.1%	86.32	85.51	0.810	-0.9%
70	0.158	2.691	2.678	0.013	-0.5%	84.95	86.2	1.250	1.5%
70	0.251	2.882	2.877	0.005	-0.2%	84.46	85.42	0.960	1.1%
70	0.398	3.074	3.064	0.010	-0.3%	83.69	85.38	1.690	2.0%
70	0.631	3.260	3.256	0.005	-0.1%	82.87	84.47	1.600	1.9%
70	1	3.444	3.444	0.001	0.0%	82.00	83.92	1.920	2.3%
70	1.58	3.627	3.633	0.005	0.1%	80.84	83.1	2.260	2.8%
70	2.51	3.808	3.817	0.009	0.2%	79.94	82.23	2.290	2.9%
70	3.98	3.984	3.998	0.014	0.4%	79.03	81.35	2.320	2.9%
70	6.31	4.159	4.182	0.023	0.6%	78.22	80.41	2.190	2.8%
70	10	4.330	4.350	0.019	0.4%	77.59	79.62	2.030	2.6%
80	0.1	1.935	1.936	0.001	0.0%	88.08	85.62	2.460	-2.8%
80	0.158	2.120	2.131	0.011	0.5%	86.81	84.1	2.710	-3.1%
80	0.251	2.321	2.326	0.005	0.2%	86.16	86.49	0.330	0.4%
80	0.398	2.518	2.512	0.006	-0.2%	85.78	85.08	0.700	-0.8%
80	0.631	2.712	2.700	0.012	-0.5%	85.46	85.66	0.200	0.2%
80	1	2.902	2.888	0.015	-0.5%	84.69	86.12	1.430	1.7%
80	1.58	3.092	3.084	0.007	-0.2%	83.81	85.33	1.520	1.8%
80	2.51	3.279	3.278	0.001	0.0%	82.94	84.91	1.970	2.4%
80	3.98	3.468	3.468	0.000	0.0%	82.04	84.4	2.360	2.9%
80	6.31	3.648	3.652	0.005	0.1%	81.23	83.86	2.630	3.2%
80	10	3.829	3.827	0.001	0.0%	80.62	83.29	2.670	3.3%

Appendix 5 – RA3b and PMB HSM 72h rheology comparison

		LOG Complex Modulus |G*| (Pa)				Phase Angle (°)			
Temp. (°C)	Freq. (Hz)	RA3b	PMB HSM 72h	Absolute Differ.	Relative Differ.	RA3b	PMB HSM 72h	Absolute Differ.	Relative Differ.
0	0.1	8.284	7.332	0.952	-11.5%	20.30	44.9	24.600	121.2%
0	0.158	8.333	7.428	0.905	-10.9%	19.21	50.07	30.860	160.6%
0	0.251	8.380	7.521	0.859	-10.3%	18.11	38.99	20.884	115.3%
0	0.398	8.419	7.595	0.824	-9.8%	17.22	36.13	18.907	109.8%
0	0.631	8.456	7.677	0.779	-9.2%	16.36	35.05	18.692	114.3%
0	1	8.484	7.736	0.748	-8.8%	15.66	34.07	18.405	117.5%
0	1.58	8.520	7.822	0.698	-8.2%	14.85	31.07	16.223	109.3%
0	2.51	8.549	7.885	0.663	-7.8%	14.15	29.13	14.983	105.9%
0	3.98	8.579	7.946	0.633	-7.4%	13.42	27.34	13.918	103.7%
0	6.31	8.609	8.002	0.606	-7.0%	12.69	25.9	13.212	104.1%
0	10	8.635	8.053	0.582	-6.7%	12.01	24.57	12.559	104.6%
10	0.1	7.909	6.545	1.364	-17.2%	27.52	60.29	32.768	119.1%
10	0.158	7.958	6.678	1.280	-16.1%	26.49	56.93	30.435	114.9%
10	0.251	8.014	6.814	1.200	-15.0%	25.38	54.17	28.788	113.4%
10	0.398	8.076	6.919	1.156	-14.3%	24.17	52.7	28.527	118.0%
10	0.631	8.132	7.037	1.094	-13.5%	23.10	50.69	27.593	119.5%
10	1	8.182	7.148	1.035	-12.6%	22.11	48.53	26.420	119.5%
10	1.58	8.232	7.251	0.981	-11.9%	21.12	46.22	25.098	118.8%
10	2.51	8.277	7.348	0.929	-11.2%	20.22	44.4	24.176	119.5%
10	3.98	8.320	7.447	0.872	-10.5%	19.37	42.11	22.740	117.4%
10	6.31	8.362	7.538	0.824	-9.9%	18.51	40.31	21.797	117.7%
10	10	8.403	7.623	0.779	-9.3%	17.68	38.55	20.874	118.1%
20	0.1	7.429	5.555	1.873	-25.2%	36.96	72.53	35.568	96.2%
20	0.158	7.506	5.717	1.790	-23.8%	35.44	70.69	35.249	99.5%
20	0.251	7.574	5.874	1.700	-22.4%	34.15	69.47	35.316	103.4%
20	0.398	7.637	6.030	1.607	-21.0%	32.98	67.88	34.900	105.8%
20	0.631	7.708	6.181	1.527	-19.8%	31.66	66.23	34.568	109.2%
20	1	7.776	6.328	1.448	-18.6%	30.43	64.45	34.020	111.8%
20	1.58	7.843	6.471	1.371	-17.5%	29.20	62.47	33.266	113.9%
20	2.51	7.909	6.609	1.301	-16.4%	28.00	60.51	32.506	116.1%
20	3.98	7.971	6.742	1.229	-15.4%	26.88	58.53	31.648	117.7%
20	6.31	8.030	6.867	1.162	-14.5%	25.80	56.56	30.755	119.2%
20	10	8.085	6.989	1.096	-13.6%	24.79	54.68	29.889	120.6%
25	0.1	7.137	5.008	2.129	-29.8%	42.65	77.11	34.462	80.8%
25	0.158	7.236	5.178	2.058	-28.4%	40.80	75.55	34.751	85.2%
25	0.251	7.329	5.348	1.980	-27.0%	39.09	74.55	35.461	90.7%
25	0.398	7.415	5.514	1.901	-25.6%	37.49	73.48	35.987	96.0%
25	0.631	7.498	5.678	1.820	-24.3%	35.98	72.29	36.309	100.9%
25	1	7.576	5.839	1.738	-22.9%	34.57	70.99	36.421	105.4%
25	1.58	7.652	5.997	1.655	-21.6%	33.20	69.5	36.301	109.3%
25	2.51	7.723	6.152	1.572	-20.4%	31.91	67.99	36.077	113.0%
25	3.98	7.792	6.303	1.490	-19.1%	30.68	66.28	35.600	116.0%
25	6.31	7.859	6.448	1.411	-17.9%	29.50	64.61	35.115	119.1%
25	10	7.923	6.589	1.333	-16.8%	28.35	62.86	34.509	121.7%
30	0.1	6.813	4.483	2.330	-34.2%	48.58	80.69	32.109	66.1%
30	0.158	6.930	4.665	2.265	-32.7%	46.55	79.41	32.858	70.6%
30	0.251	7.037	4.845	2.191	-31.1%	44.74	78.63	33.887	75.7%
30	0.398	7.137	5.023	2.114	-29.6%	43.01	77.68	34.674	80.6%
30	0.631	7.231	5.197	2.034	-28.1%	41.35	76.75	35.400	85.6%
30	1	7.322	5.368	1.954	-26.7%	39.76	75.65	35.885	90.2%
30	1.58	7.408	5.537	1.871	-25.3%	38.24	74.59	36.352	95.1%
30	2.51	7.492	5.703	1.789	-23.9%	36.77	73.44	36.672	99.7%
30	3.98	7.572	5.868	1.704	-22.5%	35.36	72.16	36.800	104.1%
30	6.31	7.649	6.028	1.621	-21.2%	34.01	70.87	36.863	108.4%

		LOG Complex Modulus |G*| (Pa)				Phase Angle (°)			
Temp. (°C)	Freq. (Hz)	RA3b	PMB HSM 72h	Absolute Differ.	Relative Differ.	RA3b	PMB HSM 72h	Absolute Differ.	Relative Differ.
30	10	7.722	6.182	1.540	-19.9%	32.73	69.47	36.745	112.3%
40	0.1	6.127	3.581	2.546	-41.6%	56.20	84.95	28.751	51.2%
40	0.158	6.251	3.772	2.479	-39.7%	54.47	84.33	29.861	54.8%
40	0.251	6.370	3.965	2.406	-37.8%	52.69	83.66	30.967	58.8%
40	0.398	6.486	4.156	2.329	-35.9%	50.87	82.96	32.088	63.1%
40	0.631	6.596	4.344	2.252	-34.1%	48.93	82.01	33.077	67.6%
40	1	6.702	4.525	2.177	-32.5%	46.94	81.27	34.333	73.1%
40	1.58	6.803	4.706	2.096	-30.8%	44.86	80.4	35.542	79.2%
40	2.51	6.898	4.888	2.010	-29.1%	42.71	79.42	36.705	85.9%
40	3.98	6.989	5.070	1.918	-27.4%	40.57	78.28	37.709	92.9%
40	6.31	7.073	5.248	1.825	-25.8%	38.35	76.76	38.405	100.1%
40	10	7.152	5.417	1.735	-24.3%	36.22	75.55	39.326	108.6%
50	0.1	5.402	2.798	2.605	-48.2%	65.09	87.74	22.650	34.8%
50	0.158	5.546	2.992	2.555	-46.1%	63.61	87.35	23.737	37.3%
50	0.251	5.686	3.189	2.497	-43.9%	62.19	87.01	24.824	39.9%
50	0.398	5.823	3.386	2.437	-41.9%	60.75	86.49	25.738	42.4%
50	0.631	5.958	3.580	2.378	-39.9%	59.30	85.75	26.448	44.6%
50	1	6.087	3.770	2.317	-38.1%	57.79	85.16	27.371	47.4%
50	1.58	6.215	3.961	2.254	-36.3%	56.23	84.69	28.461	50.6%
50	2.51	6.338	4.150	2.188	-34.5%	54.60	84	29.404	53.9%
50	3.98	6.458	4.336	2.122	-32.9%	52.88	83.56	30.680	58.0%
50	6.31	6.573	4.521	2.052	-31.2%	51.07	83	31.930	62.5%
50	10	6.684	4.704	1.981	-29.6%	49.16	82.58	33.423	68.0%
60	0.1	4.672	2.091	2.581	-55.2%	73.15	89.39	16.240	22.2%
60	0.158	4.833	2.284	2.549	-52.7%	71.57	88.94	17.371	24.3%
60	0.251	4.990	2.484	2.506	-50.2%	70.03	88.07	18.041	25.8%
60	0.398	5.144	2.687	2.457	-47.8%	68.56	88.45	19.887	29.0%
60	0.631	5.294	2.885	2.409	-45.5%	67.14	87.69	20.551	30.6%
60	1	5.441	3.080	2.361	-43.4%	65.78	87.05	21.267	32.3%
60	1.58	5.586	3.275	2.311	-41.4%	64.43	87.05	22.617	35.1%
60	2.51	5.728	3.473	2.255	-39.4%	63.11	86.55	23.442	37.1%
60	3.98	5.867	3.665	2.202	-37.5%	61.77	86.29	24.518	39.7%
60	6.31	6.003	3.857	2.146	-35.7%	60.39	85.84	25.449	42.1%
60	10	6.136	4.045	2.091	-34.1%	58.96	85.59	26.627	45.2%
70	0.1	3.954	1.567	2.387	-60.4%	80.22	89.43	9.207	11.5%
70	0.158	4.132	1.775	2.357	-57.0%	78.73	89.58	10.854	13.8%
70	0.251	4.306	1.979	2.327	-54.1%	77.24	88.9	11.658	15.1%
70	0.398	4.476	2.178	2.299	-51.4%	75.69	89.31	13.621	18.0%
70	0.631	4.643	2.377	2.266	-48.8%	74.17	88.02	13.852	18.7%
70	1	4.806	2.584	2.222	-46.2%	72.69	88.73	16.040	22.1%
70	1.58	4.965	2.781	2.185	-44.0%	71.25	88.01	16.757	23.5%
70	2.51	5.122	2.970	2.151	-42.0%	69.88	88.05	18.169	26.0%
70	3.98	5.275	3.179	2.096	-39.7%	68.56	88.51	19.951	29.1%
70	6.31	5.426	3.373	2.053	-37.8%	67.28	88.01	20.728	30.8%
70	10	5.574	3.564	2.010	-36.1%	66.03	88.52	22.490	34.1%
80	0.1	3.268	1.075	2.193	-67.1%	85.11	89.04	3.934	4.6%
80	0.158	3.458	1.314	2.143	-62.0%	84.03	86.17	2.142	2.5%
80	0.251	3.645	1.517	2.128	-58.4%	82.86	87.64	4.780	5.8%
80	0.398	3.829	1.688	2.141	-55.9%	81.58	88.19	6.607	8.1%
80	0.631	4.009	1.900	2.109	-52.6%	80.25	87.35	7.102	8.9%
80	1	4.187	2.105	2.082	-49.7%	78.86	88.78	9.923	12.6%
80	1.58	4.361	2.304	2.057	-47.2%	77.44	87.8	10.358	13.4%
80	2.51	4.531	2.496	2.035	-44.9%	76.00	85.54	9.535	12.5%
80	3.98	4.698	2.706	1.992	-42.4%	74.60	88.74	14.143	19.0%
80	6.31	4.862	2.901	1.961	-40.3%	73.23	88.73	15.495	21.2%
80	10	5.023	3.088	1.934	-38.5%	71.94	89.1	17.156	23.8%