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Thank you all.

STATEMENT OF INTEGRITY

I hereby declare having conducted this academic work with integrity. I confirm that I have not used plagiarism or any form of undue use of information or falsification of results along the process leading to its elaboration.

I further declare that I have fully acknowledged the Code of Ethical Conduct of the University of Minho.

Influência da modificação de misturas betuminosas no seu comportamento a baixas temperaturas

RESUMO

Os regulamentos nacionais têm sido substituídos por regulamentos europeus nos vários países da comunidade europeia. Esta progressiva uniformização das normas europeias exige que as empresas nacionais submetam os seus produtos a ensaios padronizados na Europa, mas permite melhorar os produtos para responder à procura de novos mercados, expandindo o comércio das empresas. Neste contexto, as empresas portuguesas podem diversificar o seu portfólio e investir em produtos destinados a regiões mais frias, que em geral são mais prósperas. No entanto, estas regiões têm um clima muito diferente da realidade nacional, com invernos mais frios e longos, para o qual há pouca experiência. Quando as temperaturas descem para níveis muito baixos, os pavimentos são submetidos a tensão térmica e podem apresentar várias degradações, como fendilhamento térmico. Nestes casos, podem ser usados polímeros para modificar o betume e melhorar algumas das suas propriedades, como a recuperação elástica, coesão e ductilidade. Os polímeros também reduz alguns dos problemas das misturas betuminosas, como o fendillhamento térmico e por fadiga e a deformação permanente.

O objetivo deste trabalho foi estudar o comportamento das misturas betuminosas a baixas temperaturas, em particular quando se utilizam betumes modificados. Assim, foram selecionados e ensaiados três ligantes neste estudo: um betume convencional com penetração 50/70 e dois betumes modificados com polímeros (PMB) obtidos pela adição de 2,5% e 5,0% de SBS, respetivamente, ao betume 50/70. Em seguida, os PMB foram então incorporados em misturas do tipo SMA 11, que foram submetidas a ensaios mecânicos a baixas temperaturas com base na norma europeia EN 12697-46. Os materiais e os métodos utilizados serviram para se definir quais as melhores alternativas futuras para climas frios. Algumas das propriedades dos ligantes e das misturas betuminosas que foram avaliadas foram a resistência ao fendilhamento térmico, fluência, recuperação elástica, força coesiva e ductilidade.

Em geral, concluiu-se que as misturas betuminosas avaliadas neste trabalho tiveram um desempenho adequado a baixas temperaturas, em particular as produzidas com PMB. O conhecimento dos ensaios de laboratório a baixas temperaturas (EN 12697-46) e suas conclusões inerentes ajudarão a desenvolver novas soluções de pavimentação para as zonas climáticas mais frias.

Palavras-chave:

Baixas Temperaturas; Betume Modificado com Polímeros (PMB); Estireno-Butadieno-Estireno (SBS); *Stone Mastic Asphalt* (SMA); Norma Europeia; Desempenho das Misturas Betuminosas.

Influence of the modification of asphalt mixtures on their behaviour at low temperatures

ABSTRACT

The national regulations have been replaced by European regulations in the several countries of the European Community. This progressive uniformisation of European standards require the national companies to submit their products to standardised tests validated in Europe, but enables the development of improved products to meet the demand of new markets, thus expanding business trade. In this context, Portuguese companies can diversify their portfolio and invest in products aimed at colder regions, which in general are more prosperous. However, these regions have a climate different from the national reality, with colder and longer winters, in which our companies have little experience. When temperatures drop to significantly low levels, pavements are subjected to thermal stress and can present several distresses, such as thermal cracking. In these situations, polymers can be used as modifiers in asphalt binders to improve their properties, such as elastic recovery, cohesion and ductility. Polymers also minimize some of the problems of asphalt mixtures, such as thermal and fatigue cracking and permanent deformation.

The objective of this work was to study the behaviour of asphalt mixtures at low temperatures, in particular when using modified bitumens. Thus, three binders were selected and tested in this study: a standard 50/70 penetration grade bitumen, and two polymer-modified binders (PMB) obtained adding 2.5% and 5.0% of SBS, respectively, to the 50/70 pen grade bitumen. Then, the PMBs were incorporated into SMA 11 mixtures, which were subjected to low-temperature mechanical tests based on the European Standard EN 12697-46. The materials and methods used in this work were applied to support the definition of the best future alternatives for cold climates. Some of the properties of the asphalt binders and mixtures evaluated in this work were the thermal cracking resistance, creep, elastic recovery, cohesive strength and ductility strength.

Overall, it is concluded that the asphalt mixtures produced and evaluated in this work performed adequately at low temperatures, in particular those with PMB. The knowledge regarding laboratory tests at low-temperatures (EN 12697-46) and their derived conclusions will help to develop new pavement solutions for those colder climate zones.

Keywords:

Low-temperature; Polymer Modified Binder (PMB); Styrene-Butadiene-Styrene (SBS); Stone Mastic Asphalt (SMA); European Standard; Asphalt Mixture Performance.

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LIST OF ACRONYMS

ABS – acrylonitrile-butadiene-styrene

AC – Asphalt Concrete

BD – Bulk density

CV – Coefficient of variation

DSR - Dynamic Shear Rheometer

EVA – Ethylene-vinyl-acetate

LVDT – Linear Variable Differential Transformer

PMB – Polymer modified bitumen

PMB25 – Polymer modified bitumen with 2.5% SBS

PMB50 – Polymer modified bitumen with 5.0% SBS

PB – Polystyrene

PS – Polybutadiene

RET – Reactive elastomeric terpolymer

RT – Relaxation test

SBS – Styrene-butadiene-styrene

SBR – Styrene-butadiene-rubber

SD – Standard deviation

SIR – Styrene isoprene rubber

SMA – Stone Mastic Asphalt

TCT – Tensile creep test

TSRST – Thermal stress restrained specimen test

UCTST – Uniaxial cyclic tensile stress test

UTST – Uniaxial tension stress test

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1. INTRODUCTION

"Adapt what is useful, reject what is useless, and add what is specifically your own."

Bruce Lee

1.1. Scope

Transport infrastructures are subjected to a constant evolutionary process, a consequence of the increase in traffic, the variation in the type of traffic and the increasing demand for safety, quality, comfort and sustainability standards of its pavements. The cycle of interdependence between the evolution of technology and the evolution of the infrastructure itself is of great importance. The evolution of technology permits the development and improvement of knowledge, techniques and materials that produce roadways, which in turn makes those characteristics evolve and require new technologies to allow for their improvement and consequently the creation of new methods and materials.

Based on these premises and a set of established knowledge, the scientific community decided to provide a methodological answer to these cyclical needs. Therefore, specific procedures, including standards and guidelines, were established to evaluate the materials' characteristics, and the processes to be followed for sampling, and laboratory and "in situ" testing.

In Portugal, previous national regulations have been, or will soon be, replaced by European regulations, as in many other countries of the European Community. This progressive replacement and consequent uniformization of standards in several European countries allow their national companies to be able to subject their current products and services to this standardization, which is recognized and validated by the vast majority of these states. Besides, those companies can now develop new and improved products in order to meet the demand of previous unexplored markets and, thus, expand their trade.

Additionally, Portuguese companies must not be so dependent on the national construction market and its economic fluctuations and should diversify their product portfolio and invest in types of products aimed at colder international regions. Those regions are economically prosperous, in the case of Northern Europe, or expanding markets in developing economies, in the case of Central and Eastern Europe. These nations have different climates in comparison to the national scenario, in which our companies have little experience.

These climate zones are usually characterized by humid, long-lasting cold winters in which there is usually heavy snowfall and mild, humid summers for the Scandinavia region. For the Central and Eastern European regions, the climate is usually explained by long cold winters, with the significant presence of frost and snow, and predominantly hot summers (EC, 2019, WorldAtlas, 2019).

During these winters, when temperatures drop to significantly low levels, the pavements are subjected to thermal stress, and as a result, cracking of the asphalt layers can occur. This cracking results from the retraction of the pavement given the temperatures to which it is exposed. Cracking can also occur as a result of thermal fatigue to which road surfaces are exposed in the alternating cycle of heating to cooling while cracking by extremely low-temperatures is a primary problem in icy regions (Alataş and Yilmaz, 2017). Cracking due to thermal susceptibility at low temperatures can additionally result from the retraction of binders, which occurs when their maximum tensile strength is reached. Open cracks allow water to penetrate, which gradually freezes and expands and causes a partial destruction of the bituminous layer due to this volumetric expansion (Pangarova and Nikolov, 2016).

Polymeric materials can be used as additives in asphalt mixtures so that their characteristics can be improved and they can achieve better performance (Polacco *et al.*, 2008). Some thermomechanical properties of asphalt binder targeted in this work, such as elastic recovery, cohesion and ductility, are potentially improved by using polymer modified binders (PMBs). PMBs can also minimize some of the main problems of asphalt mixtures, such as permanent deformation and cracking due to thermal susceptibility and bitumen ageing (Fernandes *et al.*, 2016).

The stresses that occur in the bituminous layers derived from the temperature during cooling are quite challenging to measure directly in the pavement structure. Therefore, the correct analytical estimate is crucial, as well as the results obtained through laboratory tests. It is then possible to compare the analytical design with the empirical results and calibrate and rectify both (Pszczola *et al.*, 2019a).

1.2. Objectives and contributions

The main objective of this dissertation is to evaluate the influence of asphalt binder and its modification in the low-temperature performance of asphalt mixtures. Another primary goal is to verify the applicability of laboratory methods and techniques to obtain the properties of these mixtures at cold temperatures. These objectives derive mainly from an attempt to assess the influence of the type of asphalt binder and mixture composition on the tensile strength properties of asphalt mixtures and strength reserve, as well as the influence of cooling rate on strength, elastic recovery and the behaviour of these mixtures when they are subjected to cyclic fatigue stress.

By analysing the traditional types of asphalt mixtures used generally in European regions with colder climates, as well as their characteristics and behaviour at low temperatures, the Stone Mastic Asphalt (SMA) mixture was selected for this study as an excellent example of those mixtures. Moreover, that mixture is used both in Portugal and in those colder regions.

From the extensive literature review, it was also decided to study the effect of one of the typical polymers used to modify asphalt binders, Styrene-butadiene-styrene (SBS), in two different proportions, in order to analyse its effect on the workability, and resistance to deformation and cracking at low temperatures.

Several laboratory tests were performed to analyse the unmodified bitumen and the two polymer-modified binders, as well as the corresponding asphalt mixtures. The low-temperature performance was analysed through the tests contained in the European Standard EN 12697-46 (CEN, 2012) for low-temperature cracking properties by uniaxial tension tests, but many other EN standards were used to evaluate other properties of the binder and the mixtures. However, the first one will be of particular importance, given the limited national experience in the low-temperature performance of asphalt mixtures until now. The feasibility of carrying out the selected laboratory tests (laboratory restrains) will also be assessed, as well as the calculations and final analysis of the characteristics of the binder and mixtures.

This dissertation should contribute to depict as closely as possible the real conditions and behaviour to which asphalt pavements are subjected to and expand the current knowledge about the variables involved in the complete process that occurs when low temperatures are present. Subsequently, this work aims to add some knowledge on the response to challenges transport infrastructures face in regions with cold climates, both at the national level (Northeast of Portugal) but particularly at the European level, to facilitate and encourage the expansion and participation of Portuguese construction companies in an increasingly global market.

1.3. Dissertation structure

This dissertation is structured into five distinct chapters. This distribution was established following the main phases of the study and the timeline in which they were executed. Its primary purpose is to better clarify and translate the different components of the research into a flawless and objective report.

The first chapter, the current one, serves as an introductory segment. It conveys the general context in which the research was brought up as well as clear objectives of the work and broad parameters.

Chapter two entails the literature review. Different components of asphalt mixtures are discussed in this chapter to contextualize this research and the advantages and disadvantages of their application under the studied conditions. The temperature conditions this research involves are also analysed.

The third chapter describes the materials utilized in the production of asphalt binders and mixtures, as well as the material used as the bonding agent for the mechanical tests. In this chapter, the methods and techniques used to produce and characterize the asphalt mixtures and their components over the various phases of the study are also detailed and further explained. Besides, the processes for characterization of the binders are also described. Lastly, special attention is given to the mechanical procedures used to study the low-temperature properties of the asphalt mixtures.

The fourth chapter presents the analysis of the results obtained from the previously mentioned methods and tests, starting with the constituents of the asphalt mixtures and their respective assessments: the aggregates and the asphalt binders. Then, an evaluation of the asphalt mixtures performance is carried out, and finally, their mechanical properties are analysed.

In the last chapter, chapter five, the conclusions of the research are presented, based on the results and analysis obtained from the previous chapter. In this chapter, it is also further explained the particular challenges that arose during the study and its limitations. Future works are suggested in order to advance the research further after this dissertation report.

2. LITERATURE REVIEW

"If I have seen further, it is by standing on the shoulders of giants."

Isaac Newton

2.1. Asphalt mixtures

2.1.1. General properties of asphalt mixtures

Since immemorial times, the movement of people and goods has been directly related to human and civilizational development and the increase in the quality of life. These paths, initially paths traced by pedestrian movement, have also evolved in number, in their shape, layout, location and quality of the materials used for their execution, as the means of transport and technology have evolved around them.

The development of a nation is extensively dependent on the connectivity between its various places of importance, whether they are urban centres, which concentrate populations and services, or places of strategic importance for their function of production or development of resources. This connectivity is provided by transport infrastructures, which are of extreme importance in the development of any region, let alone, any nation.

The benefits from the investment in the road sector are indirect, long-term and not immediately visible. Roads are essential assets for any nation. In the past, gravel road surfaces, cobblestone and granite setts were extensively used, but these surfaces have mostly been replaced by asphalt or concrete pavements.

Pavement design is one of the significant components in road construction. When building highways and roads, the pavement consists of a structure composed of one or more courses, to assist the passage of traffic with layers of finite thickness on a support surface obtained by earthworks (Senço, 2001). The primary function of a pavement is to resist and distribute to the foundation the vertical loads produced by the traffic; a second function is to improve the rolling conditions in terms of comfort and safety, also by resisting the horizontal efforts that act on it, creating a more durable rolling surface, allowing the circulation of vehicles in a safe, comfortable and economical way.

The carrying capacity is a function of the load distribution characteristics of the layer system. The layers with the higher quality are closer to the applied load, favouring the waterproofing of the pavement and preventing water from entering the lower layers of the foundation and thus, preventing stability issues related to the presence of water.

The road pavement can be categorized into several types, according to its constitution and mode of behaviour: flexible, rigid and semi-rigid (Branco *et al.*, 2011).

Rigid pavements are those possessing a considerable flexural strength of flexural rigidity. Usually, the surface layer is constituted by cement concrete. A rigid payment derives its capacity to withstand loads from flexural strength. In these types of pavements, thicknesses are obtained from the flexural strength of concrete slabs and the compressive strength of the underlying layers. The rigid pavement has rigidity and a high stiffness modulus in order to distribute the load over a relatively wide area of soil. The underlayer of the concrete slab is conventionally designated as base course, as shown in Figure 1.

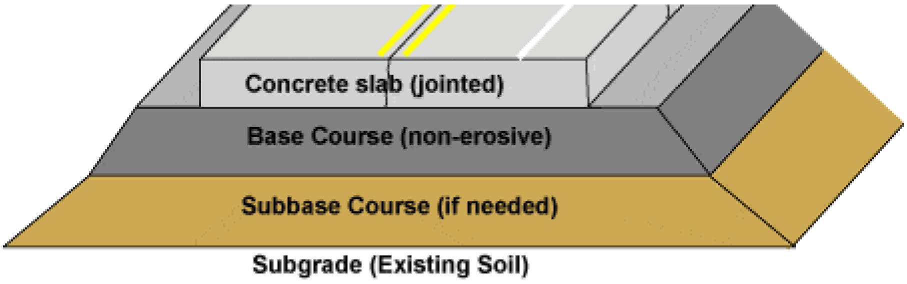


Figure 1 – Rigid pavement cross-section (The Constructor, 2020)

Cement concrete pavement is the best example of rigid pavements. This type of pavement has some advantages, which include: low maintenance costs, very long life, high value as a potential base for future resurfacing with asphalt, load distribution over a wide area, decreasing base and subgrade requirements, ability to be placed directly on poor soils, little to no damage from oils and greases and firm edges. Despite these benefits, it does also present some considerable challenges: high initial costs, the requirement of installation of joints for contraction and expansion, generally a rough-riding quality and high repair costs.

Flexible pavements are those with low flexural strength and are flexible in their structural behaviour under the wheel loads. Because of its low flexural strength, this type of pavement deforms if the subgrade deforms, so a well compacted granular structure is an essential characteristic in order to obtain a flexible pavement of good quality. Flexible pavements (Figure 2) are formed by three main layers: asphalt layers, granular layers (base and sub-base) and reinforcement of foundation or pavement bed. These layers are built over the subgrade soils or foundation of the pavement.

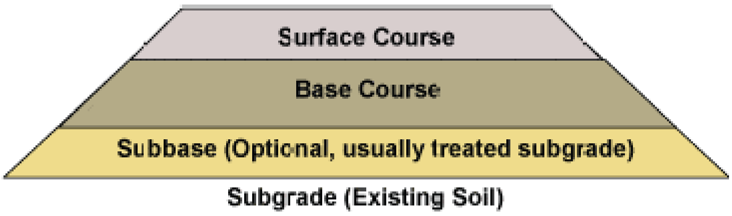


Figure 2 – Flexible pavement scheme (The Constructor, 2020)

The asphalt layers are composed of bituminous mixtures, also called asphalt mixtures, consisting basically of aggregates and asphalt binders. These mixtures are utilized in the surface or wearing course layer that sits in direct contact with the vehicle wheels, in the intermediate or binder layer and the base layer. Flexible pavements are designed based on the principle that the intensity of a load diminishes as it is transmitted downwards from the surface by spreading over an increasingly more substantial area. The load is carried deep enough into the ground by the use of successive layers of granular material. These traffic loads are transmitted to the successive lower layers by grain to grain transfer. In sum, the wear layer is designed to resist the actions of traffic directly and transmit them smoothly to the lower layers, in addition to waterproofing the pavement and improving rolling conditions, that is, comfort and safety (Bernucci *et al.*, 2010).

Flexible pavements offer some great benefits, which include: adaptability to construction in stages; availability of lower-cost pavements that can be quickly built; ability to be easily opened and patched; frost heave and thaw settlement can be easier to repair than in rigid or semi-rigid pavements, and; they possess a higher resistance to the formation of ice glaze. Albeit these benefits, they also pose some issues in the form of higher maintenance costs, shorter life span under heavy use, potential damage by oils and certain chemicals and weak edges that may require curbs or edge devices. The bituminous pavements and gravel pavements are some examples of flexible pavements.

Semi-rigid pavements are a transitional state between flexible and rigid pavements. This type of pavement possesses some traits of both of the other two. From the rigid pavement, it borrows the flexural strength, although in a much lower capacity. However, to compensate for this lower strength capacity, it derives support by the lateral load distribution given by the flexible pavement. Lean cement concrete, soil cement and lime-pozzolanic concrete construction are examples of semi-rigid pavements.

A vast portion of roadways and highways are constructed with flexible pavements because of their advantages in low capital cost, high riding quality and ease of construction. Thus, a great deal of research has been done concerning the asphalt mixtures utilized in these pavements, since they are the main component in this configuration. The reason for the vast and prolific analysis, research and development of asphalt mixtures lies in the fact that with its development and improvement, specific challenges presented by flexible pavements can be surmounted or at least, much improved. The current study will also focus on asphalt mixtures for flexible pavements.

As previously stated, asphalt mixtures consist of aggregates of different sizes and gradings, which are mixed with a certain amount of binder, so that the desired characteristics for each bituminous layer of the pavement can be attained. The aggregates used for asphalt mixtures can be crushed rock, sand, gravel or slag along with particular waste and by-products resulting from the construction and demolition fields. The latter ones can contribute to increasing the sustainability of the asphalt mixture. The matrix material is the binder, and this substance is usually a black viscous mixture of hydrocarbons obtained naturally or as a residue from petroleum distillation, in which case is called bitumen. Nowadays, different binders not derived from petroleum are being developed, such as bio-based binders, to minimise the environmental impact the construction of roads has (EAPA, 2019).

There are several types of asphalt mixtures capable of responding to the most varied demands required by traffic loads and weather agents. These mixtures can be classified in different ways. One of the categories for classification concerns the temperature of production. They can be divided into four classes, with increasing production temperature: cold mix asphalt (CMA), half warm mix asphalt (HWMA), warm mix asphalt (WMA) and hot mix asphalt (HMA). CMA mixtures are made at room temperature using bitumen emulsion or foam, and HWMA is produced with similar materials at temperatures slightly below water vaporization. WMA is made in the 120 to 140 °C range, above the water vaporization temperature but below HMA production temperature. Finally, HMA is mixed up in the 150 to 180 °C temperature range depending on the used binder (Vaitkus *et al.*, 2009). This classification is represented in Figure 3. This research will use HMA mixtures since they are the most commonly used type of mixtures.

Asphalt mixtures are mostly used either in the surface course or in the binder course. This study will focus on mixtures to be used in the surface course. The surface or wearing course consists in the top layer of the pavement and must be able to withstand high traffic and stresses derived from the environment around it without the asphalt mixture exhibit unsatisfactory cracking and rutting (EAPA, 2019). In order for the mixture to provide comfort for the user and assure adequate skid resistance, it should possess specific parameters and characteristics such as, an even profile, suitable texture that allow for noise reduction, among others.

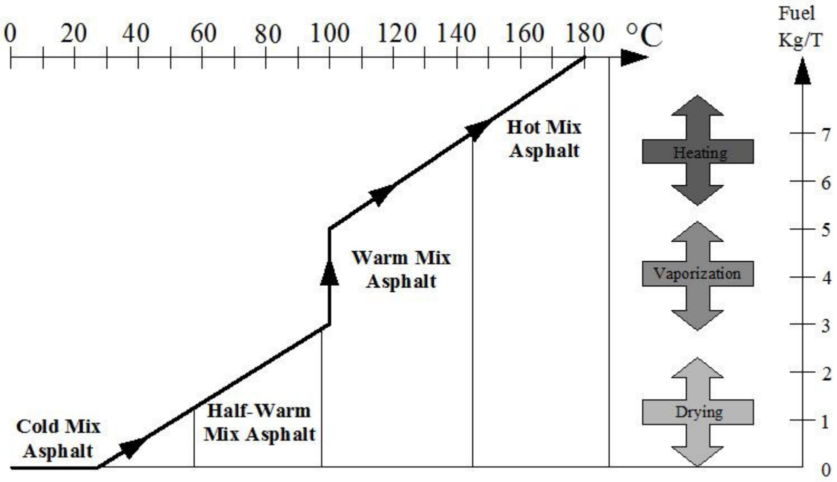


Figure 3 – Classification of asphalt mixtures according to production temperature (Vaitkus *et al.*, 2009)

Different types of asphalt mixtures that attain to provide solutions to these requirements, among others, have been created over time and a great deal of research and investigation has been put into their development. Some of these mixtures are asphalt concrete (AC), *béton bitumineux mince* (BBM) or thin layer asphalt concrete (AC-TL), very-thin layer asphalt concrete (AC-VTL), ultra-thin layer asphalt concrete (AC-UTL), stone mastic asphalt (SMA), hot rolled asphalt (HRA), porous asphalt (PA), mastic asphalt (MA), among others (EAPA, 2019). Figure 4 shows visual differences in aggregate distribution and porosity between commonly used asphalt mixtures.

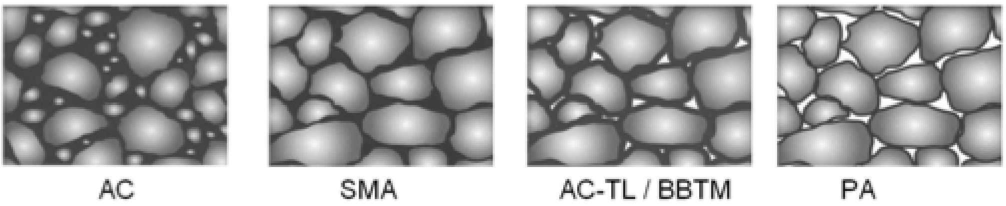


Figure 4 – Difference in the structure of typical asphalt mixtures (EAPA, 2019)

2.1.2. Stone Mastic Asphalt

This work will study a Stone Mastic Asphalt (SMA) mixture for the surface layer. SMA type of mixtures was developed in Germany in the 1960s. At the time, studded tyres were abundant in Germany, which in conjunction with the loads from the heavy vehicles that frequented the roads in abundance, became an increasing issue concerning the low durability of the pavements (Blazejowski, 2011). SMA mixtures were created to overcome this issue, providing heavily trafficked roads with a durable, rut-resistant wearing course by using a gap-graded aggregate structure and a high content of modified binder.

Due to the vast experience, Germany has had with SMA type mixtures, several empirically specifications based on compositional recipes have been put forward since the technical ZTV bit Stb-84 was initially published in 1984 (Miranda *et al.*, 2019). Since then, the use of this type of asphalt mixture spread throughout the world. In the United States, SMA mixtures have been in use since the early 1990s. In both continents, these mixtures have performed well for many years, and they continue to be used when specific characteristics are required for the pavement (Brown *et al.*, 1997).

Currently, the European Standard EN 13108-5 (Bituminous mixtures - Material specifications - Part 5: Stone Mastic Asphalt) provides some requirements to SMA properties, including volumetric characteristics, resistance to permanent deformation and others.

SMA is an asphalt mixture which is mainly characterized by a discontinuous gradation of the mineral particles, which results in a coarse aggregate skeleton. This stone on stone effect provided by the skeleton does not occur in the more conventional dense mixtures characterized by continuous particle gradation. The bituminous mastic has the requirement of filling the air voids volume presented between the coarse aggregate to be able to ensure excellent cohesion of this skeleton. This mastic is usually constituted by fine aggregates, filler, asphalt binder and sometimes stabilisers (Miranda *et al.*, 2019).

According to EAPA (2018), the binders utilized in these types of mixtures are usually polymer-modified bitumens and standard bitumens with fibres, the latter selected on lower traffic volume roads.

This mixture design has usually been based on empirical specifications in order to achieve the stone on stone effect between the coarser aggregate and thus, ensuring adequate performance to permanent deformation. This process has been iterative, by continuously varying the particle grading, until obtaining a minimum binder content that can still ensure adequate porosity and minimum voids in the mineral aggregate, along with additional performance-related requirements (Miranda *et al.*, 2019). The performance of these mixtures is mostly related to its skeleton effect, which can affect its essential properties, such as rutting resistance and workability.

SMA possesses several benefits because it can reduce light reflection, improve surface drainage and reduce the level of traffic noise. With its discontinuous skeleton, it can offer an improved dispersion of the traffic loads and thus, prevent rutting and increase resistance to wearing. Likewise, it also provides good macrotexture and high surface roughness, which contribute to improving skid resistance (Blazejowski, 2011). With the experience over the years, it has also been found that stripping, surface cracking (either by temperature fluctuation or induced by traffic loads), and ravelling, do not present severe issues in this type of mixture. The excellent durability of SMA mixtures derives from the impervious nature of the mastic mortar, which is very rich in asphalt binder and presents a lower air voids content. The improved performance of SMA mixtures is also justified by the general use of stabilizing agents such as fibres and bitumen modifiers. All these qualities result in an asphalt mixture that possesses an extremely low potential for deterioration (EAPA, 2018).

However, SMA mixtures retain some drawbacks, including the potential for the presence of fat spots and bleeding, which can be solved by the use of fibres. Another consideration is its costs because of the materials used in SMA mixtures, particularly the high amount of binder and use of additives, resulting in higher costs than those of other typical mixtures (Shekar *et al.*, 2018). Despite the initial investment cost, the return can be positive since the initial investment can be recovered in savings on repairs and rehabilitation due to its higher durability. Other disadvantages of this mixture are, in its initial phase, its lower resistance to sliding, and some exudation problems can also occur, these derived from errors in the production or application of the asphalt mixture (Blazejowski, 2011).

In Europe, the most used types of SMA are SMA 8, SMA 11 and SMA 16. In Germany and the Netherlands, SMA 6, SMA 8 and SMA 11 are generally the most used. The United Kingdom prefers SMA 6, SMA 10 and SMA 14. Sweden usually opts for SMA 4, SMA 8, SMA 11 and SMA 16 and, in general, Nordic countries prefer the use of SMA 16 in order to increase the resistance to studded tyres, which are in need considering the winters have a lot of snow and frost (EAPA, 2018). Therefore, SMA 11 is significantly used by nations in colder regions, which is one of the reasons to select this type of mixture to carry out the current research work. According to Judycki (2014), SMA is becoming the most popular wearing or surface course on newly constructed main roads in Europe.

2.2. Polymers

A polymer by definition is a natural or synthetic substance composed of very large molecules, called macromolecules, that are multiples of simpler chemical units called monomers (Encyclopaedia Britannica, 2019). The etymology of the word refers from the Greek polymerēs, having many parts, from *poly-* + *meros*, part (Encyclopaedia Britannica, 2019).

They are, in short, substances made of many small molecules bonded together in a repeating sequence. Industrial polymers are synthesized from simple compounds joined together to form long chains.

The first synthetic polymer, although derived from a natural polymer, was invented in 1869 by John Wesley Hyatt of the Hyatt brothers, who was attempting to develop a substitute for ivory, fuelled by a New York firm's offer of $10000 for anyone who could achieve this innovation (SHI, 2019). Hyatt mixed pyroxin made from cotton, a natural polymer, and nitric acid with camphor, and this mixture resulted in cellulose nitrate, which he called celluloid (Ebewele, 2000).

His work came about after the work of many others in discovering and perfecting the process of manufacturing polymers of natural origins, such as Charles Goodyear with his development of various types of rubbers, including vulcanized rubber, from 1834 onwards, Eduard Simon with the accidental discovery of polystyrene in 1839 (Scheirs, 2003) and Alexander Parks with the discovery of the process to obtain waterproofing woven fabrics with an almost plastic-like quality in the 1850s (Encyclopaedia Britannica, 2019).

It was a slow pace in the journey of the development of polymers up until the 1920s. However, it is worth noticing the invention in 1907 of the first genuinely human-made plastic, containing no molecules found in nature, a thermosetting phenol-formaldehyde resin, patented in 1909 by Leo Hendrick Baekeland which he called Bakelite, having found many uses such as electric iron and cookware handles, grinding wheels and electrical plugs. Other polymers soon followed, cellulose acetate, urea-formaldehyde, poly (vinyl chloride) and nylon. The pace of development quickly picked up momentum throughout the 1930s, during which acrylic resins, polystyrene, and melamine resins were introduced, and the 1940s, which took a massive incentive for the research into new polymers derived from the World War II defence efforts, this resulted in the development of polyethene, thermosetting polyester resins and terpolymer acrylonitrile-butadiene-styrene (ABS), among others (Ebewele, 2000).

Following the wartime period, a period of peace, stability and progress soon was reached, and the 1950s saw the development of the so-called engineering thermoplastics, such as the families of acetal and polycarbonates, nylon, phenoxy, polyimide, poly (phenylene oxide), and polysulfone. These types of materials possessed a much higher strength, thermal and dimensional stability, making them able to compete directly with more conventional materials, like metals. The subsequent decades were greatly influenced by the aerospace and aircraft industries and saw the introduction of new polymers such as thermoplastic polyesters, high-barrier nitrile resins and high-temperature plastics like polyphenylene sulphide and polyethersulfone.

In the more recent decades, the concept of a genuinely tailor-made polymer has turned from wish to reality, as a result of higher and more profound knowledge of the relationships between its structure and properties, the discovery and research of new polymerization techniques and the increased availability of new monomers as well as its reduced cost.

Today it is possible to create polymers from different elements with qualities extraordinarily varied. Their advantages rely on higher economic value but with characteristics similar to conventional materials for some polymers. For other polymers, they represent a significant improvement over existing materials and even for others, they present unique characteristics previously unseen in other materials. Polymers can be created in the form of solid plastics, elastomers, fibres and foams, they can be soft or hard, they can be presented as films, adhesives or coatings, and they can have many other qualities and properties. Polymers, today, hold almost an endless array of applications and purposes (Ebewele, 2000).

In this context, polymers are widely used as modifiers for binders. They include a broad range of subclasses that can meet the demands imposed by asphalt mixtures requirements. Some of these subclasses include elastomers and plastomers, these being some of the most commonly used types of bitumen modifiers (Porto *et al.*, 2019).

2.2.1. Styrene-butadiene-styrene

Styrene-butadiene-styrene or SBS is a block copolymer of styrene and butadiene. A block copolymer is a polymer containing molecules in which there is a linear arrangement of blocks (Quirk *et al.*, 1989). These types of polymers comprise alternating segments of different compositions, which are linked together through their reactive ends. In the copolymer, while polystyrene (PS) blocks impart strength to the resin, soft polybutadiene (PB) blocks provide elasticity (Airey, 2003).

This structure of PB blocks can be adjusted by the use of special catalysts that partially transfer the double bonds on the PB blocks to the side chains. According to Vonk *et al.* (2010), this modification can provide many advantages for the PMB, such as low viscosity, better compatibility at equivalent molecular weight, resistance to oxidation and thermal stability.

SBS can also be classified as either linear or radial. A radial block copolymer presents much higher values of viscosity and softening point for the same addition of polymer. These characteristics make radial copolymers more challenging to mix with bitumen, in comparison with linear copolymers.

Another classification made to SBS is its thermoplastic nature. It can be considered a thermoplastic elastomer, and it is characterized by its glass transition point. One of the primary advantages of elastomer modification is that the effectiveness of the cross-links diminishes rapidly above the glass transition point of the polystyrene, at about 100 °C. On cooling, the polystyrene domain reforms and, thus, the strength and elasticity are restored (Mandal *et al.*, 2015). SBS absorbs the maltenes in bitumen, swells, and at higher dosages, forms a continuous molecular network, which makes up a significant fraction of the bitumen. These networks provide an increase in the elastic component of the binder, which, in turn, permits a better recovery after deformation (SABITA, 2015).

In sum, the advantages provided by the use of SBS copolymer in bitumen modification are essential in the current work due to its higher flexibility at low temperatures, because the addition of SBS improves the flexibility of the bitumen which inhibits cracking and improves the resistance of the binder to crack reflection. Besides, SBS modification assures better flow and rutting resistance at high temperatures, improved strength and excellent elasticity. However, it presents some challenges, such as higher cost, reduced penetration resistance, higher viscosity at compaction temperatures, lower resistance to heat and oxidation comparable to polyolefins (Becker, 2001).

2.2.2. Styrene-butadiene-rubber

Styrene-butadiene-rubber (SBR) latex is a synthetic rubber comprising styrene and butadiene monomers. It is manufactured through the copolymerization of the hard styrene monomer and the soft butadiene monomer. The styrene provides the material with stiffness and strength, while the butadiene contributes to the elasticity and makes the binder more flexible. SBR is very similar to SBS in its mechanical bonding mechanisms and also in its chemistry with the exception that during the process of copolymerisation of the butadiene, single-unit monomers are randomly linked to form large, multiple units (SABITA, 2015).

SBR can be used both in hot and cold modified binders, although it is predominantly used as an emulsifier. SBR is mostly utilized in cold applied binders for applications such as seals, bond coats, micro surfacing and crack sealants. Hot polymer modified binders with SBR are less used in seals or asphalt mixtures, because the polymer percentage must be controlled to avoid the viscosity increase and the water evaporation of latex when blended with hot bitumen.

The advantages of SBR pertain to the improvement of binder adhesion with the aggregates in sealing applications. Another advantage is the improved elasticity and flexibility of the binder, in its excellent performance as a crack sealant to prevent water from accessing the pavement. In asphalt applications, SBR increases the resistance to deformation, enhances the fatigue properties and retards crack reflection (SABITA, 2015).

2.2.3. Ethylene-vinyl-acetate

Ethylene-vinyl-acetate or EVA is a polymeric plastomer which consists of two monomers of ethylene and vinyl acetate. The two monomers form long crystalline molecular strings which dissociate when the temperature is increased above the material's glass transition point. The morphology of EVA is semi-crystalline and interconnected by polyethene crystalline molecules and vinyl-acetate molecules, which form amorphous chains contributing to its flexibility. The copolymers are polar and, together with the maltenes of the bitumen, will dictate the compatibility and adhesive properties of the asphalt binder.

The properties of EVA are controlled by two main parameters. One of these concerns the vinyl acetate content, because the more it increases, the higher the proportion of rubbery regions (which increases the flexibility) and the lower the proportion of crystalline regions will be (decreasing the stiffness). The other main factor has to do with the molecular weight, which can be derived by measuring the melt flow index, because the higher the melt flow index, the lower the molecular weight and viscosity will be.

The main advantages of this polymer concern the ability to provide improved resistance to rutting in hot mix asphalt compared to conventional bitumen, as well as its improved compatibility, safer handling and better workability compared to SBS and SBR modifiers. Specific grades of EVA also increase the resistance to damage caused by fuel spillages. EVA modified bitumen is also more heat stable and does not deteriorate as fast as SBR and SBS modified bitumen products at elevated temperatures during storage. Storage stability will, therefore, be better than that of SBS and SBR. EVA also has a lower cost in comparison with block copolymers (Becker, 2001).

However, EVA presents some disadvantages. Its properties may vary for different grades depending on the chain length and its molecular weight, the vinyl acetate monomer content and the crystallinity. The compatibility of bitumen and EVA may vary, depending on the bitumen composition and type or grade of the polymer (SABITA, 2015). EVA also does not improve the elastic recovery of asphalt binders.

For binder modification, the vinyl acetate content and melt flow rate of EVA are as crucial as the styrene content and linear or radial structure in SBS when determining specific properties. Nevertheless, SBS and EVA are two polymers most frequently used for bitumen-modification (Sengoz and Isikyakar, 2008).

2.2.4. Natural rubber

Natural rubber latex consists of polymerised isoprene monomers which serve to increase the elasticity of the bitumen. Its main advantages concern its better rutting resistance, higher ductility and increased elasticity, among other properties required under cyclic loads.

However, it has some disadvantages, such as sensitivity to decomposition and oxygen absorption, too high molecular weight and, therefore, low compatibility. Thus, it must be partially decomposed and mechanically homogenized (Becker, 2001). Natural rubber is also more sensitive to heat and is mainly used in the modification of cold asphalt binders (SABITA, 2015).

2.2.5. Crumbed rubber

Crumbed rubber has had an auspicious use in hot mix asphalt production since, in addition to its inherent benefits, it also possesses a waste recycling and sustainability dimension. This type of rubber is usually manufactured from used vehicle tyres. The disposal of used tyres has been posing a considerable ecological challenge worldwide, due to their long-lasting nature. The only solutions are either their burning or disposal, both having a considerable amount of environmental impact deriving from the methods. Those solutions are not desirable in the global environment. Thus, a new methodology was developed to reuse this material, previously considered a waste, and revaluating it as an incorporating material in other applications. This process reduces both the pollution caused by the previous solutions, thus contributing to a more sustainable world, as well as potentiates the development of new industries that impact positively from an economic standpoint.

The use of crumb rubber in asphalt materials had its start in the 1920s, employing the dry method, where the rubber is utilized as a partial substitute for the aggregates. This method did not achieve the expected performance, and so, from the 1960s onwards, researchers have been using the wet method, adding rubber as asphalt binder modifier. Crumb rubber became of interest to this industry because of its elastic properties, which had the potential to improve skid resistance and durability of asphalt mixtures. Several factors can contribute significantly to the extent to which rubber functions as an elastic aggregate. Among these are included, the rubber gradation, rubber content and aggregate gradation (Hassan *et al.*, 2014).

Even though crumb rubber is utilized in many countries through the wet process, it presents some issues such as storage stability. This problem has prevented the broad application of crumb rubber bitumen (CRM) or rubber modified bitumen (RmB) in pavement solutions. Additionally, the high viscosity of this material may cause some difficulties during the manufacturing process as well as during paving and should be considered when selecting the aggregate grading (Toth *et al.*, 2016).

Some of the advantages of using this material, along with the previously explained recycling dimension, come from its relatively lower costs in comparison with PMB, and the potential of improving fatigue resistance, reduce reflective cracking and increasing rutting resistance (Becker, 2001).

2.3. Bitumen

Bitumen can be defined, from a commercial point of view, as a thermoplastic material that was usually of relatively low cost and has been widely used for several applications such as roofing, paving, industrial products, among others. In the latter applications, pavement mixtures and industrial applications, it is crucial that the bitumen possesses resistance to both climatic challenges and demanding traffic loads; for which the rheological properties of this material are of great importance (Lesueur, 2009).

With the growing concern about the durability and quality of the pavements, it has been an increasing concern over time to provide more in-depth studies of the essential elements that constitute a pavement. One of these elements is the bitumen, which permits the involvement of all the elements through its bonding capabilities. According to the European Standard EN 12597 (Bitumen and bituminous binders – Terminology), bitumen is defined as a "*virtually involatile, adhesive, and waterproofing material derived from crude oil, or present in natural asphalt, which is completely or nearly completely soluble in toluene, and very viscous or nearly solid at ambient temperatures*".

Analysis of the molecular weight distribution shows that bitumen is a complex mixture of around 300 up to 2000 chemical compounds, with an average value of 500 to 700. This quantity of chemicals makes bitumen a challenge to thoroughly characterize. Bitumen is usually divided into two principal constituents: asphaltenes, a constituent of the discontinuous phase, and maltenes, a constituent of the continuous phase, with the latter being further classified into saturates, aromatics and resins. These are known as an analysis method named SARA, Saturate, Aromatic, Resin and Asphaltene (Porto *et al.*, 2019).

Having a thorough understanding of the different aspects of the characteristics of bitumen is of paramount importance. This knowledge proves its importance when particular challenges arise, mainly in some bitumen applications, which include uneven dispersion, discontinuity in phase, instability with polymers or additives, among many others. These difficulties can appear in the production stage as well as in the application (Porto *et al.*, 2019). It is commonly accepted that the original characteristics of bitumen are highly dependent on its production and processing techniques and its crude oil characteristics (Paliukait *et al.*, 2014). Crude oils of a higher quality and proper distillation processes can enhance the bitumen attributes, generally heavier crude oils derive in higher bitumen yields (Read and Witheoak, 2003).

Due to their function in asphalt mixtures, binders must have an adequate chemical composition as well as a sufficient viscosity to be able to perform their function as the matrix in the mixture. They should be neither too liquid nor too viscous to involve the aggregates adequately. The binder is a fundamental component of the asphalt mixture, as it is responsible for acting as the glue between the aggregates and ensuring their cohesion (Burguete, 2013).

From a practical point of view, bitumen must be fluid enough at very high temperatures, such as 160 °C, to possess enough workability in order for the aggregates to be appropriately and homogeneously coated during mixing. It must also be stiff enough at high temperatures to be able to resist rutting (Porto *et al.*, 2019). Finally, it must remain soft and elastic enough at low temperatures to resist thermal cracking (Lesueur, 2009). All of these requirements can appear almost opposite and hard, if not impossible, to be found in one single material. Moreover, in some applications, the performance of conventional bitumens may not be satisfactory enough. In some engineering projects, conventional bitumen can be too brittle in cold environments and too soft in warm ones. This limited performance in temperature ranges is one of the main disadvantages of neat bitumen, which can limit its use in some roofing and pavement applications (Porto *et al.*, 2019).

Since the traffic speed and volume have been consistently increasing in recent decades, pavements have been subjected to a considerable load increase which was not planned when pavements were first designed and executed. This situation has notably diminished the life span of the asphalt pavements, increasing its maintenance costs and posing a higher risk to the safety and comfort of its users. Therefore, a variety of additives and modifiers have been used to enhance bitumen performance, and respond adequately to the new pavement challenges. These include polymers, chemical modifiers, extenders, oxidants and antioxidants, hydrocarbons and anti-stripping additives (Porto *et al.*, 2019).

Polymers are a vast field of modifiers that have been researched in great detail. By improving bitumen characteristics such as resistance to deformations, thermal cracking, surface breakdown and susceptibility to temperature, PMBs have to replace neat bitumens in many applications, both in initial paving as well as in maintenance treatments. Some of these include hot and cold mixes, crack sealing and recycled solutions. PMBs are used when improved performance is needed and, in some cases, also when there is a necessity to reduce the pavement life cycle costs (Becker, 2001).

Particularly in SMA type mixtures, additives are usually used due to the higher quantity of bitumen present. These are intended to prevent the leaking of the binder by increasing its consistency and improving its behaviour to temperature variations (Becker, 2001). These additives are usually polymers or fibres, or even the combination of both. In the current research work, only polymers were utilized.

According to Torres (2014), the choice of base bitumen should take into account factors such as price, market availability, characteristics and even previous successful experiences of binders used in asphalt mixtures, since the binder is the component that most influences the performance of the mixtures.

2.3.1. Polymer modified binders

Bitumens that are exposed to severe or rough conditions, such as steep gradients, high traffic loading, extreme road surface temperatures and others, require better performance than ordinary bitumens. In order to improve the properties of bitumen, polymers are added to the bitumen to form modified binders. According to the European Standard EN 12597 (Bitumen and bituminous binders – Terminology), modified bitumen is a "*bituminous binder whose rheological properties have been modified during manufacture by the use of one or more chemical agents*". Modified binders perform better concerning: adhesion, durability, elasticity, deformation resistance, flexibility at low temperatures, high viscosity at high temperatures, among other characteristics (Van Amsterdam, 2000).

The polymer modified binders (PMB) are produced by mechanically mixing or by the chemical reactions of bitumen and one or more polymers, among those presented in Table 1, whose percentage can usually range from 3% to 10% or even higher (Porto *et al.*, 2019).

The PMBs can be arranged into two separate categories according to their structure, homogenous binders and non-homogenous binders. These two broad groups are classified regarding the existence, or lack thereof, of two distinct phases, observed in the binders, which can be detected on a microscopic level.

Homogenous binders are, as the name implies, binders that possess a uniform structure or composition, more specifically they are defined as a blend of bitumen and polymer where two distinct phases cannot be detected on a microscopic level. These phases are interwoven to such an extent that the modified binder behaves as a single-phase material (SABITA, 2015).

Table 1 – Categories of polymers mainly used in bitumen modification (Porto *et al.*, 2019)

Categories	Polymer
Thermoplastic Polymers	Polyethene (PE) Polypropylene (PP) Ethylene-Vinyl-Acetate (EVA) PVC EBA
Thermoplastic Elastomers	Copolymers styrene-butadiene-styrene (SBS) Copolymers styrene-isoprene-styrene (SIS)
Thermosets	Epoxy resin Polyurethane resin Acrylic resin Phenolic resin
Natural and synthetic rubbers	Styrene-butadiene-rubber (SBR) Natural rubber Polyolefines Reclaimed tyre rubber
Bitumen chemical modifiers	Sulphur (S) Polyphosphoric acid (PPA) Reactive polymers Maleic Anhydride (MAH) Nanocomposite modifiers

Some of the polymers most frequently utilized to produce homogenous binders are styrene-butadiene-styrene (SBS), styrene-butadiene-rubber (SBR), styrene isoprene rubber (SIR), ethylene-vinyl-acetate (EVA), reactive elastomeric terpolymer (RET) and natural rubber latex, among many others.

Non-homogenous binders are characterized by having two distinct phases, which can be detected on a microscopic level. These phases observe differences in properties according to what stage a test is performed, they display behaviour with a non-uniform structure or arrangement, as it is implied by their nomenclature. The primary material that falls under this category and is incorporated into binders consists of crumbed rubbers recycled from tyres (SABITA, 2015), which are partially dissolved in a bitumen matrix.

Another classification can be made according to two broad categories: plastomers and elastomers. Within these two groups, further sub-groups exist, of which, thermoplastic polymers in the plastomer group and thermoplastic rubbers in the elastomer group are the most common (Airey, 2001).

Structures with elastomeric features, those that present resistance to permanent deformation and recovery of the original shape after loading, are those obtained by using elastomeric modifiers in bitumen

(Airey, 2002). As an example of an elastomer is crumbed rubber, usually made from ground tyres. Several technologies are in place for using ground tire rubber. This material is used primarily to address rutting.

Plastomers are used to improve the high-temperature properties, i.e., the rutting resistance of modified binders and mixtures. When these are incorporated into the bitumen, the resulting structure is a rigid three-dimensional network that resists deformation (Airey, 2002). Low-density polyethene (LDPE) and ethylene-vinyl acetate (EVA) are examples of plastomers used in asphalt modification.

The most commonly used chemical modifier is polyphosphoric acid (PPA). This modifier may be used in combination with polymers to increase the high-temperature stiffness.

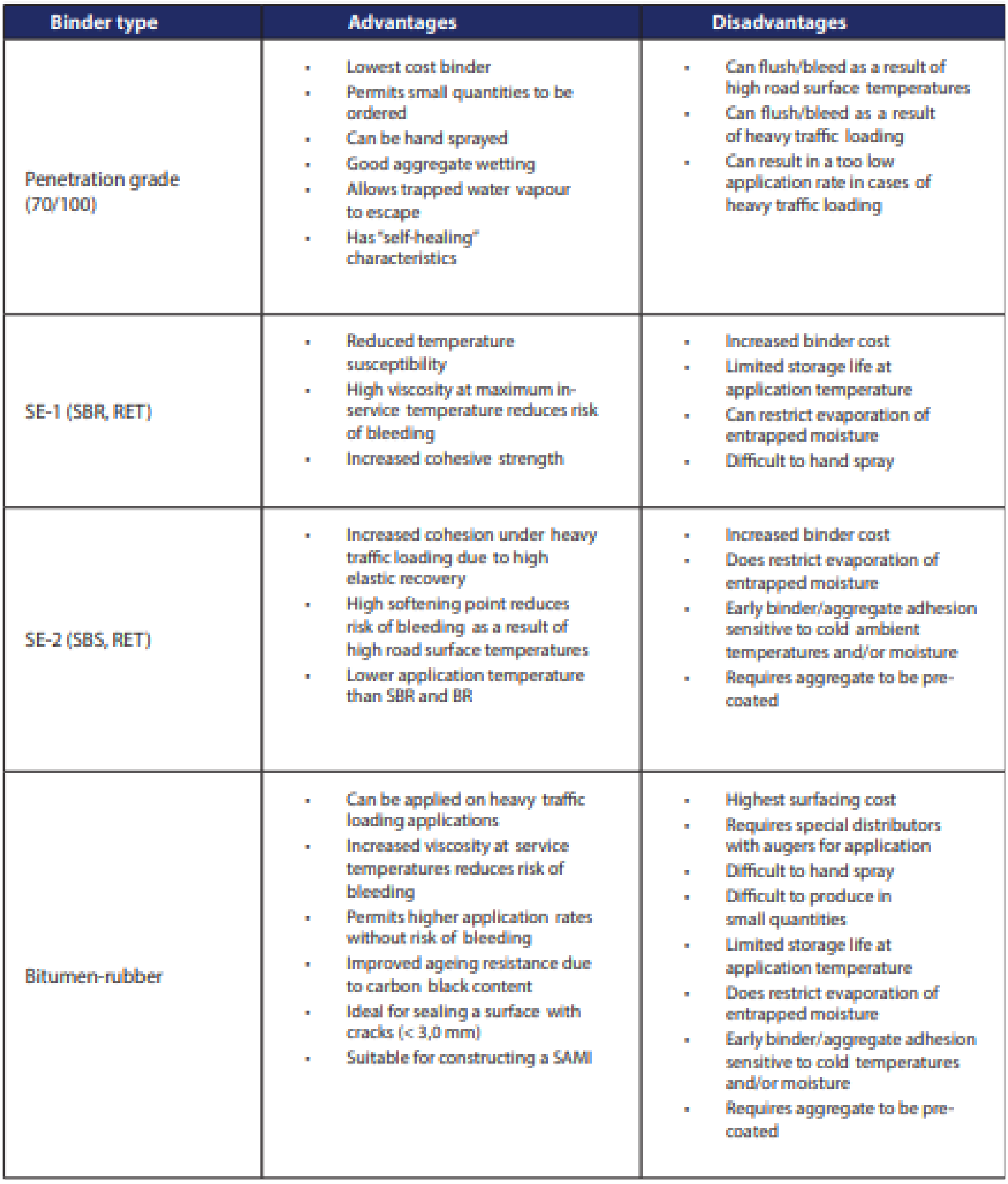
Other modifiers that may be used include asphalt binder extenders (primarily sulphur) and hydrocarbon materials. Hydrocarbons can produce either hardening or softening effects. Hardening materials can be added to increase rutting resistance. Softening agents or rejuvenators are used to lower the viscosity of aged asphalt binders in mixtures containing recycled asphalt pavement or RAP (Walker, 2019).

Extensive research in asphalt mixtures and pavement technology has produced great solutions to the problem of increasingly higher traffic volumes and loads. One of these solutions is polymer modified bitumens (PMB), with the advantages and disadvantages presented in Figure 5. These are increasingly utilized across the world, and they provide superior performance (Toth *et al.*, 2016).

2.3.2. Methods of incorporation of polymers in asphalt mixtures

There are three distinct methodologies of incorporating polymers into the asphalt mixtures: as an aggregate replacement, as an aggregate coating or as bitumen modifier. The techniques utilized for this incorporation consist of the dry process and the wet process.

As Hassan *et al.* (2014) describe, "*the dry process involves the blending of crumb rubber with hot aggregates before mixing with bitumen*". This process essentially replaces a portion of the aggregates with the modifying agent in a solid form, granules or shavings, before mixing with the bitumen, which makes this agent to effectively works as an elastic aggregate within the mixture (Brandão, 2015). The mixtures produced by the dry method are considered to be simple because no chemical reactions occur between the two components. In this case, the polymer is considered as a filler which gives specific properties to the mixture (Boutevin *et al.*, 1989).



Binder type	Advantages	Disadvantages
Penetration grade (70/100)	• Lowest cost binder • Permits small quantities to be ordered • Can be hand sprayed • Good aggregate wetting • Allows trapped water vapour to escape • Has "self-healing" characteristics	• Can flush/bleed as a result of high road surface temperatures • Can flush/bleed as a result of heavy traffic loading • Can result in a too low application rate in cases of heavy traffic loading
SE-1 (SBR, RET)	• Reduced temperature susceptibility • High viscosity at maximum in-service temperature reduces risk of bleeding • Increased cohesive strength	• Increased binder cost • Limited storage life at application temperature • Can restrict evaporation of entrapped moisture • Difficult to hand spray
SE-2 (SBS, RET)	• Increased cohesion under heavy traffic loading due to high elastic recovery • High softening point reduces risk of bleeding as a result of high road surface temperatures • Lower application temperature than SBR and BR	• Increased binder cost • Does restrict evaporation of entrapped moisture • Early binder/aggregate adhesion sensitive to cold ambient temperatures and/or moisture • Requires aggregate to be pre-coated
Bitumen-rubber	• Can be applied on heavy traffic loading applications • Increased viscosity at service temperatures reduces risk of bleeding • Permits higher application rates without risk of bleeding • Improved ageing resistance due to carbon black content • Ideal for sealing a surface with cracks (< 3,0 mm) • Suitable for constructing a SAMI	• Highest surfacing cost • Requires special distributors with augers for application • Difficult to hand spray • Difficult to produce in small quantities • Limited storage life at application temperature • Does restrict evaporation of entrapped moisture • Early binder/aggregate adhesion sensitive to cold temperatures and/or moisture • Requires aggregate to be pre-coated

Figure 5 – Advantages and disadvantages of modified binders compared with conventional binders (SABITA, 2015)

The same authors have thoroughly analysed this process and its subtleties, as well as its advantages and disadvantages. The advantages result from substituting part of the aggregates in the mixture while improving the elastic properties and assuring some bitumen modification of the mixture. Conversely, some disadvantages must also be pointed out, mainly and although this method allows for the use of more significant percentages of polymer and presents lower costs in the initial stages, in general, it appears that its performance is not as good as the results observed from the wet process (Brandão, 2015). Another concern pointed out among the scholars is the lack of standards and inconsistent performance, which in turn results in scepticism among practitioners and researchers in selecting this process over the wet method, despite having the potential to recycle more crumb rubber, since it uses a higher proportion of polymer in comparison. All of these reasons translate into the overwhelming majority of asphalt paving projects involving crumb rubber opting to use the wet process instead of the dry method (Hassan *et al.*, 2014).

In the wet method, the solid polymer is mixed with the bitumen at high temperatures giving rise to a modified binder so that it is used in the modified asphalt mixture to be applied in the pavement at a later stage. In the bitumen modification process, the polymer is introduced in the bitumen, which is still hot and fluid, during a specified period, to guarantee the interaction between the polymer and the bitumen (Torres, 2014).

In this method, the mixtures between the two components are said to be complex, because chemical reactions or some other interaction occurs between the two parts of the mixture (Boutevin *et al.*, 1989). Therefore, one of the significant advantages of the wet method pertains to the more effective modification of the binder, which is also one of the reasons this procedure is heavily utilized by the industry (Hassan *et al.*, 2015).

The modified binder, preferably produced at a bitumen processing plant, is then delivered and added to the aggregate during asphalt mix production. In this process, the polymer reacts with the base bitumen, swelling and partial dissolving the polymer and thus, absorbing the oils from the bitumen into the rubber matrix (Gawel *et al.*, 2006).

Generally, it has been suggested that there is a higher degree of rubber-bitumen interaction with enhanced properties in the wet process than the dry process, thereby making the wet process the preferred method for modification. One of the main aspects of polymer modification is the interaction between the polymer and bitumen, which is considered vital to understanding better the concept of polymer modification in both wet and dry process methods. Interaction refers to the diffusion of the lighter bitumen fractions, aromatic oils in the maltenes, into the polymer, which leads to the swelling of the polymer particles. The swelling of the polymer as a result of the polymer-bitumen interaction is shown schematically in Figure 6 (Hassan *et al.*, 2014).

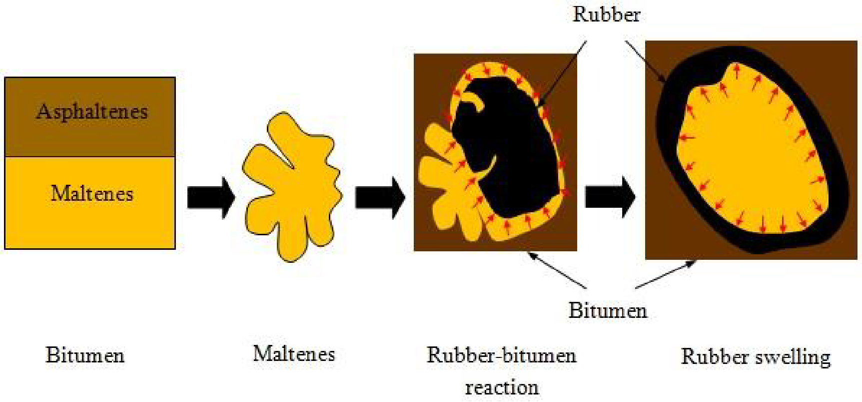


Figure 6 – Schematic of rubber swelling in rubber-bitumen interaction (Hassan *et al.*, 2014)

When the polymer gets in contact with bitumen, it generally absorbs the maltenes fraction, which has a low molecular weight and leaves the residual bitumen containing a higher portion of asphaltenes, with high molecular weight thus increasing the PMB viscosity. The maltenes fraction diffuses into the polymer particles, increasing the dimensions of the polymer network until equilibrium swelling is achieved. Factors such as the temperature and time of the contact between bitumen and polymer, the chemical composition of bitumen, polymer type and size were all found to affect the rate of swelling (Hassan *et al.*, 2014).

2.4. Low-temperature conditions

The temperature of the surface of pavements can vary significantly with the variation of climatic factors such as radiation, wind speed, atmospheric pressure, and low ambient temperature, among others. This surface temperature is mostly related to the surface characteristics of the pavement material as opposed to the heat transfer property of the pavement. According to Wang *et al.* (2014), the coefficients of thermal conductivity of asphalt mixtures can vary considerably because of the difference in aggregates. In their study on the factors influencing the temperature of the surface of the pavement, they observed that when all external elements were the same, the resulting calculated temperatures of a pavement surface were also the same, despite the difference in coefficients of thermal conductivity. This observation explains the importance the external factors have on determining the temperature of the pavement surface and therefore, the necessity to simulate them during testing conditions accurately.

More substantial variation in temperatures will be recorded at locations closer to the pavement surface, which in turn will lead to higher thermal stresses. Even though the magnitude of the temperature variation in winter is smaller than in summer, the pavement is subjected to freezing and thawing cycles. The most critical condition in the surface layer, in winter conditions, is the diurnal temperature, although both seasonal and diurnal variations of temperature should be considered when designing a surface layer (Wang *et al.*, 2014).

Low temperature cracking is one of the most common failure modes of flexible pavements in cold climate zones (Alataş and Yilmaz, 2017, Stimilli *et al.*, 2017). These regions are characterised by daily temperature drop that can be remarkably rapid, very long cold seasons and the lowest temperatures experienced can be very low. All these factors will play a role in the continuous maintenance needed to keep the roads in acceptable service conditions, which can result in high direct and indirect costs.

Pavements executed in colder regions are subjected to significant variations in temperature. Not only when it comes to the level of temperature they must endure but also, to the pace of cooling. The variation of this cooling rate can create tensile stresses derived from the characteristic of asphalt mixtures to extend when heated and contract when cooled. These stresses occur because the asphalt layer is constrained in the pavement structure and is unable to relieve the thermal stresses by internal relaxation. As a result of the drop in temperature, thermal tensile stresses increase, and when they exceed the fracture strength of the mixture, cracks due to low temperature may appear (Pszczola and Szydlowski, 2018). Microcracks initially appear on the surface of the pavement when the stress of the pavement exceeds its tensile strength. If the low-temperature cycles continue, which is the usual scenario in colder regions, these microcracks propagate through the pavement leading to a more severe cracking problem in the mixture (Das *et al.*, 2012).

In colder zones, when water fills these cracks during the winter months, it freezes, and ice lenses along with frost heave can develop. This condition results in the loss of fines and formation of voids throughout the pavement, leading to a load-bearing capacity reduction. Thus, it is of crucial importance to take into account all the variables that may affect the pavement in colder regions, during the designing stage. If these are not adequately considered, it can lead to the mentioned formation of cracks that in turn result in the reduced service life of the pavement, high maintenance costs and poor riding quality (Isacsson and Zeng, 1997). This condition, along with the change in the micro-structural stress mechanism, determines the fracture resistance behaviour of asphalt mixtures to temperature (Kim and Hussein, 1997). However, much other pavement distresses, such as rutting, moisture-induced damage, among others, may appear on asphalt pavements that have been in use for an extended period (Alataş and Yilmaz, 2017).

The low-temperature properties of asphalt mixtures can be, somewhat, indirectly derived from bitumen properties. The penetration depth is empirical, albeit only roughly, correlated with asphalt binder performance (Pavement Interactive, 2019). Despite this, binder testing alone may not be sufficient to predict the effect of improvements, such as additives and modified bitumen, regarding the resistance of the mixture to thermal stresses (Pszczola and Szydlowski, 2018).

In order to accurately assess the factors that affect the asphalt mixtures in colder regions, several laboratory tests have been developed. One of these methods is the thermal stress restrained specimen test (TSRST), which determines the critical cracking temperature that results from a single drop in temperature to an extremely low value observed during severe winters. The test presents many advantages, although with some limitations. Because the failure temperatures obtained from the test depend on the established cooling rate, the results of thermal stress and temperature at fracture should be regarded as a comparative measure between asphalt mixtures (Pszczola and Szydlowski, 2018).

Other characteristics can be tested by different methods, such as tensile strength. One of the methods that describe the direct tensile strength of asphalt mixtures at low temperatures is the uniaxial tension stress test (UTST). Another is the indirect tensile test (IDT), which also measures creep compliance. The bending beam rheometer (BBR), can also be used to obtain the asphalt binder strength at low temperatures. Another method to describe the strength properties of asphalt mixtures at low temperatures is to use flexural strength (bending beam test) at low temperatures.

Fracture properties of asphalt pavements can be defined based on fracture mechanics theory and can be strictly related to laboratory test results. There are several test methods to assess fracture parameters, including the bending of single edge and notched beams (SENB), bending of semi-circular beams (SCB), and tension of disc-shaped specimens (DC-T). One of the most suitable and frequently used methods is the bending test of semi-circular specimens (SCB). For better cracking characterization, more parameters can be assessed, like fracture energy (pre-peak and post-peak), toughness index, and flexibility index, among others (Pszczola and Szydlowski, 2018).

Another indicator of the potential for crack formation is the relaxation potential of a mixture given by the relaxation modulus. It represents the capacity of a mixture to dissipate the thermal-induced stress. This capacity plays a vital role since the higher the relaxation potential, the lower the potential to reach the failure point and thus, to form cracks. Mixtures characterized by faster decay of relaxation modulus are less prone to cracking at low temperature and can be more suitable for use in cold climate regions (Stimilli *et al.*, 2017).

3. MATERIALS AND METHODOLOGY

> *"Whatever the medium, there is the difficulty, challenge, fascination and often productive clumsiness of learning a new method: the wonderful puzzles and problems of translating with new materials."*
>
> Helen Frankenthaler

This section describes both the methodology as well as the materials adopted in the study. It entails all the norms and specifications used as guidelines, the variations in the cases these could not be followed and explanations for the reasons for this deviation.

The materials employed during this research can be divided into three categories: aggregates, polymer and bitumen. Brief descriptions of the materials, their selection processes and characterization methods are presented in this chapter. The methods of characterization of the asphalt mixtures, and their components, are also presented. Those processes evaluate the physical properties by translating them into comprehensible values or quantities to characterize their qualities and attributes.

The materials were provided by the Transport Systems and Infrastructures Road Pavements Laboratory of the University of Minho, except for the polymer, which was provided by the company Weedswest Global Solutions Group.

3.1. Aggregates

The aggregates used in this work took into consideration their local availability. Natural aggregates are abundant in the region, being explored in several quarries, and the consistent demand reduces their cost. Furthermore, an increasing concern to reduce the environmental effects of aggregates delivery justifies using local sources, with a significant reduction of their transportation distance, costs and corresponding pollution. The region has mostly igneous rocks, and thus the aggregates selected are mainly of granite origin, except the filler, which is of limestone origin to improve the mixture workability. These aggregates were obtained from the Bezerras quarry and are stored in the Transport Systems and Infrastructures Road Pavements Laboratory of the University of Minho.

The asphalt mixture selected to evaluate the mechanical performance at low temperatures was a Stone Mastic Asphalt 11 (SMA 11), which will be detailed further ahead.

The aggregates, as well as the other components, described ahead, were used with the same mix design for all the studied asphalt mixtures. The properties of these aggregates follow the indications provided by the European Standard EN 13043 (Aggregates for bituminous mixtures and surface treatments for roads, airfields and other trafficked areas).

The aggregates are characterized by their grain size. Typically, the aggregates have different grain sizes and different quantities of each size. In order to fully determine both of these properties, a procedure which entails the passing of the aggregate through sieves with different mesh sizes is performed, which then translates these quantities into a grain size distribution curve.

The grain size distribution curve is, as before stated, determined by sieving the material with a series of sieves, performed according to the European Standard EN 933-1 (Tests for geometrical properties of aggregates. Determination of particle size distribution. Sieving method).

The aggregates pass from the wider opening sieves to those with smaller openings, and the mass of aggregates retained in each sieve is recorded. From this process, it is possible to obtain the size grading curve of the sample. Figure 7 shows an example of the final distribution of one of the aggregate fractions (4/10) per grain size after concluding the sieving procedure.

Figure 7 – Grain size distribution for the 4/10 aggregate

After the sieving process is performed for all the aggregate types, several grain size distribution curves are obtained, one for each type or fraction of aggregate. Figures 8 and 9 present the different types of aggregates used during this research work, respectively, for coarser and finer fractions.

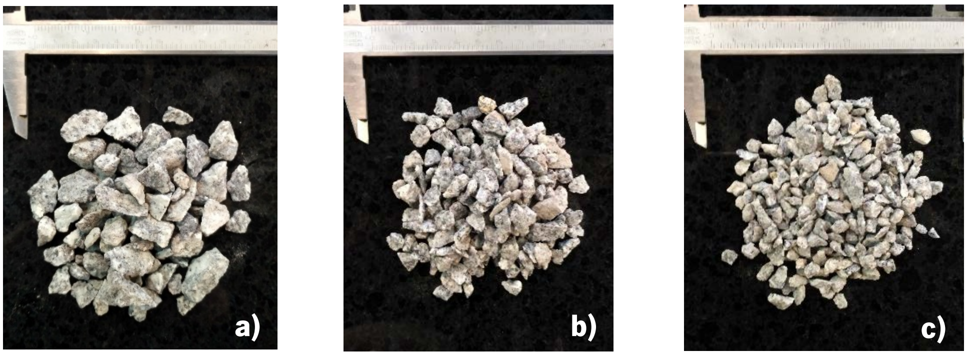


Figure 8 – Different coarse aggregate fractions used in this work: (a) 6/14; (b) 4/10; (c) 4/6

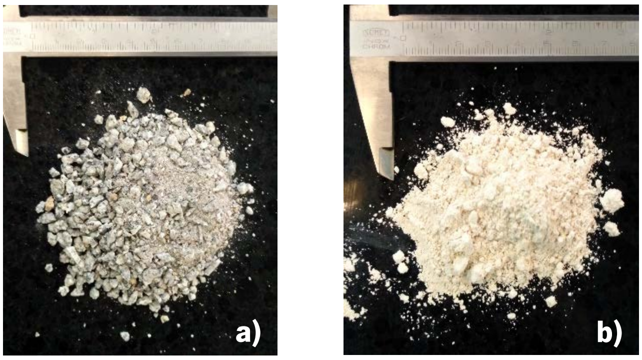


Figure 9 – Different fine aggregate fractions used in this work: (a) 0/4; (b) filler

The following task consisted in obtaining the final grading composition required for the selected asphalt mixture SMA 11, by determining the amount of each aggregate fraction to incorporate in the mixture. This task was executed by taking into account a standard minimum and maximum proportion for each grain size for this specific type of asphalt mixture, SMA 11, shown in Table 2.

Table 2 – Minimum and maximum grain size curves for SMA 11

Sieves (mm)	SMA 11 (%)
16	100
11.2 (11)	90 – 100
8	55 – 80
4	22 – 33
2	20 – 30
0.5	12 – 20
0.063	6 – 10

The two curves, minimum and maximum percentages, are plotted in a graph together with grain size distributions of the mixture of the several aggregate fractions. The grain size curve is adjusted between the minimum and maximum proportions until an optimal curve is obtained for SMA 11 mixture.

3.2. Polymer

The same type of polymer, styrene-butadiene-styrene (SBS), was utilized for both of the asphalt mixtures produced with PMB, just in different proportions of the total weight of the mixture.

This polymer varies its grain size from approximately 3 to 6 mm, having the majority of the grains with dimensions around 3 mm. SBS is characterized by a rough surface and variable shape. This roughness provides a better chance for the bitumen to adhere and interact with this polymer. Figure 10 shows the SBS polymer utilized during this study.

Figure 10 – SBS utilized in this study

3.3. Asphalt binders

3.3.1. Bitumen and polymer modified binders

For the production of both of the polymer-modified bitumens, the same type of virgin bitumen was used as the base binder, which was a 50/70 penetration grade bitumen.

In order to compare the influence of SBS polymer in the behaviour of the asphalt mixtures, two asphalt mixtures were produced with the same constituents, only varying in the amount of polymer. This variation entails two proportions of incorporation of SBS polymer in the asphalt binder: i) the first one, hereafter named PMB25, is composed of 2.5% SBS and 97.5% base bitumen; ii) the second one, hereafter named

PMB50, is comprised of 5.0% SBS and 95.0% base bitumen. Table 3 summarizes the studied asphalt binders and their compositions.

Table 3 – Asphalt binders used in this work and their compositions

Asphalt binder	50/70 bitumen (%)	SBS (%)
50/70	100.0	0.0
PMB25	97.5	2.5
PMB50	95.0	5.0

3.3.2. Modification of the bitumen to produce the PMBs

The wet method was selected as the asphalt binder modifying process. The difference between the two available processes, the dry method and the wet method, has been explained in Chapter 2, and the option for the wet method resulted from the advantage of assuring a more effective modification of the binder. This reason also justifies the heavy use of this method by the asphalt industry (Hassan *et al.*, 2015) and thus, this research work will better transpose the typical PMBs applied in road construction.

For the production of the modified binders, the base bitumen was first heated to 170 °C, and the polymer was initially incorporated using a low shear mixer, IKA – model RW20 DZM, so that the polymer could be added slowly and more homogeneously. The quantities for each PMB were previously calculated.

The container where the modified bitumen was being blended was then transferred to a high shear mixer, IKA – T5 Ultra Turrax Basic, where the actual modification process could be performed. The bitumen and polymer blend was continuously mixed at a constant rotation of 7400 rpm and maintaining a constant temperature of 170 °C for approximately 90 minutes. These factors allow the adequate digestion of the polymer in the binder, to obtain the final polymer-modified bitumen. The high shear mixer, shown in Figure 11, can thoroughly crush the SBS polymer granules because of the geometric properties of its rotor-stator.

As previously explained, the modified bitumens produced in this work consist of two types with the same base binder, a 50/70 penetration grade, but with different percentages of the modifying agent (2.5% and 5.0% of SBS polymer).

Figure 11 – High shear mixer IKA – T5 Ultra Turrax Basic

3.4. Characterization of the asphalt binders

The tests for characterization of a polymer-modified bitumen to be used in SMA mixtures must comply with the European Standard EN 14023 (Bitumen and bituminous binders - Specification framework for polymer-modified bitumens), which includes standards for penetration and softening tests, among others. This PMB standard has a combination of classes that specifies the basic requirements for PMBs that can be used in SMA mixtures (Blazejowski, 2011).

3.4.1. Softening point by ring and ball method

The ring and ball method is utilized to evaluate the softening point of an asphalt binder. This technique is based on the European Standard EN 1427 (Bitumen and bituminous binders - Determination of the softening point - Ring and Ball method), and it consists in determining the softening point of the bitumen, meaning, it corresponds to the maximum temperature that the binder allows in the asphalt mixture without significant deformations. Above this temperature, the bitumen consistency is too low and, thus, fails to guarantee the necessary stability when used in asphalt mixtures.

The process entails the placement of two flat discs of asphalt binder, cast in shouldered brass rings, in a liquid bath, either water or glycerol (dependent on the temperature obtained in a first trial), which are heated at a controlled rate, while each supports a steel ball. The softening point is reported as the mean of the temperatures at which the two discs soften enough to allow each ball, enveloped in the asphalt binder, to fall to a distance of 25.0 mm ± 0.4 mm.

This method is performed with a ring and ball apparatus, shown in Figure 12(a). The initial test temperature is established at 5 °C and then gradually augmented at a rate of 5 °C per minute, until the softened bitumen touches the metal plate placed at a distance of 25.0 mm ± 0.4 mm, as exemplified in Figure 12(b). The temperature at the end of the test is then recorded, which reveals the softening point.

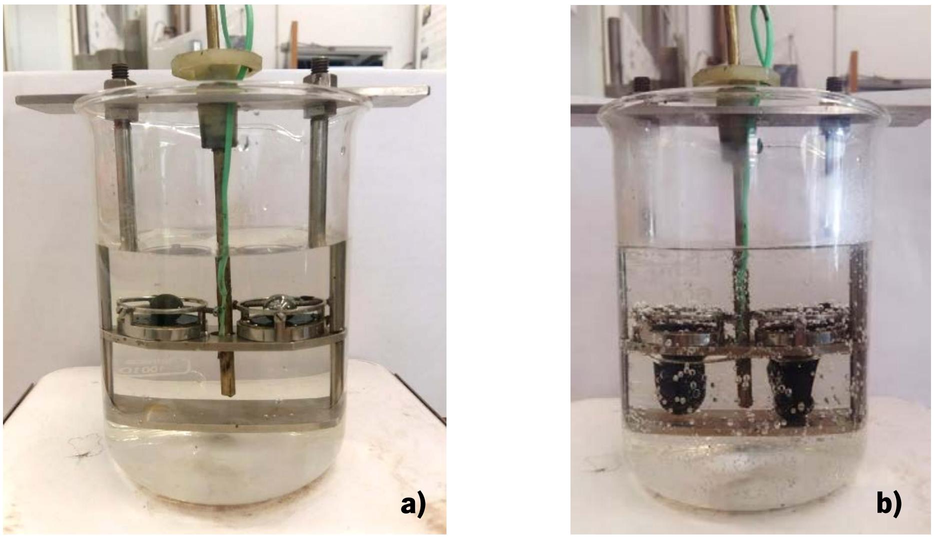


Figure 12 – (a) Ring and ball apparatus; (b) Softening point after testing

3.4.2. Needle penetration

The needle penetration determination method is a test standardized by the European Standard EN 1426 (Bitumen and bituminous binders - Determination of needle penetration). This procedure indirectly determines the consistency, more specifically the relative hardness, of bitumen and asphalt binders. The higher the penetration, the softer the bitumen. Conversely, the lower the penetration, the harder the bitumen. Bitumens, in Europe, are classified according to the values obtained from this test.

In the penetration test, a standard needle penetrates vertically into a sample of the bitumen under specific conditions of temperature, load and duration of loading. According to the standard EN 1426, for penetrations up to approximately 330 × 0.1 mm (the most common ones for typical bitumen), the operating parameters must be at a test temperature of 25 °C. However, for expected penetrations above approximately 330 × 0.1 mm, the test temperature must be reduced to 15 °C, maintaining the parameters of value and load duration, in addition to the conditions shown in Table 4.

The needle, weighing 100 g, is released for 5 seconds. After this time, the depth of penetration into the bitumen is measured in tenths of a millimetre. Figure 13 illustrates this process.

Table 4 – Criteria for conducting the needle penetration test

Penetration (0.1 mm)	Internal depth (mm)	Internal diameter (mm)	Approximate volume of sample (ml)	Approximate time of air cooling or water conditioning (min)
pen < 160	35	55	80	60
160 ≤ pen < 330	45	70	170	75
330 ≤ pen < 500	60	70	230	90

Figure 13 – Bitumen penetration test

Three tests must be carried out in different locations of the sample and should not differ significantly from each other for the test to be considered valid. The final penetration value of the bitumen is given by the average of the three measurements. For example, a penetration of 50 to 70 × 0.1 mm indicates a 50/70 penetration grade bitumen. The penetrometer used in this test is the semiautomatic penetrometer from Controls Group, model 81-B0101/D, shown in Figure 14.

Figure 14 – Semiautomatic penetrometer, Control Groups, model 81-B0101/D

3.4.3. Resilience

The resilience test was carried out in accordance to the European Standard EN 13880-3 (Hot applied joint sealants - Part 3: Test method for the determination of penetration and recovery – resilience), and it aims to determine the elastic recovery capacity of the modified bitumen indirectly. Resilience is the ability of an elastic material to absorb energy and release that energy as it springs back to its original shape (Asphalt Institute, 2020), i.e., is the capacity of a strained body (the PMB) to recover its size and shape after deformation caused by compressive stress.

This technique consists of a method for determining the penetration and recovery (resilience) of a modified bitumen using a standard penetrometer fitted with a ball penetration tool. In this procedure, a bitumen sample is placed in a water bath for 60 to 90 minutes at 25 °C. Then, a compression load is applied to the bitumen sample, through a 17 mm diameter metal sphere, until a 10 mm deformation is achieved. Once this value is reached, the load is maintained for an additional 5 seconds without increasing deformation. The load is then removed from the sample for 20 seconds, and the deformation recovery in the tested bitumen sample is measured. The resilience or recovery of the penetration of the bitumen sample is calculated according to Equation 1, where P is the initial penetration value of the sphere (0.1 mm) and F is the penetration value after recovery (0.1 mm).

$$Resilience\ (R)\ (\%) = P + 100 - F \tag{1}$$

The resilience test was only performed on the polymer-modified binders because the base bitumen does not present resilient characteristics since it is not modified by any polymer, i.e., it is not able to recover imposed deformation.

3.4.4. Dynamic viscosity

The dynamic viscosity test is a method based on the European Standard EN 13302 (Bitumen and bituminous binders - Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus) but with an adaptation to the procedure proposed by Silva *et al.* (2009) in which the initial value of the test temperature is 100 °C and is increased by 10 °C for each new measurement. This procedure was performed with a Brookfield rotational viscometer, model DV-II+Pro (Figure 15). The procedure consists of rotating a spindle at a constant speed, inside a fluid bitumen sample (which forces the test to be carried out at high temperatures), measuring the torque applied by the viscometer.

Figure 15 – Brookfield rotational viscometer, model DV-II+Pro

The spindle (Figure 16 (a)) is a cylindrical object, and the bitumen sample will be poured in a cylindrical container (Figure 16 (b)) with a diameter slightly larger than the spindle.

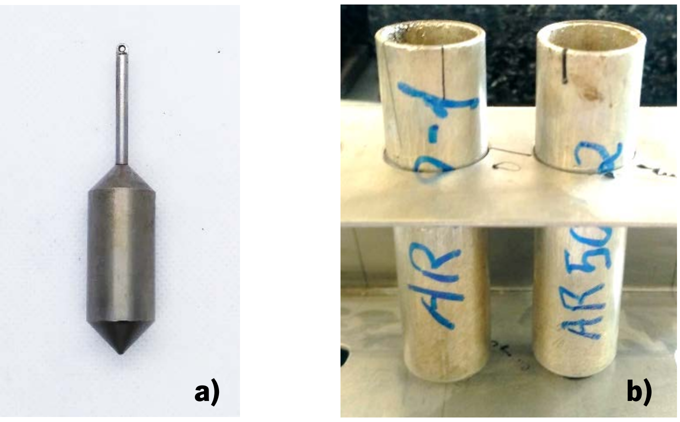


Figure 16 – (a) spindle and; (b) bitumen containers used in the viscometer

The rotation effort determines the torque necessary to overcome the resistance provided by the viscosity of the fluid material. The rotational movement is exerted between the bitumen and the spindle surface so that the rotation speed is constant and is measured using the equipment mentioned. This effort is proportional to the viscosity. According to Van Amsterdam (2000), the viscosity measures the flow rate of a liquid and its consistency (thickness and smoothness). A liquid has a low viscosity if a force is applied to it and, in response, it moves quickly. Conversely, if it moves slowly, it has a high viscosity.

In this work, the viscosity measurement started at 100 °C and was gradually increased by 10 °C until the next temperature is stabilized, which usually happens after about 10 minutes, and the measured values are then recorded, with each step monitored by a computer. This technique is repeated until the temperature of 180 °C is reached. Above 100 °C, the bitumen acts almost entirely as a viscous fluid, and so this procedure characterizes the bitumen stiffness by determining its rheological properties.

3.4.5. Rheological properties

The rheological properties of the asphalt binders at high and intermediate temperatures were determined using the dynamic shear rheology test. This test was based on the European Standard EN 14770 (Bitumen and bituminous binders - Determination of complex shear modulus and phase angle using a Dynamic Shear Rheometer), and the equipment used for this procedure was a Dynamic Shear Rheometer (DSR) by Bohlin Instruments, shown in Figure 17.

Figure 17 – Dynamic Shear Rheometer (DSR) by Bohlin Instruments

The procedure involves determining the complex shear modulus and phase angle of the asphalt binders over a range of test frequencies and temperatures when tested in oscillatory shear. This procedure involves determining the viscoelasticity of the bitumen, which is characterized by the phase angle, δ, the complex modulus, G^*, the elastic modulus, G' and the viscous modulus, G''. The DSR rheometer applies torsional strains to bitumen samples at a frequency of 10 rad/s, while measuring the torque applied to achieve these strains. By knowing the torque effort and the respective torsion deformation, it is possible to determine the rheological properties of the bitumen.

The bitumens were tested at different temperatures, ranging from 19 °C to 88° C. The rheological properties of the binders are determined with different plates according to the tested temperatures. For the lower temperatures, between 19 °C and 40 °C, a smaller plate of 8 mm in diameter is used with a bitumen sample with 2 mm of thickness. For the highest temperatures, from 46 °C to 88 °C, a larger plate with 25 mm in diameter is used with a bitumen sample with a thickness of 1 mm (Figure 18). This change of the plates ensures a linear rheological behaviour of the bitumen.

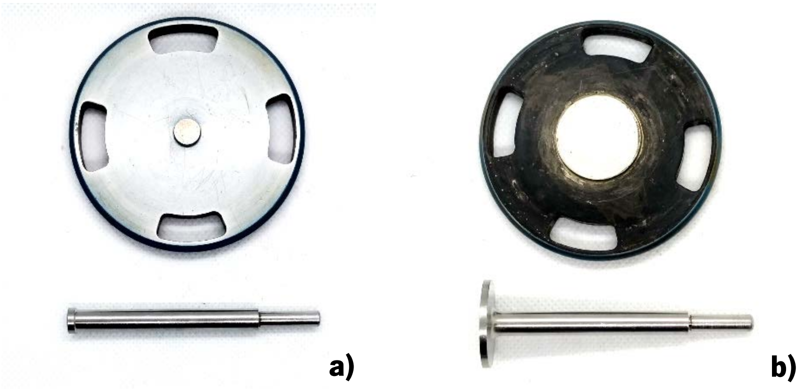


Figure 18 – (a) 8 mm plate; (b) 25 mm plate

The characterization of the rheological properties of the bitumen is given by different properties, as previously stated. One of these properties is the phase angle, δ, which provides a relative indication of the viscous and elastic behaviours of the binder, with values between 0°, for a fully elastic material, and 90°, for a total viscous binder. At intermediate temperatures (for example, at 19 °C), asphalt binders are said to be viscoelastic with a phase angle near 45° (Asphalt Institute, 2019). Another main property is the complex shear modulus, G*, which measures the stiffness of the asphalt binder, and can be understood as the complex sum of the elastic and viscous components of the material, which are represented by the elastic modulus, G', and the viscous modulus, G'' (Domínguez, 2018).

3.4.6. Elastic recovery

The elastic recovery is a measure of the return capacity of an asphalt binder after an interruption of specified mechanical stress. The method that allows this measurement is also named elastic recovery method and is essentially a procedure performed to determine the elastic recovery of asphalt binders in a ductilometer at a specific test temperature (typically 25 °C or 10 °C although other temperatures can be used). It is especially applicable to PMBs modified with thermoplastic elastomers, but can also be used with other asphalt binders even though these will generate a small recovery. This process is carried out according to the European Standard EN 13398 (Bitumen and bituminous binders - Determination of the elastic recovery of modified bitumen). Samples must be stretched at a speed of 50 mm/min at the stipulated temperature until they reach a predetermined elongation of 200 mm, in order to determine the elastic recovery. Then, they are cut in the central section and, after 30 minutes, the distance recovered between the two halves is measured, as exemplified in Figure 19.

Figure 19 – Elastic recovery measurement

The water bath shall be temperature controlled, capable of maintaining the specimen and the attachment device at the specified temperature throughout the test to an accuracy of ± 0,5 °C. The test must be carried out on at least two specimens in parallel, which should be conditioned in the water bath at the test temperature for no less than 90 minutes. The mould used to obtain these specimens is the one shown in Figure 20. The test samples were prepared following the European Standard EN 12594 (Bitumen and bituminous binders. Preparation of test samples), as specified in EN 13398.

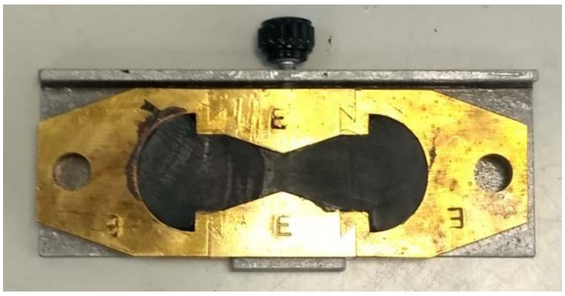


Figure 20 – Elastic recovery mould

The final elastic recovery value R_E is given by Equation 2.

$$R_E\ (\%) = \frac{d}{L} \times 100 \tag{2}$$

where,

d = distance between half-threads, in mm;

L = stretching length, in mm (for a premature break due to brittleness, L is the elongation at break).

In the current work, this method was performed at two test temperatures, +5 °C and +20 °C, for both polymer-modified binders, PMB25 and PMB50.

3.4.7. Force ductility method

Ductility is the ability of a material to undergo a visible enduring deformation through elongation (decreasing of cross-section area) without breaking. It expresses the extent to which the material can be plastically deformed without fracture. In practical terms, it is defined as the distance, in millimetres, to which a standard sample of the material will be stretched without breaking. The method was performed according to the European Standard EN 13589 (Bitumen and bituminous binders - Determination of the tensile properties of modified bitumen by the force ductility method). The asphalt binder sample must be stretched 50 mm/min in a ductilometer at the stipulated temperature until it reaches a predetermined elongation of 400 mm or until it breaks, as shown in Figure 21.

Figure 21 – Elongation of force ductility specimens

The water bath shall be temperature controlled, capable of maintaining the specimen and the attachment device at the specified temperature throughout the test to an accuracy of ± 0.5 °C. The test must be carried out on at least two specimens in parallel, which should be conditioned in the water bath at the test temperature for no less than 90 minutes. The mould used to obtain these specimens is the one shown in Figure 22. The test samples were prepared following the European Standard EN 12594, as specified in EN 13589. The force ductility is given by the deformation energy, E_i by using Equation 3.

$$E = \int_{L1}^{L2} F(x)dx = \Delta L \times (\frac{F_0}{2} + \frac{F_n}{2} + \sum_{1}^{n-1} F_i) \tag{3}$$

where,

L1 = length at 0.200 m elongation;

L2 = length at 0.400 m elongation or the length at break;

ΔL = incremental length between force determinations; normally 0.005 m;

F0 = force at 0.200 m elongation;

Fi = force at (L1 + i × ΔL);

Fn = force at break or 0.400 m elongation;

n = total amount of force values used in Equation 3 (n = (L2 – L1) / ΔL), if needed rounded to the nearest integer value.

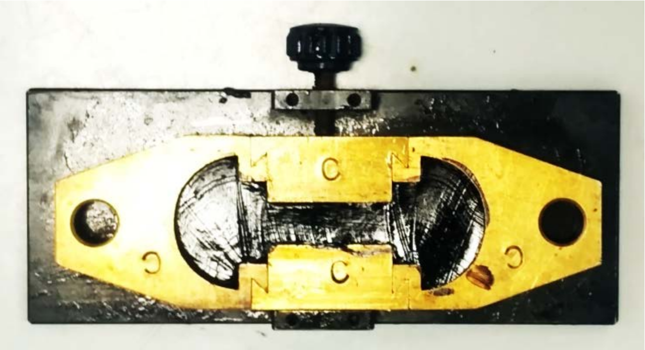


Figure 22 – Force ductility mould

The cohesion energy is also a property obtained from this procedure and is given by Equation 4.

$$Cohesion\ energy = \frac{E_i}{cross\ section\ (mm^2)} \tag{4}$$

Cohesion is a measure of the tensile stress required to break the bond between molecules of the asphalt binder. The inherent strength, tenacity and toughness of the asphalt binders are improved by modification with thermoplastic polymers and rubber crumbs. Hence, higher tensile stress is required to break the molecular bonds of modified binders and cause failure compared with lower tensile stress required to break the bonds of conventional binders (SABITA, 2015).

This method was performed at two test temperatures, +5 °C and +20 °C, for both polymer-modified binders studied in this work (PMB25 and PMB50).

3.5. Asphalt mixtures

After performing the several laboratory tests to characterize in detail both the aggregates and the asphalt binders, the asphalt mixtures were then selected and produced.

The asphalt mixtures follow the indications provided by the European Standards EN 13108-1 (Bituminous mixtures - Material specifications - Part 1: Asphalt Concrete) and EN 13108-5 (Bituminous mixtures - Material specifications - Part 5: Stone Mastic Asphalt).

The standard EN 13108-5 specifies the requirements for mixtures of the group Stone Mastic Asphalt for use on roads, airfields and other trafficked areas. Although the specification for the SMA mixture provided in this European Standard has yet to be transposed to Portuguese norms, successful mix designs have already been applied by *Infraestruturas de Portugal, S.A.*, the state-owned company in charge of managing the rail and road infrastructure.

The European Standard provides the designation of the mixture as follows: SMA D Binder, where SMA is Stone Mastic Asphalt, D is the upper sieve size, and Binder refers to the binder grade or class in the final product. In this study, a Stone Mastic Asphalt mixture with a maximum aggregate size of 11 mm was selected for a surface course, for both asphalt mixtures, and the modified bitumens penetration grades will be accessed further ahead, but the base bitumen will be used here as an exemplification. In that example, the designation of the asphalt mixture will be SMA 11 50/70, with the 50/70 being replaced by the obtained penetration grades for each polymer-modified bitumen.

Stone Mastic Asphalt or SMA for short, is a type of graded asphalt mixture that consists of a high concentration of coarse aggregate held together by a matrix of mineral fillers and stabilizers and a thick bitumen film (Shekar *et al.*, 2018).

The final composition of both asphalt mixtures shall be comprised of 6% PMB and 94% aggregates by weight of the mixture. Even though there is no specific standard to determine a minimum or maximum content of the polymer to use when it concerns modified bitumens, the selected percentage was based on previous research by many different authors such as Shinohara *et al.* (2011), Fernandes *et al.* (2016), Saboo and Kumar (2016), Torres (2014) and Porto *et al.* (2019), among many others.

3.5.1. Asphalt mixtures production

After the selection and production of the polymer-modified bitumens and subsequent laboratory analysis to understand further their characteristics as well as the final grading composition of the aggregates, the conditions are met to produce the final asphalt mixtures. This process will entail two parts: a first phase, for mixture production, and a second phase, for slabs compaction. These asphalt mixtures for the surface course were prepared using three asphalt binders included in this study, using the same aggregates.

According to EN 13108-5, the minimum and maximum temperatures of the mixture shall be given by Table 5, noticing that higher temperatures may be applicable when using modified bitumen or hard grade bitumen or additives.

Table 5 – Temperature limits of the mixture depending on the bitumen grade

Bitumen grade	Temperature (°C)
30/45, 35/50, 40/60	160 to 200
50/70	150 to 190
70/100	140 to 180
100/150, 160/220	130 to 170
250/330, 330/430	120 to 160

From the rheology results obtained by the DSR procedure as well as the rotational viscometer, along with the reasoning provided by several authors with years of experience in this field of expertise, the temperatures selected for production and compaction can be observed in Table 6. The justifications for these values are further analysed during the rheology characterization of the asphalt binders.

Table 6 – Conditions for production and compaction of the asphalt mixtures

Condition	PMB25 mixture	PMB50 mixture
Mixing temperature (°C)	170	170
Mixing time (minutes)	2	2
Compaction temperature (°C)	170	170

For the production of the asphalt mixtures, the process is as follows: firstly, the aggregates are weighted and heated, while the binder is also being heated. Secondly, the aggregates are introduced in a mixer, after which the binder is weighted and also introduced. The equipment is then started, and a timer is set, after two minutes this process is stopped. These processes can be seen in Figure 23.

When discharged from the mixer, the asphalt mixture should be homogenous in appearance with the aggregate wholly coated with the asphalt binder, and there should be no evidence of clustering of fine aggregates. The mixture is then spread in a mould with the slab dimensions, shown in Figure 24 (a), and carried to the compaction equipment. The compaction of the different asphalt mixtures in slabs was carried out using the heated asphalt rolling compactor shown in Figure 24 (b).

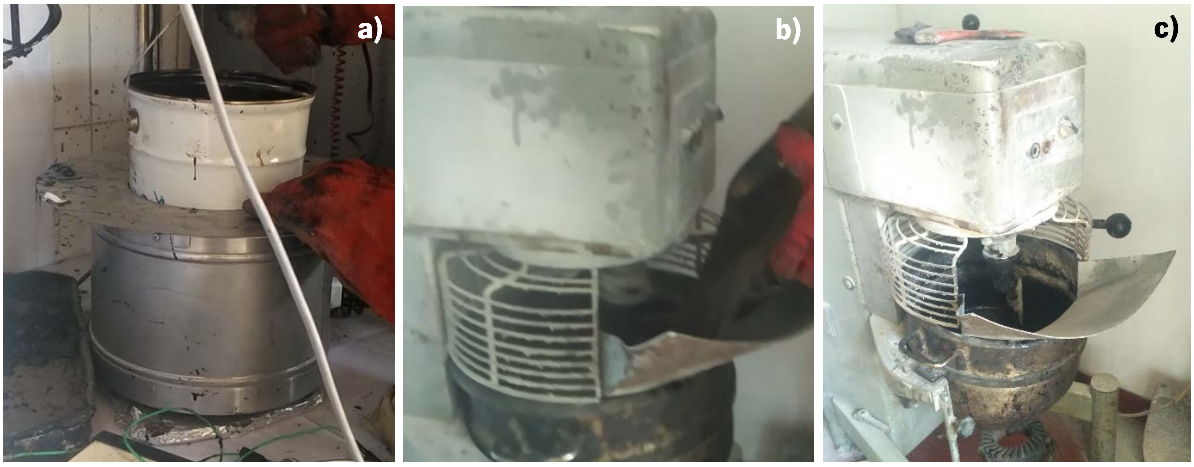


Figure 23 – (a) Heating the binder; (b) Introducing the aggregates; (c) Mixing the asphalt mixture

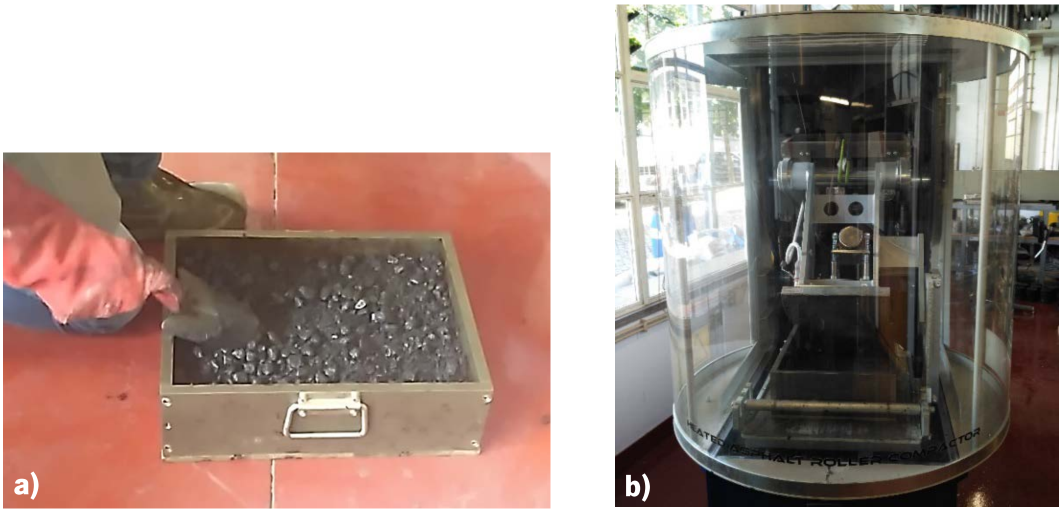


Figure 24 – (a) Mixture spread in the mould; (b) Rolling compactor

The specimens were obtained by sawing them from slabs (Figure 25) made in the laboratory according to EN 12697-33 (Bituminous mixtures - Test method - Part 33: Specimen prepared by roller compactor).

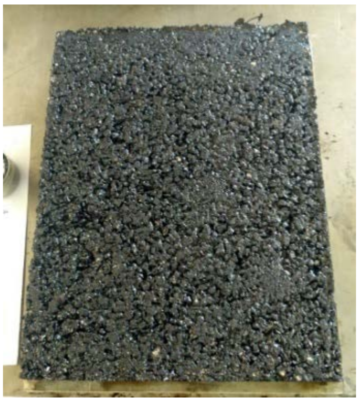


Figure 25 – Asphalt mixture slab after compaction

3.5.2. Asphalt mixtures characterization

Asphalt mixtures can be characterized by calculating the air voids content in the compacted asphalt mixture as well as other volumetric-related properties. Some of those properties are the maximum density, together with the bulk density and, of course, the air voids content.

This type of volumetric analysis is required to be performed for each type of mixture, since it is possible to obtain slightly different values from the ones the mixture was designed to, incurring in possible derived consequences on the performance of the mixture over time if the composition is not adjusted.

- Maximum density determination by the procedure A

This process is performed according to the European Standard EN 12697-5 (Bituminous mixtures - Test methods - Part 5: Determination of the maximum density). The maximum density procedure aims to determine the void volume of a mixture based on the mass and volume ratio, without voids, at a given temperature. This process can be performed by three different procedures: A, a volumetric procedure, B, a hydrostatic procedure and C, a mathematical procedure. The method selected in this work is A, the volumetric procedure. In the volumetric procedure, the sample volume is measured by the displacement of water in a pycnometer. The volume of the sample should be without air voids.

In method A, a dry sample of a mixture, not compacted, is placed inside a pycnometer of known volume and weight, and the whole is weighed and recorded. The pycnometer is immediately filled with distilled water, and the air is removed from the interior using a 4.0 kPa vacuum for 15 minutes together with a vibrating table. The device and the vibrating table are shown in Figure 26.

Figure 26 – Pycnometer and vibrating table

Then, the pycnometer is placed in a water bath at 25 °C for 30 minutes and, the whole system is weighed and recorded another time. The maximum density is determined by Equation 5.

$$\rho_{mv} = \frac{m_2 - m_1}{10^6 \times V_p - \dfrac{m_3 - m_2}{\rho_w}} \tag{5}$$

where,

ρ_{mv} = maximum density of the bituminous mixture as determined by the volumetric procedure, in megagrams per cubic metre (Mg/m^3) to the nearest 0.001 Mg/m^3;

m_1 = mass of the pycnometer plus headpiece and spring, in grams (g);

m_2 = mass of the pycnometer plus headpiece, spring and test sample in grams (g);

m_3 = mass of the pycnometer plus headpiece, spring, test sample and water in grams (g);

V_p = volume of the pycnometer, when filled up to the reference mark, in cubic metres (m^3);

ρ_w = density of the water at test temperature, in megagrams per cubic metre (Mg/m^3) to the nearest 0.001 Mg/m^3.

- Air voids content

This process is performed according to the European Standard EN 12697-8 (Bituminous mixtures - Test methods - Part 8: Determination of void characteristics of bituminous specimens). The air voids content of an asphalt specimen (Equation 6) is calculated using the maximum density of the mixture and the bulk density of the specimen, which is explained further ahead.

$$V_a = \frac{\rho_m - \rho_b}{\rho_m} \times 100 \tag{6}$$

where,

V_a = air voids content of the bituminous specimen, in 0.1% (by volume);

ρ_m = maximum density of the mixture, in megagrams per cubic metre (Mg/m^3);

ρ_b = bulk density of the specimen, in megagrams per cubic metre (Mg/m^3).

3.5.3. Specimens production

The specifications for the dimensions required for the specimens to evaluate their low-temperature mechanical characteristics are described in the European Standard EN 12697-46 (Bituminous mixtures - Test methods for hot mix asphalt - Part 46: Low temperature cracking and properties by uniaxial tension tests). The specimen must be either a prismatic or a cylindrical beam with nominal dimensions according to Table 7, and their ends must be perpendicular to its axis.

Table 7 – Dimensions for prismatic and cylindrical specimens presented in EN 12697-46

Specimen type	Dimension	Maximum aggregate size D (mm)		
		D ≤ 11.2	11.2 < D < 22.4	D ≥ 22.4
Prismatic specimens with a square cross-section	Width and height (mm)	40 ± 2	50 ± 2	60 ± 2
Cylindrical specimens	Diameter (mm)	50 ± 2	50 ± 2	60 ± 2
All specimens	Minimum length (mm)	160	160	160

The minimum dimensions allowed for the specimens take into account the size of the aggregates and the ability to test the mixture without changing the aggregate distribution. Those dimensions should reflect, as much as possible, the real conditions of the mixture in the pavement. Conversely, the testing device used for the mechanical tests, the servo-controlled universal testing machine CS7400S, has a limited internal height with the plates in place of 160 mm. As a third challenge, the executing of such a quantity of cylindrical specimens would be an enormous task, in opposition to sawing the slabs into prismatic specimens. The final limitation pertains to the maximum dimension of the aggregate of the studied mixtures, since the aggregate constitution is the same for all of them, the dimensions of the specimens will also be the same. Based on all these restrictions, the selected and more feasible solution was to opt for a prismatic specimen with the following dimensions: height of 160 mm (the minimum allowed by the standard, being limited by the test device) and square cross-section of 40 mm, given that the maximum aggregate size of the studied mixtures is 11.2 mm. Figure 27 shows the structure of the final specimen.

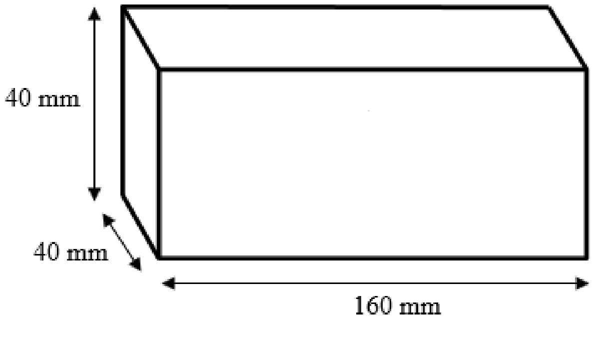


Figure 27 – Adopted specimen, with dimensions in mm

After defining the dimensions of the specimens, their distribution in the slabs was optimized according to the standard restrictions. Five slabs were produced for each mixture. Each slab was divided into ten specimens, with the configuration shown in Figure 28.

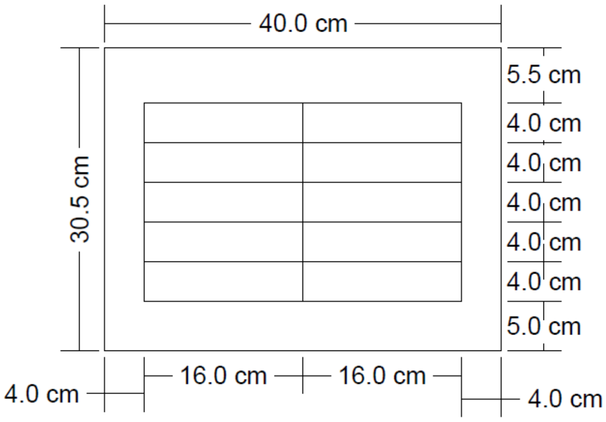


Figure 28 – Specimens distribution in the slab, with dimensions in cm

The slabs were sawed according to the European Standard EN 12697-33. The specimens were mainly sawed from the slab central part to assure the required distance of 20 mm to its border. The final number of specimens obtained was 50 for each type of mixture.

The only requirement given by the standard that was not possible to be met was that the slabs made in the laboratory should have at least a thickness of the required height. That condition could not be respected because the rolling compactor has a limited slab height below the specimens minimum height, so to overcome this difficulty, the slabs had to be made with the height correspondent with the cross-section dimension plus an additional 20 mm on both top and down, making the final height 80 mm.

3.5.4. Specimens characterization

The bulk density (BD) can be determined using several methods, which are presented in the European Standard EN 12697-6 (Bituminous mixtures - Test methods for hot mix asphalt Part 6: Determination of bulk density of bituminous specimens). This determination aims to obtain the mass per unit volume, including the air voids, of a specimen at a known test temperature, or bulk density. This process can be performed by four different procedures: A, a dry method, B, a dry and saturated surface method (SSD), C, a sealed method and D, a method by dimension determination. All the methods, except method D, require the recording of the water temperature and subsequent density determination, due to a weighing influence this value presents in the final calculations.

Method A, dry method, consists of weighing the mass of the test pieces dry, submerging them in the water and immediately weighing them in the water after they have settled. Procedure B consists of weighing the test pieces dry, then placing them submerged in water for 30 minutes until they are fully saturated, after which they are again weighed while in water. They are then taken from the water, and their surfaces are dried with a damp Chamois so they can be weighed again. The method C involves again weighing the specimens dry, sealing the specimen with a film (so no voids are penetrated by water, and no other voids are included between the seal and the surface), and weighing this sealed specimen, submerging it in water and weighing it in water. Finally, the method D comprises determining the dimensions of the specimen and subsequently measuring the corresponding mass. During this research work, all methods described in the European Standard were used to obtain the bulk density for each specimen and provide a comparison between them, except method C, which was only utilized on three specimens for logistic reasons. Figure 29 shows the apparatus used for weighing the specimens in water.

Figure 29 – Bulk density weighing apparatus

Equations 7 to 10 used in each method for bulk density determination are presented below.

- Procedure A:

$$\rho_{bdry} = \frac{m_1}{m_1 - m_2} \times \rho_w \tag{7}$$

- Procedure B:

$$\rho_{bssd} = \frac{m_1}{m_3 - m_2} \times \rho_w \tag{8}$$

where,

ρ_{bdry} = bulk density dry, in kilograms per cubic metre (kg/m^3);

ρ_{bssd} = bulk density (SSD), in kilograms per cubic metre (kg/m^3);

m_1 = mass of the dry specimen, in grams (g);

m_2 = mass of the specimen in water, in grams (g);

m_3 = mass of the saturated surface-dried specimen, in grams (g);

ρ_w = density of the water at test temperature, in kilogram per cubic metre (kg/m^3).

- Procedure C:

$$\rho_{bsea} = \frac{m_1}{(m_2 - m_3/\rho_w) - (m_2 - m_1/\rho_{sm})} \tag{9}$$

where,

ρ_{bsea} = bulk density sealed, in kilograms per cubic metre (kg/m^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 test temperature, in kilogram per cubic metre (kg/m^3).

ρ_{sm} = density of the sealing material at test temperature, in kilograms per cubic metre (kg/m^3);

- Procedure D:

$$\rho_{b,dim} = \frac{m_1}{h \times l \times w} \times 10^3 \tag{10}$$

where,

$\rho_{b,dim}$ = bulk density of the specimen, in kilograms per cubic metre (kg/m^3);

m_1 = mass of the dry specimen, in grams (g);

h = height of the specimen, in millimetres (mm);

l = length of the specimen, in millimetres (mm);

w = width of the specimen, in millimetres (mm).

3.6. Mechanical methods of characterization of the asphalt mixtures

The methods by which the mechanical properties and characteristics of asphalt mixtures are evaluated are based on the European Standard EN 12697-46 (Bituminous mixtures - Test methods for hot mix asphalt - Part 46: Low temperature cracking and properties by uniaxial tension test). This standard (CEN, 2012) details the devices and conditions required to evaluate an asphalt mixture under low-temperature conditions and is, therefore, fundamental to the analysis carried out during this research work.

3.6.1. Bonding agent

The bonding agent is applied for the mechanical tests, and it works as a glue between the plates and the specimen to hold it in place and allow the tensile tests to be performed.

The adopted bonding agent is a two-part, fast curing, steel-filled epoxy putty by Devcon. According to its technical sheet, it hardens one hour after mixing and application and fully cures in 16 hours at 21 °C. The two parts consist of a hardener and a resin, shown in Figure 30.

Figure 30 – (a) Bonding agent; (b) Mixing board and spatula

The mixing should be made on a mixing board using a spatula and for best results at room temperature of around 21 °C; it should be thoroughly mixed by adding the hardener to the resin in a ratio of 1:1 for one minute since it only has a 5-minute pot life.

3.6.2. Testing devices and other conditions

The mechanical methods used to characterize the specimens require an apparatus consisting of a thermostatic chamber with forced air circulation in which the specimen can be conditioned and in which the test can be performed. The chamber temperature must be maintained during the tests, between -40 and 30 °C (with an accuracy of ± 0.5 °C). The thermostatic chamber must be capable of enabling a temperature rate in the core of the specimen of 10 °C/h.

Inside this thermostatic chamber, it must be located a loading device able to generate movements with an accuracy of 0.1 µm. The specimen should be connected to the loading device by two gimbal suspensions to avoid radial and transversal forces as well as moments in the test specimen.

The device utilized was the servo-controlled universal testing machine CS7400S with fixed endplates. This equipment has a load cell connected to the vertical actuator with a capacity of 22 kN, being particularly suitable for small specimens. However, it does not meet the entire temperature requirements since it is only able to maintain a minimum low-temperature of approximately -20 °C, under the current set conditions, which can be a challenge for some of the tests. The device is presented in Figure 31, as well as its thermostatic chamber.

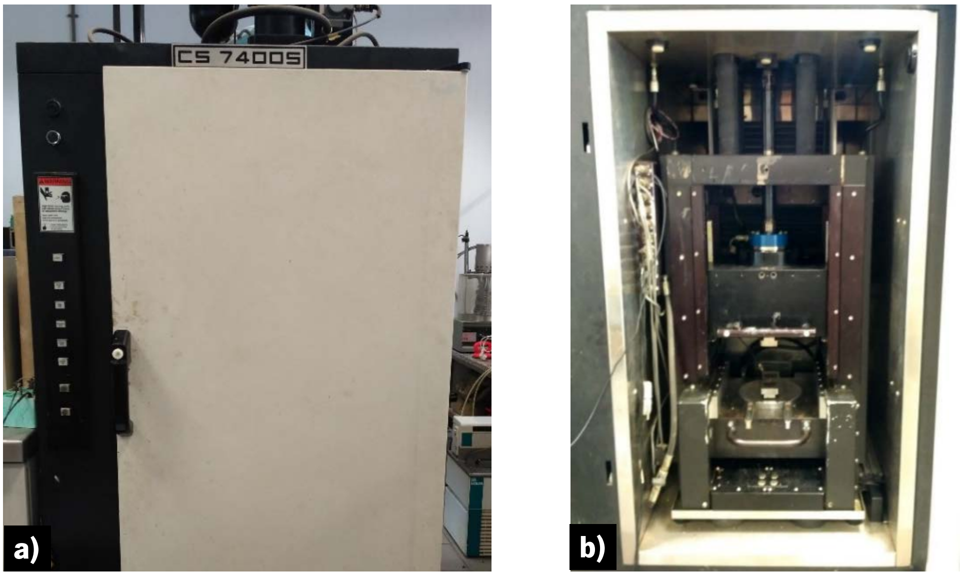


Figure 31 – (a) CS7400S device; (b) Interior of the chamber

As previously mentioned, the distance between plates is limited to 160 mm. A detail of the loading device is shown in Figure 32. The testing device for the uniaxial cyclic tensile stress test (UCTST) requires some special conditions in addition to the ones of all other mechanical tests. The loading device must be a dynamic testing device consisting of a bending-resistant load frame with at least two supports, a temperature chamber, a hydraulic system and a control unit controlling force or displacement. The testing device must be capable of applying a dynamic load of at least the applied test frequency with an accuracy of 0.1 Hz along the longitudinal axis of the test specimen. The load should be sinusoidal with or without a rest period. The same servo-controlled universal testing machine CS7400S was utilized for the UCTST since it is capable of meeting all the requirements established by the standard.

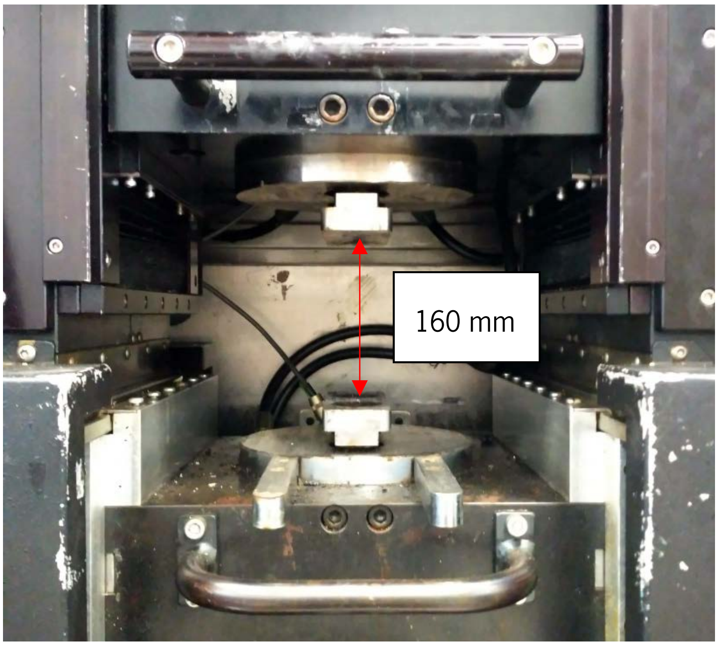


Figure 32 – Detail of the loading device

According to the Standard EN 12697-46, at least three specimens must be tested for each asphalt material and test condition (temperature and level of stress) combination. Initially, the logistics were in place to meet this requirement. However, so many obstacles appeared during the experiments, that this condition was not met for all tests.

In order to glue the two adapters to the specimen, the standard suggests the use of a mounting bench where the two adapters can be mounted vertically, and the specimen adjusted horizontally to them through height-adjustable support. This support is used to create a positive centric connection between the test specimen and the two connectors. There was no mounting bench available, but after several different configurations in different devices, a similar device was arranged, where the adapters are mounted horizontally, and the specimen is glued vertically to them. The mounting device needs to hold the top connector in place to avoid deformations of the specimen due to the weight of the top plate.

The test system must be equipped with a system monitoring the load acting on the test specimen with a load cell that shall have a minimum measuring range of ±15 kN with an accuracy of ±10 N. The displacement transducers shall have a minimum measuring range of ±2.5 mm with an accuracy of ±5 μm. Both the load cell and the two chosen displacement transducers, Linear Variable Differential Transformer (LVDT), meet these requirements, having a third internal transducer in the testing machine CS7400S, which also meets the conditions. Figure 33 presents the final system used in the tests.

Figure 33 – Specimen glued to connectors and measuring devices (LVDT)

The specimen will be installed into the test device by joining the adapters with the load device. The specimen must be stabilized at the test temperature T (or T0, in case of TSRST) before starting the test for an adequate duration without applying any load. During the conditioning phase, the closed-loop control of the test device must ensure that the specimen is not subjected to any loads. The duration of this phase depends on the specimen size and the material tested, but it must ensure that the temperature in the specimen is constant within ±1 °C of the test temperature for not less than 10 minutes. A dummy specimen is placed near the specimen being tested for that verification. This dummy specimen consists of a specimen of the same asphalt mixture as the tested specimen, but with a temperature measuring device installed in its core to emulate the temperature of the tested specimen. Figure 34 presents one of the dummy specimens utilized.

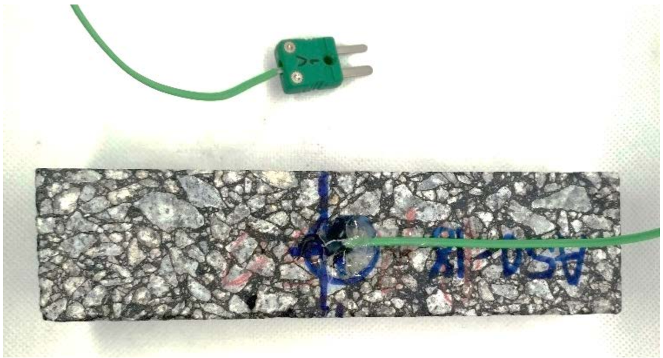


Figure 34 – Dummy specimen used for temperature control

3.6.3. Uniaxial tension stress test

In the uniaxial tension stress test (UTST), a specimen is pulled with a constant strain rate at a constant temperature until failure. Results of the UTST are the maximum stress (tensile strength) β_t(T), and the corresponding tensile failure strain $\varepsilon_{failure}$ (T) at the test temperature T. The principle of this test method is shown in Figure 35. As previously stated, the specimens are subjected to a strain-dependent test on time, which is based on the initial length. The constrain strain rate (dε) is equal to 0.625 ± 0.025 %/min.

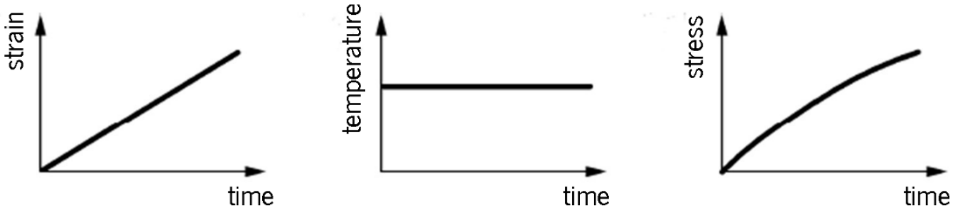


Figure 35 – Test principle of UTST

For each temperature, three specimens were tested, in order to have a more precise range of results. The results obtained from this mechanical test are the tensile strength βt (MPa) and the failure strain $\varepsilon_{failure}$ at each temperature. These are respectively calculated according to Equations 11 and 12.

$$\beta t(MPa) = \frac{F_{failure}(N)}{A(mm^2)} \tag{11}$$

$$\varepsilon_{failure}(‰) = \frac{\varepsilon(mm)}{L(mm)} \times 1000 \tag{12}$$

where,

βt = tensile strength in MPa;

$F_{failure}$ = measured tension force at failure in N;

A = initial cross-section in mm^2;

$\varepsilon_{failure}$ = failure strain in permillage (‰);

ε = measured strain at the failure time in mm;

L = initial length in mm.

3.6.4. Thermal stress restrained specimen test

In the thermal stress restrained specimen test (TSRST), the specimen, whose length is held constant, is subjected to a decrease in temperature with a constant temperature rate. Cryogenic stress is built up in the specimen, due to the prohibited thermal shrinkage. This procedure permits obtaining results concerning the progression of the cryogenic stress over the temperature σ_{cry} (T), and the failure stress $\sigma_{cry,failure}$ at the failure temperature $T_{failure}$. The failure stress is equivalent to the strength of the specimen at the failure temperature (Pszczola and Szydlowski, 2018).

This test was performed with three different rates of decrease in temperature in order to observe the effect this cooling rate has on TSRST results and, indirectly, on the tensile strength reserve analysis, explained further ahead. The starting temperature, T0 = +20 °C, recommended to initiate the test, was observed, as well as the cooling rate indicated by the Standard, dT = -10 °C/h. According to Pszczola *et al.* (2019b) the cooling rate affects the experimental measurements in this test significantly. To determine this effect, two additional cooling rates were selected: dT = -2 °C/h and dT = -5 °C/h. This selection was made based on the research made by Pszczola *et al.* (2016) in which the pavement temperatures are analysed in a country with a climate representative of low-temperature conditions. In that study, it was observed that a cooling rate of -2 °C/h has a probability of occurrence of 99%, which can be a good representation of the typical low-temperature conditions during a considerable portion of the winter. However, and although the probability of occurrence of cooling rates lower than -3 °C/h is less than 1%, it is significant to notice that low-temperature cracks are initiated by a combination of the most adverse factors. Then, the selected -5 °C/h cooling rate can be an adequate representation of more extreme conditions, that even though may happen more infrequently, their effect can be more severe on the pavement when they occur. The principle of this test method is shown in Figure 36.

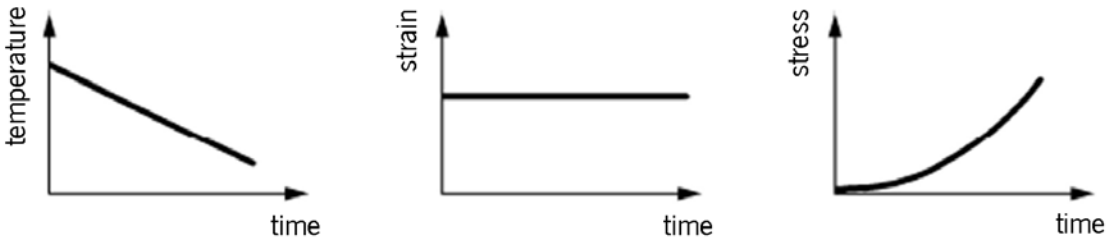


Figure 36 – Test principle of TSRST

Based on UTST and TSRST tests, and their results, it is possible to determine the tension strength reserve, Δβt(T), which the mixture can support under external loads in addition to the low-temperature thermal stresses (Figure 37). It is calculated as the difference between the tensile strength, βt(T) – obtained from the UTST as a temperature/tensile strength diagram using a cubic spline function – and the cryogenic stress σ_{cry}(T) – obtained from the TSRST at the same temperature T using the Equation 13.

$$\Delta\beta_t(T) = \beta_t(T) - \sigma_{cry}(T) \qquad (13)$$

where,

Δβt(T) = tensile strength reserve in MPa;

βt(T) = tensile strength in MPa;

σ_{cry}(T) = cryogenic (thermal) stress in MPa.

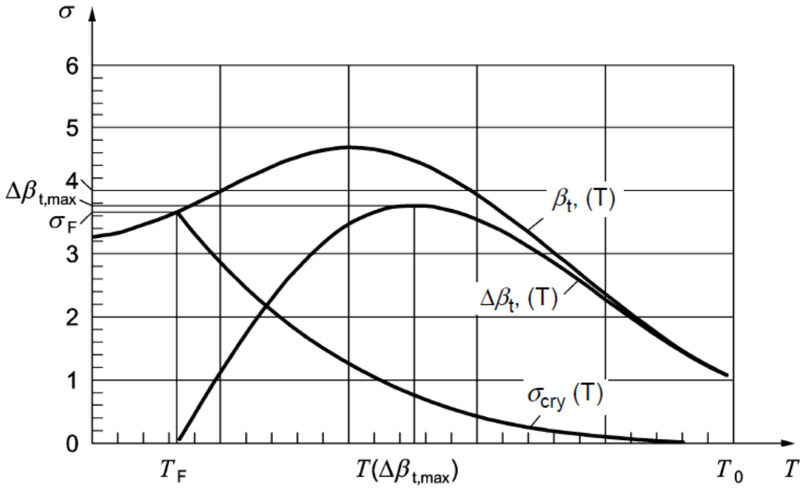


Figure 37 – Principle of the tensile strength reserve

3.6.5. Relaxation test

In the relaxation test (RT), the specimen is subjected to a spontaneous strain ε, which is held on a constant level. The decrease of tension stress by relaxation over the testing time is monitored. The initial stress should not be higher than 75% of the tensile strength βt, obtained from the UTST tests. The relaxation time is the time when the stress is reduced to 36.8 ± 0.1% (1/e = 1/2.718 = 0.368) of its initial value. The results are the time of relaxation t_{rel}, and the remaining tension stress σ_{rem} after the test has ended. The principle of this test method is shown in Figure 38.

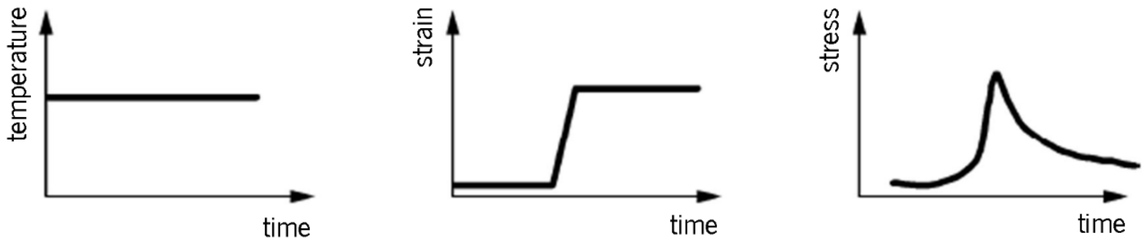


Figure 38 – Test principle of RT

3.6.6. Tensile creep test

In the tensile creep test (TCT), the specimen is subjected to a constant tension stress σ at a constant temperature T, during which the progression of the strain ε is measured. After a given time, the stress is withdrawn, and the regression of the strain is measured. Rheological parameters describing the elastic and viscous properties of the asphalt can be determined by interpreting the strain measurements. It is recommended to hold the constant load for 8 hours and record the regression after unloading for an additional 2 hours.

Table 8, presented in Standard EN 12697-46, suggests the test temperatures and suitable stress levels as a proportion of the tensile strength obtained in UTST. Besides, and since one of the selected test temperatures is -20 °C, the corresponding stress level proportion was interpolated between the adjacent values. The principle of this test method is shown in Figure 39.

Table 8 – Recommended test conditions for TCT

Temperature (°C)	The proportion of βt (T) (%)
-25	50
-20	43
-10	30
+5	10
+20	5

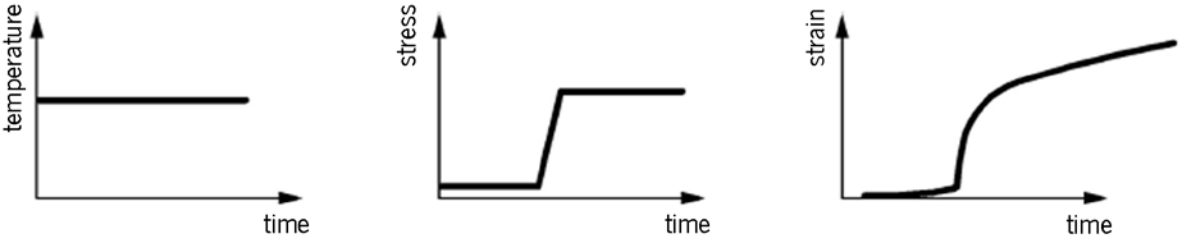


Figure 39 – Test principle of TCT

3.6.7. Uniaxial cyclic tensile stress test

In the uniaxial cyclic tension stress test (UCTST), a specimen is subjected to cyclic tensile stress which is characterised by sinusoidal stress, shown in Figure 40, to simulate the dynamic loading condition by traffic in combination with constant stress, which symbolises the cryogenic stress. During the test, the strain response is monitored, and the evolution of stiffness is recorded until the fatigue failure occurs. Results of the tests are the number of applied load cycles until failure $N_{failure}$ and the number of load cycles until the conventional fatigue criterion $N_{f/50}$ is reached.

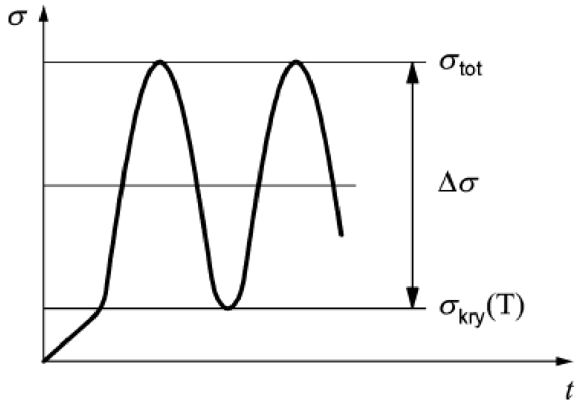


Figure 40 – Sinusoidal load applied in UCTST test

The fatigue behaviour can be characterised by relating the strain of a mixture to the number of load applications to failure. Fatigue testing can be conducted by either controlling the load (stress) or the deformation (strain) (Hassan *et al.*, 2014). The principle of this test method is shown in Figure 41.

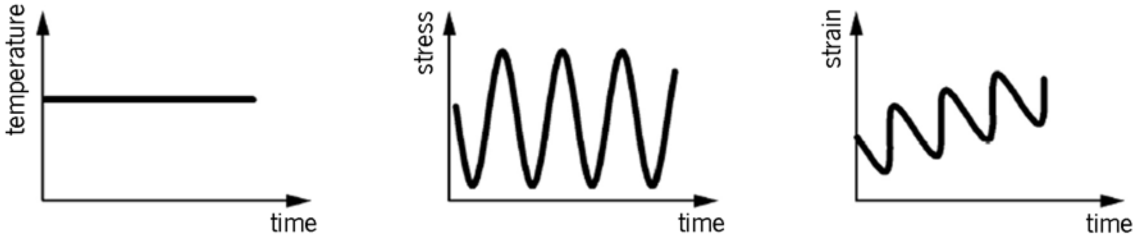


Figure 41 – Test principle of UCTST

The UCTST must be carried out at one or more test temperatures T and testing frequencies fr. It is recommended to use a testing frequency of 15 Hz and a test temperature of -15 °C. Because of the testing machine CS7400S condition of only being able to maintain a stable frequency of 10 Hz, all the tests were performed at this frequency. Concerning the temperatures, and to maintain the same logic, they were the same of all other mechanical tests. The base stress at test temperature T shall be derived from the TSRST, where the base stress corresponds to the cryogenic tensile stress $\sigma_{cry}(T)$ at test temperature T. The maximum stress σ_{tot} shall be the sum of the base stress $\sigma_{cry}(T)$ and the stress caused by the traffic load $\Delta\sigma$. For surface course asphalt mixes, traffic stress of $\Delta\sigma$ = 1,6 MPa is appropriate.

4. RESULTS AND ANALYSIS

> *"Experience does not ever err. It is only your judgment that errs in promising itself results which are not caused by your experiments."*
>
> Leonardo da Vinci

This report describes the results obtained from the previously described methodology and materials adopted in the study and discusses and analyses the corresponding findings and outcomes.

4.1. Analysis of the properties of the aggregate

As previously explained, after the sieving process is performed for each of the aggregate types or fractions, their particle size distribution is obtained, as shown in Table 9.

Table 9 – Particle size distribution for each aggregate type or fraction

Sieves (mm)	Filler Limestone	0/4 Granite	4/6 Granite	4/10 Granite	6/14 Granite
16	100%	100%	100%	100%	99%
14	100%	100%	100%	100%	94%
12.5	100%	100%	100%	100%	80%
10	100%	100%	100%	99%	43%
6.3	100%	95%	97%	19%	7%
4	100%	90%	11%	3%	2%
2	100%	72%	2%	2%	2%
0.5	100%	39%	2%	1%	2%
0.125	87%	11%	1%	1%	1%
0.063	65%	4%	1%	0%	1%

According to Table 9, several grain size distribution curves are obtained, one for each type of aggregate. These are shown in the graphs of Figure 42.

After obtaining the grain size distribution curves for all the types of aggregates were obtained, they were utilized to obtain an optimal grading composition for the asphalt mixtures.

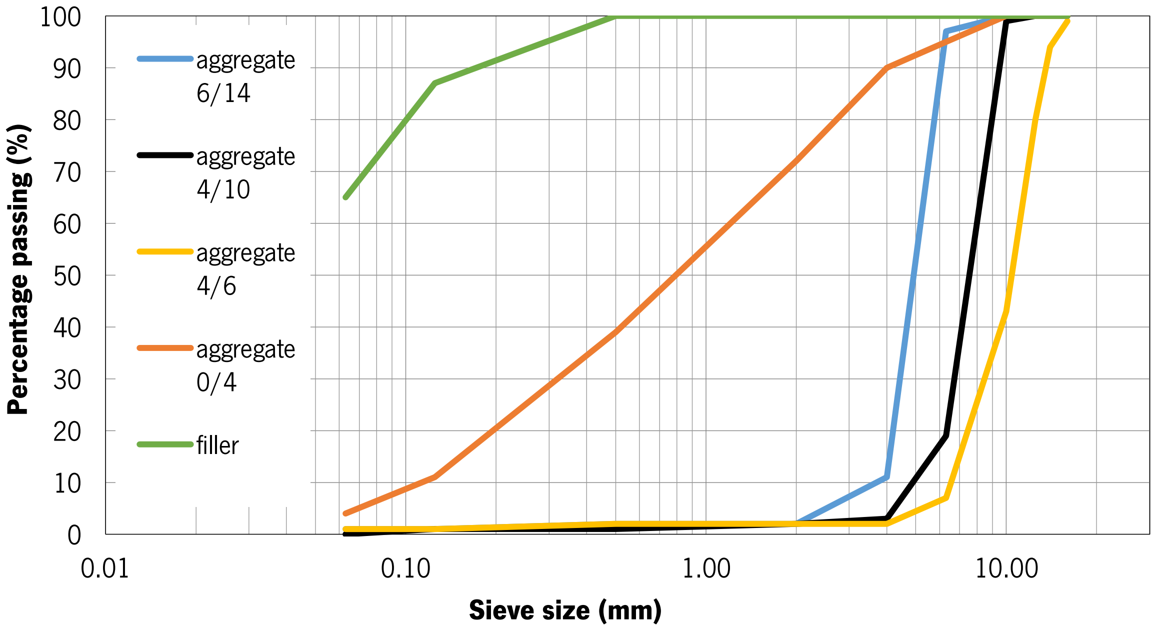


Figure 42 – Grain size curves for all the utilized aggregate types

It should be noticed that all mixtures were produced with the same amounts of each aggregate fraction because the optimal grading composition will be the same for all the studied asphalt mixtures. The selected optimal grading composition percentages of each aggregate type are shown in Table 10.

Table 10 – Percentages for each aggregate in the selected optimal grading composition

Aggregate type	Amount of each material used in the mixture
Filler	8 %
0/4	20 %
4/6	15 %
4/10	42 %
6/14	15 %

As can be perceived, the most used aggregate is the 4/10 type, with 42 % of the total. This situation is typical for SMA 11 mixtures, which will somewhat reflect the discontinuous graduation of the aggregates in this mixture that presents a stone-to-stone skeleton that is held together by a rich mixture of binder and filler (Behnood and Ameri, 2012). The high amount of filler (8%) used in this mixture is essential to obtain the necessary amount of mastic of SMA mixtures.

The optimized grading composition curve of SMA 11 mixtures used in this work is shown in Figure 43.

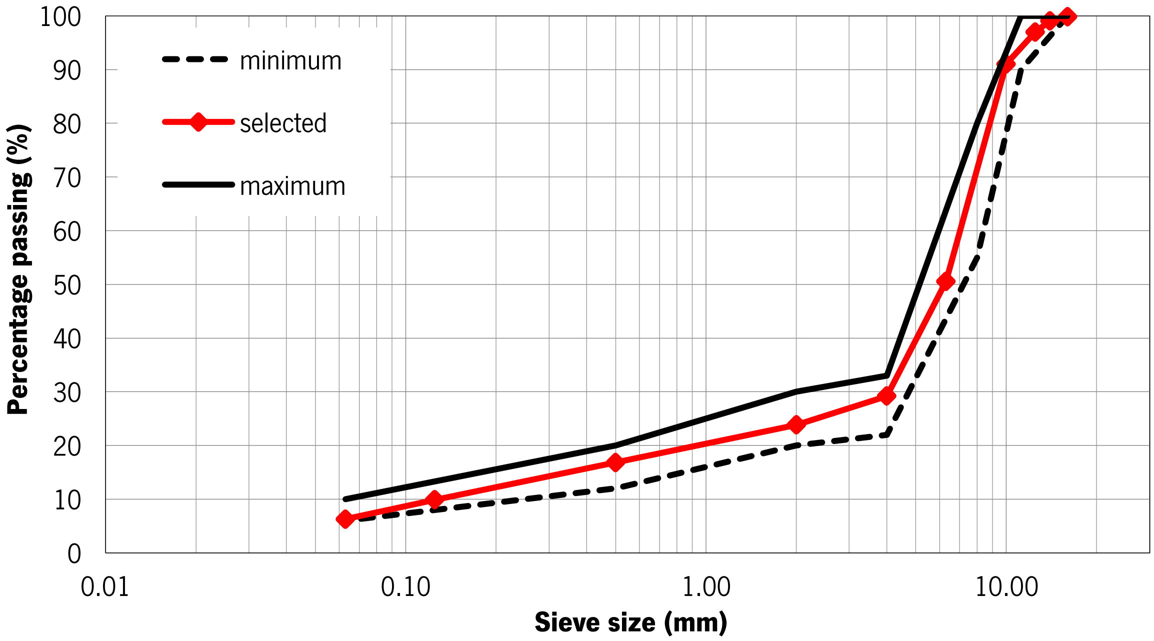


Figure 43 – Selected grading composition curve for SMA 11

The discontinuous nature of this type of mixture, SMA, can be observed. It can also be observed that the standard limits for the gradation were met using the mixture of aggregates previously presented.

4.2. Analysis of the asphalt binders

From acquired knowledge over time and experience from several authors and researchers, it can be understood that the binder is the component that most influences the performance of an asphalt mixture (Branco *et al.*, 2011). Thus, it is essential to establish parameters that permit to thoroughly analyse the asphalt binder in a given mixture and indirectly evaluate the corresponding mixture.

4.2.1. Softening point, penetration and resilience

The initial tests performed in order to characterize the physical properties of the asphalt binders were the softening point by the ring and ball method, the penetration procedure by the needle penetration method and the evaluation of the resilience by the penetration and recovery method. These processes, as well as their specificities and respective parameters, were described in a previous chapter.

The penetration and the softening point procedures were performed on both PMBs as well as the base bitumen. The resilience evaluation process was performed only on the PMBs because the base bitumen does not present resilient characteristics. Table 11 presents the results obtained from these three tests, after which an evaluation and discussion of these results are carried out.

Table 11 – Comparison of the basic properties of the different asphalt binders evaluated

Asphalt binder	Penetration at +25 °C (0.1 mm)	Softening point (°C)	Resilience (%)
Base 50/70 bitumen	55.2	50.5	-
PMB25	42.7	90.9	33%
PMB50	42.3	89.5	34%

The results of the base bitumen 50/70 are according to the limits of the standard (EN 12591) and technical data sheet for this product. The softening temperature values should vary between 46 to 54 °C and the penetration values, as the name indicates, must be between 50 and 70 tenths of a millimetre. Both of the results comply with the pre-established parameters, being therefor validated. There is no resilience value because this asphalt material is not modified with elastomers.

Regarding the PMBs, both have a lower penetration value in comparison with the base bitumen, not diverging a lot between them, with both PMB values being approximately the same. Thus, the PMBs are more viscous than the conventional bitumen, since the penetration grading's underlying assumption is the less viscous the binder, the deeper the needle will penetrate. This result will be further ahead confirmed with viscosity and rheology related tests.

Considering the softening point, the PMBs have, again, very similar values of temperature, but much higher than that of the conventional bitumen, as expected. This high softening point indicates that these PMBs are less susceptible to temperature, which is a required attribute considering the purpose of this research. Both PMBs are also substantially more stable to deformation since the higher temperature of the softening point increases the resistance of asphalt mixtures to permanent deformation (Remišová and Holý, 2017).

As expected, both PMBs have resilient properties, and the obtained values indicate a significantly good performance concerning their elastic recovery after penetration.

According to the European Standard EN 14023 (Bitumen and bituminous binders. Specification framework for polymer-modified bitumens), both PMBs evaluated in this work can be included in class 3 of penetration at +25 °C, (25-55 × 0.1mm) and classified as class 2 (≥ 80 °C) concerning the softening point temperature.

Both PMBs present similar performance in these tests, and they could be classified as PMB 25/55-80 hereafter. Nevertheless, to maintain the same nomenclature and avoid confusion between both asphalt binders, the PMB25 and PMB50 terminologies will be upheld.

4.2.2. Rheological characterization

The rheological analysis seeks to evaluate properties such as phase angle, δ (°), complex modulus, G* (kPa), elastic modulus, G' (kPa), viscous modulus, G'' (kPa), and viscosity (cP) for a temperature ranging between 19 °C and 190 °C. The DSR procedure permits to obtain these parameters for temperatures between 19 °C and 90 °C after which, for temperatures above 100 °C, only the viscosity parameter can be measured utilizing the rotational viscometer.

Figures 44 to 47 present, respectively, the results of the complex, elastic and viscous modulus, and phase angle of the various bitumens used in this study: i) the base binder 50/70; ii) the polymer modified bitumen with 2.5% SBS, PMB25, and; iii) the polymer modified bitumen with 5.0% SBS, PMB50.

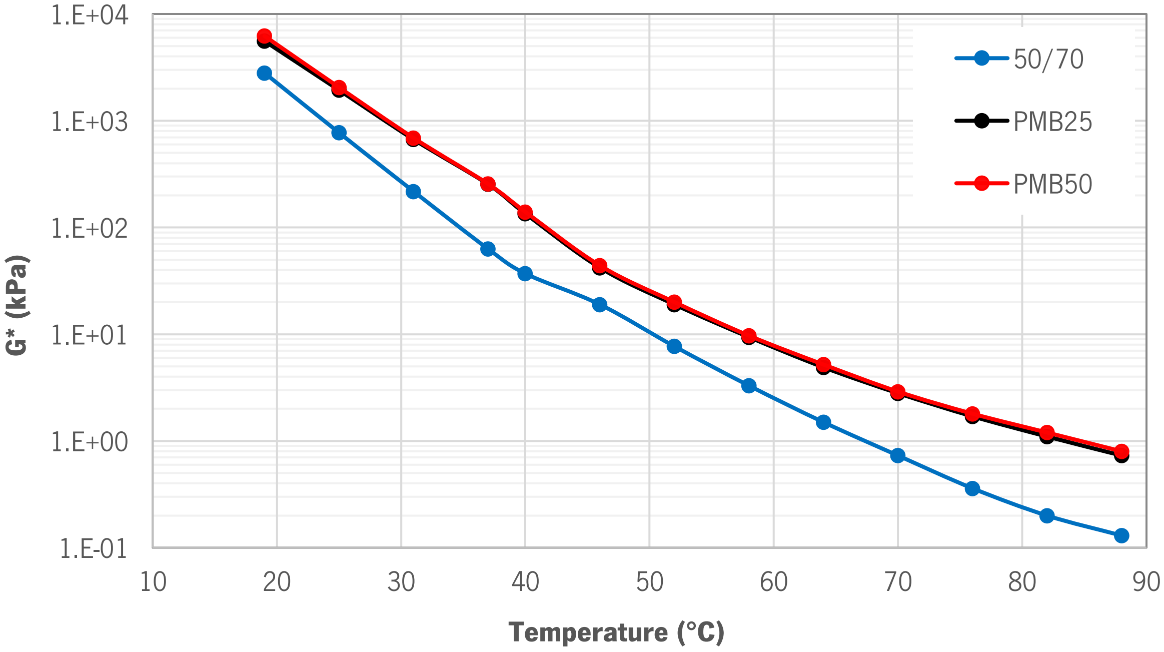


Figure 44 – Complex shear modulus G* of asphalt binders, in kPa

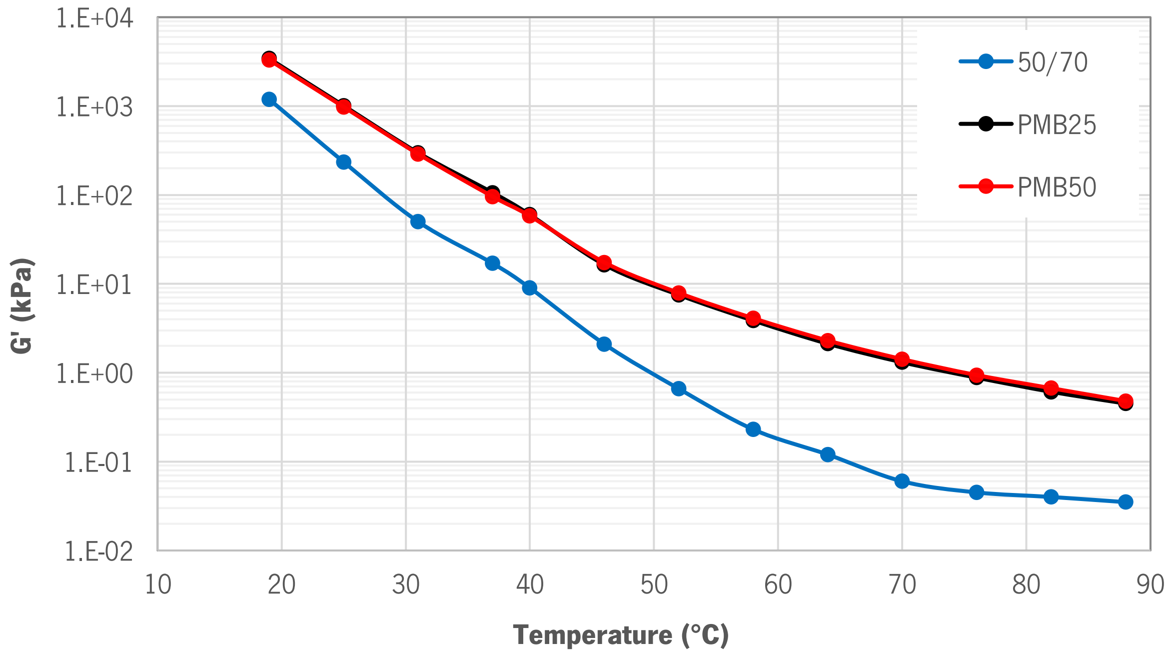


Figure 45 – Elastic modulus G’ of asphalt binders, in kPa

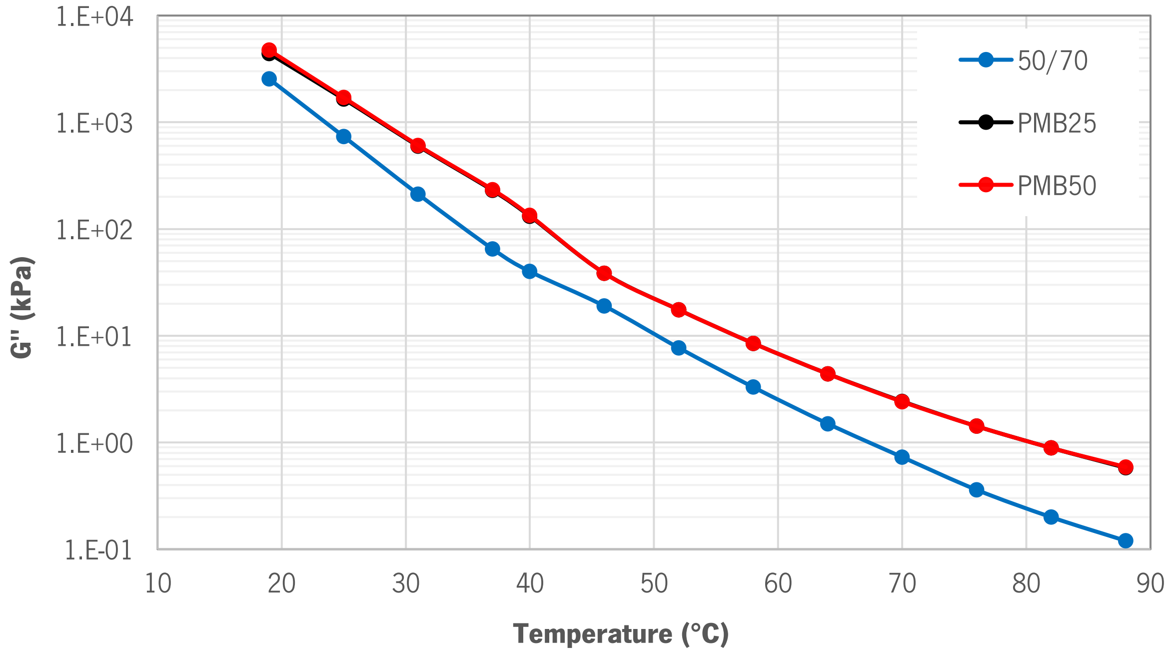


Figure 46 – Viscous modulus G'' of asphalt binders, in kPa

One of the main parameters given by the DSR test is the complex shear modulus, G*, shown in Figure 44. It can be understood as the complex sum of the elastic and viscous components of the bitumen, which are represented by the elastic modulus, G’, and the viscous modulus, G’’, shown in Figures 45 and 46, respectively.

In all evaluated bitumens, it can be observed a decrease in the elastic, G', and viscous modulus, G'', with increasing temperature, which in turn translates in a decrease in the complex shear modulus, G*. This pattern indicates the linear dependence of log G* with temperature.

The analysis of the complex modulus shows that both polymer-modified binders have identical behaviour concerning this parameter, as both curves align with each other almost perfectly. It can also be observed that the PMB present a higher stiffness over the range of analysed temperatures in comparison with the base bitumen 50/70. The differences are even higher at high temperatures, showing the excellent performance of PMBs at high temperatures. In conclusion, the SBS polymer used in the modification of both binders significantly increased the complex modulus of the base bitumen.

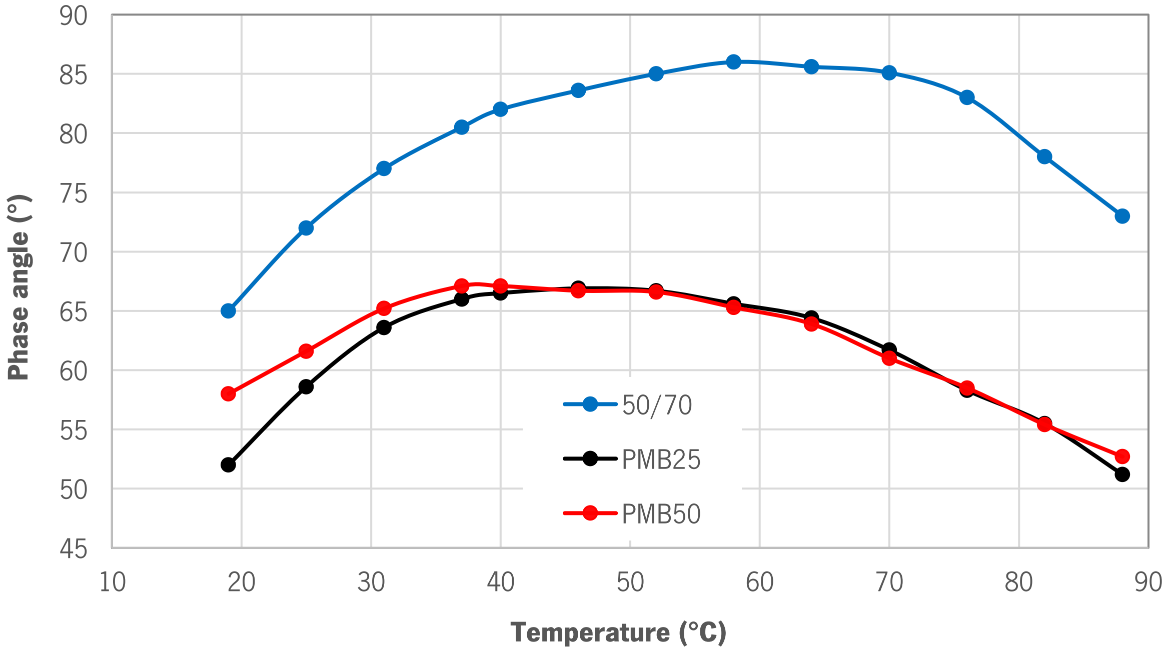


Figure 47 – Phase angle (δ) of asphalt binders, in degrees (°)

The phase angle, δ (°), provides a relative indication of the viscous and elastic behaviours of the binder, and this angle varies between 0°, for an utterly elastic material, and 90°, for an utterly viscous asphalt binder. At intermediate temperatures, such as 19 °C, asphalt binders are said to be viscoelastic with a phase angle near 45° (Asphalt Institute, 2019).

For the temperature of 20 °C, the PMB25 has the lowest value, albeit not considerably different from the other bitumens, which shows that this bitumen presents a slightly less viscous component at this temperature in comparison with the other binders. This result could indicate that PMB50 have a slightly better capacity to resist fatigue at low temperatures than PMB25, although this situation should be further evaluated because the difference between both phase angles is only 6°. All the three bitumens present phase angle curves above the 45° reference of viscoelastic material and as such can be presented as having a more viscous, rather than a more elastic behaviour.

When the temperature increases, both PMBs tend to have similar phase angle values. An increase in the phase angle values of PMBs can be seen until they reach a maximum value of 67° at the temperature of 46 °C, which indicates an increase in the viscous behaviour until that temperature. The curvature inverts after 46 °C, and both curves behave almost the same, with the viscous nature of PMBs decreasing as the temperature increases.

The base bitumen 50/70 presents an almost parallel behaviour to the PMBs, albeit with higher values of the phase angle and a maximum value of 86° obtained at higher temperatures (60 °C).

This parallelism between the base bitumen and the PMBs curves shows the contribution of the base bitumen in the PMBs behaviour. By contrast, and observing the differences between the curves, it can be seen the effect the polymer has on the base bitumen, generally lowering its viscosity and adding a more elastic component to the PMB. The smooth or undulating change in the phase angle over a broad temperature range can be primarily attributed to the addition of modifiers (Airey, 2003). This phenomenon improves the resistance of asphalt pavements to permanent deformation.

The combination of a higher complex modulus and a lower phase angle at higher temperatures of the PMB in comparison to the base bitumen 50/70 can be a good indication of the better performance of the corresponding mixtures to resist permanent deformation.

The average results of the tests performed in two samples to evaluate viscosity of both PMBs and the base bitumen 50/70 at different temperatures are illustrated in Figures 48 and 49.

According to Van Amsterdam (2000), the viscosity is a measure of a liquid flow rate and its consistency. Above 100 °C, the bitumen acts almost entirely as a viscous fluid, and so this procedure characterizes the bitumen stiffness by determining its rheological properties. The viscosity measured between 100 °C and 180 °C permits the determination of the mixing and compaction temperatures, taking into account the type of binder. The concept behind that determination is that the heated asphalt binder should be liquid enough to adequately coat the aggregates during the mixing phase and being workable enough during the compaction of the mixture.

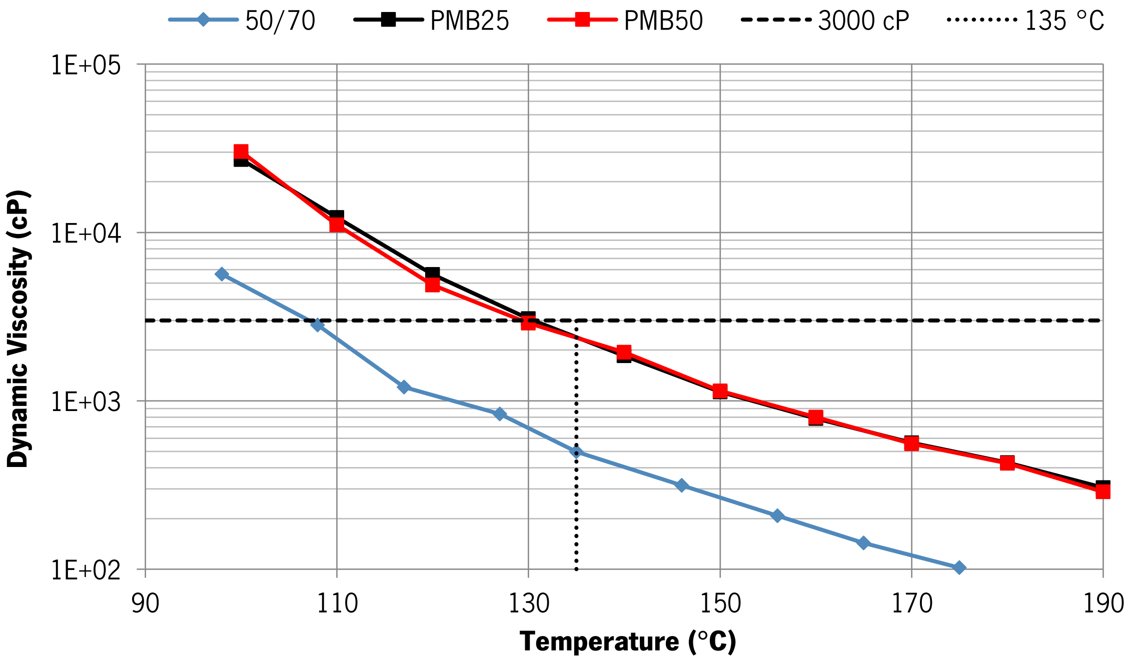


Figure 48 – Dynamic viscosity results of the binders for high temperatures, in cP

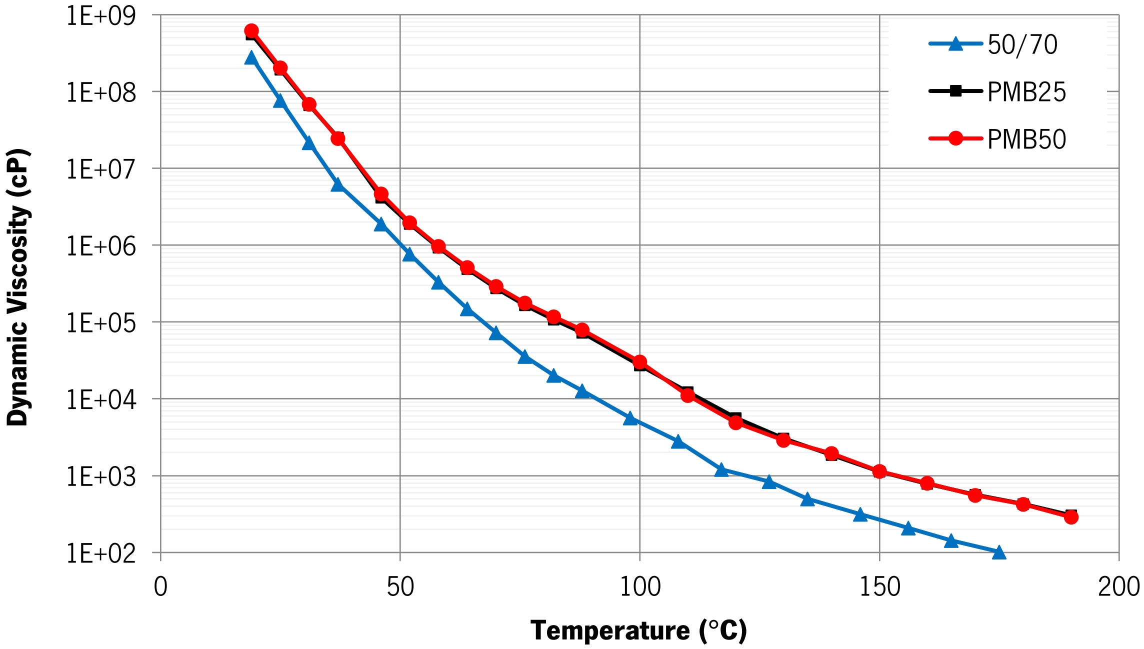


Figure 49 – Dynamic viscosity results of the binders for all temperatures, in cP

The results show the higher viscosity of the modified binders in comparison with the base bitumen 50/70 over the whole range of temperatures. Both PMB25 and PMB50 have incredibly similar values of viscosity, only with a slightly lower viscosity of PMB50 at specific temperatures. Thus, incorporating 2.5% SBS in neat bitumen changes the viscosity radically, but the addition of more SBS barely changes the viscosity.

Unmodified bitumen should have viscosities of about 170 ± 20 cP for mixing and 280 ± 30 cP for compaction (Zaniewski and Pumphrey, 2004). The viscosities of bitumen 50/70 shown in Figure 48 point out that the temperature for mixing should be no higher than 160 °C and for compaction below 145 °C.

According to Alataş and Yilmaz (2017), in order for the binders to fulfil the workability requirements, they should not exceed viscosities of 3000 cP (3 Pa.s) at 135 °C. This criterion is fulfilled as can be observed in the graph from Figure 48. Both samples of the PMB, as well as the base bitumen, verify this workability requirement, having values below the maximum 3000 cP at 135 °C.

Shenoy (2001) discusses that a preferable mixing temperature for an SBS based PMB can range from 163 to 180 °C to satisfy the following criteria: i) the temperature should be high enough to ensure the binder shows good fluidity in order to coat the aggregate during mixing uniformly and the final mix discharge does not cool to below 85 °C during laying and compaction; ii) the temperature should be low enough to ensure that the polymer in the modified binder does not degrade, and accelerated hardening of the asphalt in the modified binder does not take place due to exposure to heat and air, and; iii) the temperature should be appropriate to ensure good quality of mixing. Another recommendation made by the Asphalt Institute (2020) refers that the laboratory mixing temperatures should not exceed 177 °C. Špaček *et al.* (2017) also point out that a mixture with modified asphalt binders requires higher temperatures during compaction in comparison with a mixture with conventional bitumen 50/70. Consequently, this will result in achieving different values of bulk density and porosity. A similar analysis was performed by Toth *et al.* (2016), where a high temperature for compaction was determined according to the equiviscous principle, a principle proposed by the Japan Modified Asphalt Association. Finally, Almusawi *et al.* (2019) verify temperatures of 177 °C for compaction and 189 °C for mixing by the equiviscous method in the ASTM D2493 Standard, for a 5% SBS modified binder. Thus, the selected temperature for mixing and compaction was 170 °C taking into account all previous suggestions.

4.2.3. Elastic recovery

As previously stated, the elastic recovery test was performed for both PMB25 and PMB50 at two test temperatures, +5 °C and +20 °C. The recommended test temperatures, according to EN 13398, are typically 25 °C or 10 °C even though other temperatures can be used in order to attend to specific local conditions. In this low-temperature study of asphalt mixtures, the mechanical tests of SMA mixtures will be made at +5 °C and +20 °C temperatures, justifying the selection of the same temperatures for this test of the PMBs. The results obtained for the final elastic recovery value R_E are shown in Table 12.

Table 12 – Results of elastic recovery (RE) of PMBs

Specimen	R_E (%)			
	+5 °C		+20 °C	
	PMB25	PMB50	PMB25	PMB50
ER1	65%	52%	98%	95%
ER2	64%	51%	98%	95%
ER3	67%	52%	98%	96%
mean value	65%	51%	98%	95%
standard deviation	1%	1%	0%	0%
coefficient of variation	2%	1%	0%	0%

The minimum criterion of a predetermined elongation of 200 mm was reached at the temperature of +20 °C for both PMBs, but at the temperature of +5 °C three samples broke prematurely. The procedure detailed in the standard for brittle failures was followed in those situations. Both binders (PMB25 and PMB50) behaved remarkedly well at the test temperature of +20 °C, with almost 100% recovery, although the PMB25 presented slightly better results with a mean value of 98%, slightly higher than 95% for PMB50. Conversely, for the test temperature of +5 °C, both binders presented lower values of elastic recovery, as can be expected given the brittle behaviour and the high stiffness of asphalt binders at lower temperatures in comparison with higher ones. At this temperature, PMB25 presented, again, a higher mean value of elastic recovery, 65%, but this time with some noticeable difference to the PMB50, precisely 14% higher than the 51% mean value for PMB50.

The elongation versus force was also recorded during the elastic recovery test, and those results are shown in Figures 50 and 51.

PMB50 presents a more rigid and brittle behaviour than PMB25, presenting higher energy requirement to be elongated and breaking at lower elongation values when tested at +5 °C temperature. It is also noticeable the difference in energy requirement between both tested temperatures since the +5 °C temperature tests require over ten times the amount of energy needed to achieve the same type of behaviour when tests are performed at +20 °C temperature.

Overall, both PMBs presented an excellent elastic recovery behaviour, which may indicate a potential good cracking performance at low temperature (Zhang *et al.*, 2019).

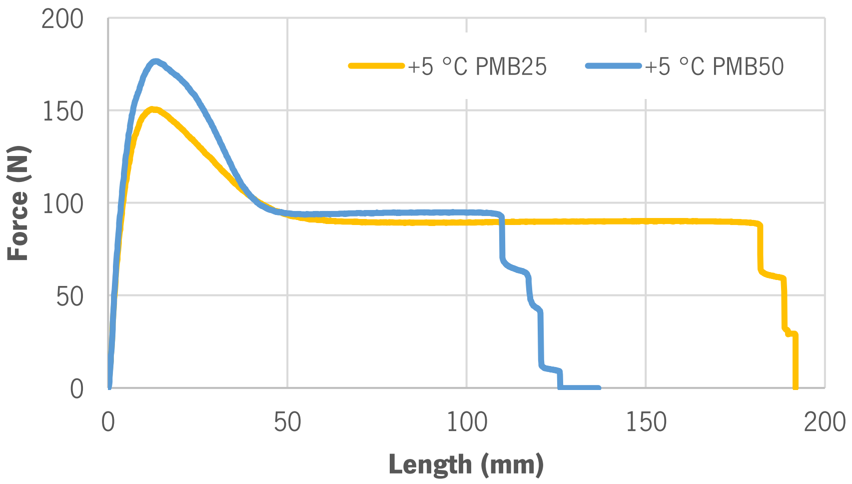


Figure 50 – Elastic recovery force of PMBs at +5 °C (mean values)

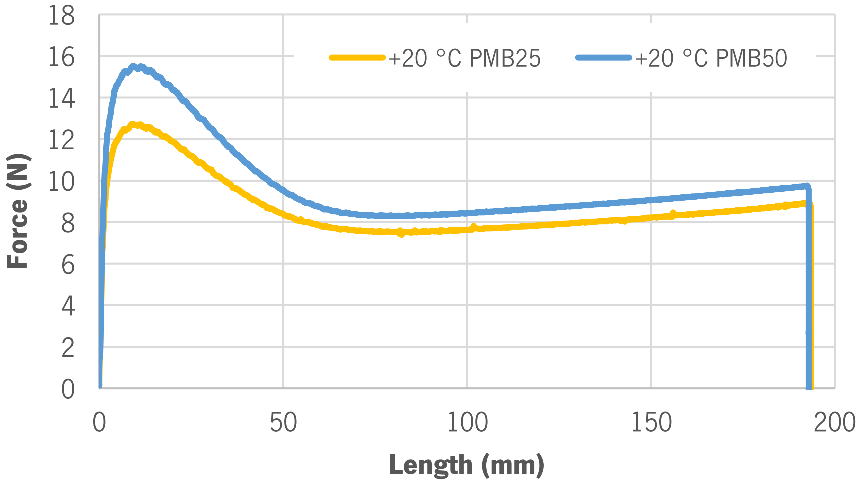


Figure 51 – Elastic recovery force of PMBs at +20 °C (mean values)

According to the European Standard EN 14023 (Bitumen and bituminous binders. Specification framework for polymer-modified bitumens), both PMBs can be included in class 2 (≥ 80%) of elastic recovery at +25 °C. Even though the tests were performed at +20 °C, it is predictable that the behaviour at +25 °C would be similar or even better.

As for the other temperature reflected in the standard (+10 °C), a linear interpolation was performed between the +5 °C and +20 °C temperatures to obtain an approximate value. The elastic recovery result was 76% for PMB25, which would predictably be in class 2 (≥ 75%) at +10 °C. PMB50 obtained a value of 66%, which would result in class 3 (≥ 50%) at +10 °C.

4.2.4. Force ductility

The force ductility method was performed at two test temperatures, +5 °C and +20 °C for both PMB25 and PMB50, as previously mentioned. The recommended test temperature, according to EN 13589, is typically +5 °C even though other temperatures can be used. Figures 52 and 53 show the resulting graphs obtained for the tested binders in the force ductility method.

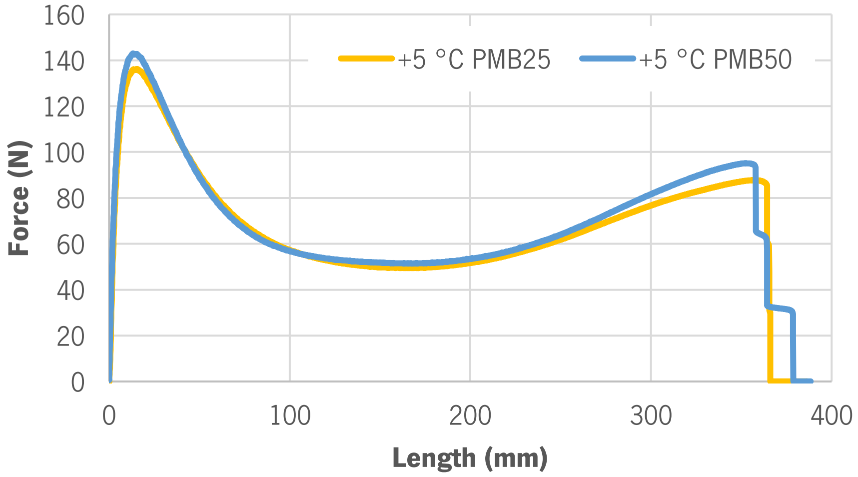


Figure 52 – Force ductility of PMBs at +5 °C (mean values)

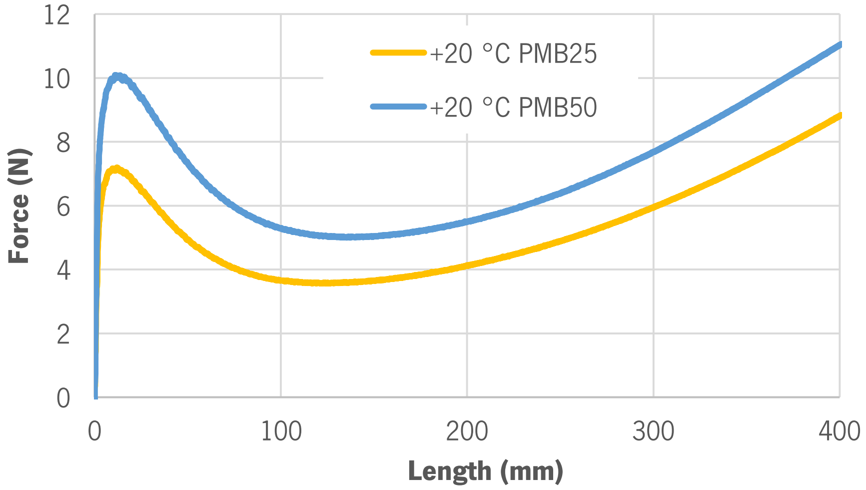


Figure 53 – Force ductility of PMBs at +20 °C (mean values)

The force ductility performance of both PMBs at the +20 °C test temperature was satisfactory, given the fact that it was possible to deform all samples up to the 400 mm established by the standard. The force required by the PMB50 to elongate was almost one and a half times the force required by the PMB25, which demonstrates the higher tensile stress limit and ductility energy retained by the PMB50 at this temperature.

Concerning the +5 °C test temperature, the one recommended by the standard, both PMB did not reach the established 400 mm elongation, having a premature break near that elongation. When comparing both PMBs, it is possible to observe in Figure 52 that they behaved very similarly, having a high level of ductility up to 350 mm elongation.

By comparing both temperatures, it is possible to observe the much higher value of energy required to stretch the samples at the +5 °C temperature than that needed at +20 °C, almost fourteen times higher. In both the tested temperatures, the shapes of the obtained curves are consistent with the increase in strength that should occur in polymer-modified bitumens after the initial peak, having an increase in strength between 200 and 400 mm, even though the +20 °C test temperature presented a much higher increase in comparison to the initial peak value. Thus, both PMBs have excellent ductility performance.

Relationships between force ductility test results and low-temperature cracking resistance of asphalt mix have been found. An increase in FD maximum force can be an indication for higher bitumen stiffness, which in turn will result in reduced low-temperature cracking resistance, indicated by higher failure temperatures (Mollenhauer and Tušar, 2016). From that study, the high FD force encountered by both PMBs can be seen as a problem for the low-temperature cracking resistance of the mixtures.

4.3. Analysis of the asphalt mixtures and specimens

As according to EN 13108-1 and EN 13108-5, the designation for these mixtures is:

- the mixture containing PMB25: SMA 11 PMB 25/55-80;
- the mixture containing PMB50: SMA 11 PMB 25/55-80.

Since the designation for both would not permit the differentiation between them, the terminology used for the PMB from the initial stages will be upheld, and the asphalt mixtures will be designated as:

- the mixture containing PMB25: SMA 11 PMB25;
- the mixture containing PMB50: SMA 11 PMB50.

Figures 54 and 55 present the obtained bulk densities for each specimen by the different procedures provided by the standard, respectively for asphalt mixtures SMA 11 PMB25 and SMA 11 PMB50.

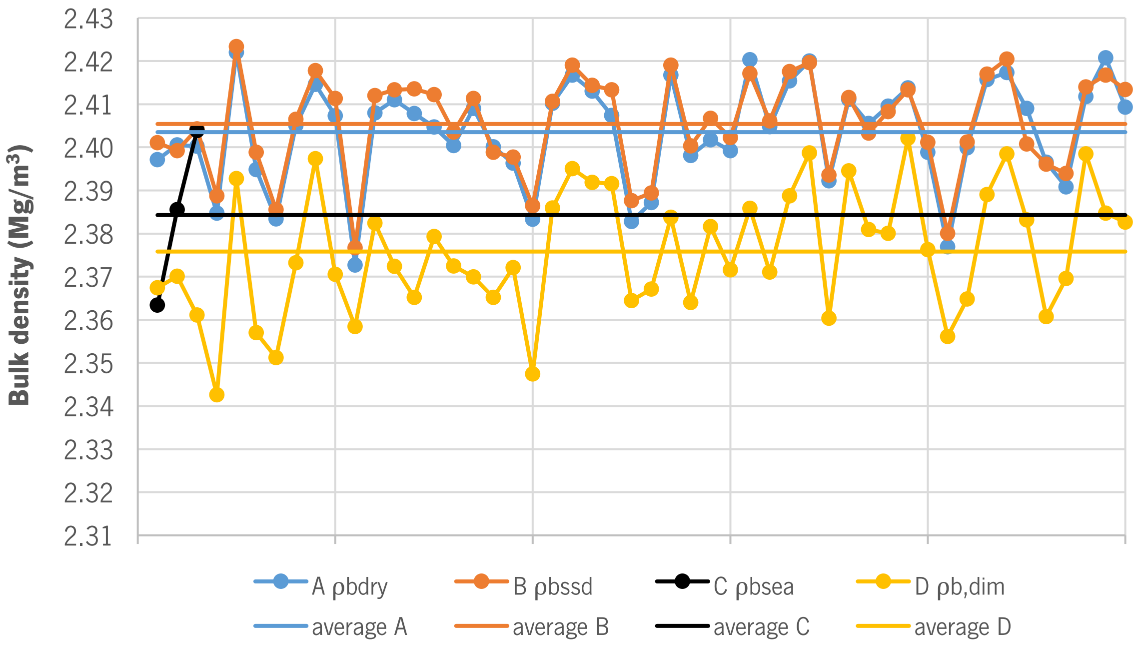


Figure 54 – Bulk densities of the SMA 11 PMB25 specimens in Mg/m³

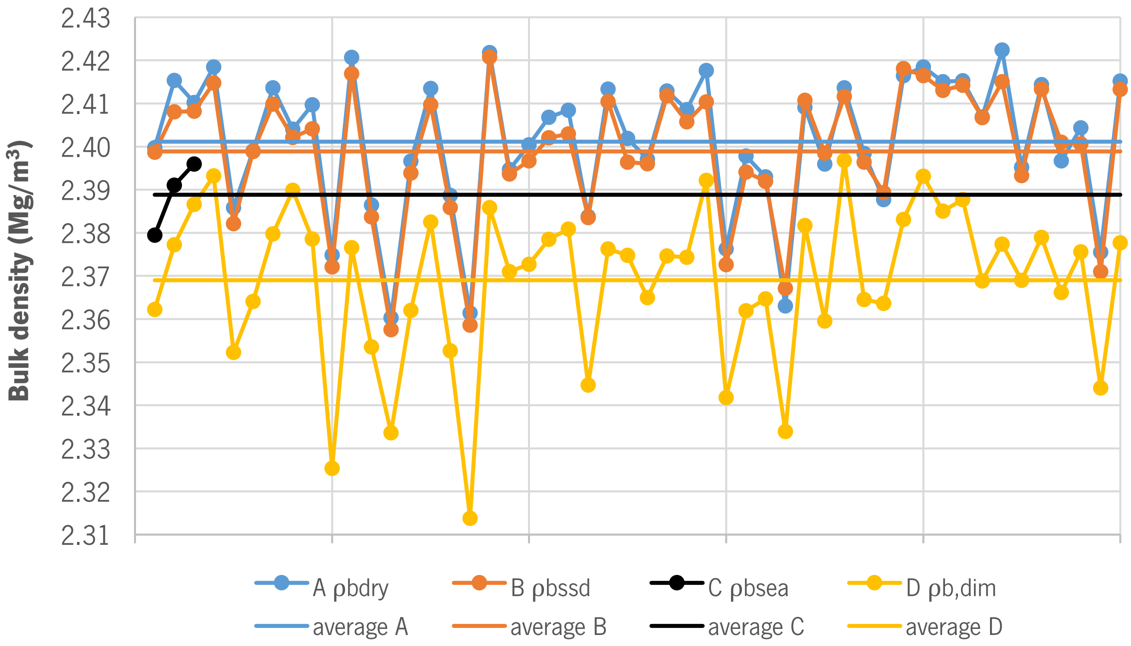


Figure 55 – Bulk densities of the SMA 11 PMB50 specimens in Mg/m³

By observing both graphs showing the bulk density obtained by the four different methods provided in the standard, it can be seen that the highest bulk density obtained for the PMB25 mixture was generally obtained by method B (with a neglectable difference to procedure A). Even though method C was used in just three specimens, it is also possible to observe that it obtained usually lower values of bulk density.

Finally, method D, based solely on the dimensions of the specimen for volumetric assessment, obtained the lowest values of bulk density.

Concerning the PMB50 mixture, the results did not vary that much from PMB25 mixture, except for method A that presented slightly higher values of bulk density.

By comparing both mixtures, it can be observed that their values of bulk density, regardless of the method used, did not present significant differences. This conclusion can also be verified by observing the difference between their mean values in Table 13.

These differences between methods will, in turn, impact the results of air voids content, since these are dependent on the obtained bulk densities. Thus, the air voids content was also determined for each method involved in the bulk density calculations.

Based on the bulk density of the several specimens of each asphalt mixtures, the air voids were also determined. The maximum density has been previously determined for the whole mixtures with PMB25 and PMB50. These characteristics are summarized in Table 13.

Table 13 – Volumetric characteristics of the studied asphalt mixtures

Characteristic	Procedure		PMB25			PMB50		
			mean value	standard deviation	coefficient of variation	mean value	standard deviation	coefficient of variation
Maximum density (Mg/m^3)	ρ_{mv}	A	2.461	-	-	2.469	-	-
Bulk density (Mg/m^3)	ρ_{bdry}	A	2.404	0.012	0.5%	2.401	0.016	0.7%
	ρ_{bssd}	B	2.405	0.011	0.5%	2.399	0.015	0.6%
	ρ_{bsea}	C	2.384	0.017	0.7%	2.389	0.007	0.3%
	$\rho_{b,dim}$	D	2.376	0.014	0.6%	2.369	0.016	0.7%
Air voids V_a (%)	A		2.35	0.48	20.3%	2.74	0.64	23.4%
	B		2.27	0.46	20.1%	2.83	0.62	22.1%
	C		3.13	0.67	21.5%	3.23	0.28	8.7%
	D		3.47	0.58	16.9%	4.03	0.73	18.1%

Concerning the maximum density obtained by method A, it can be observed that there only a slight difference between the values of each mixture. However, this small difference will impact the values of air voids content, thus indicating the need to be cautious and precise when assessing these values. The researcher must be vigilant when determining the maximum density, not only when carrying out the calculations but even more when performing the test method.

The mean values obtained for the bulk density by the different methods reflect, again, what is observed in the graphs. The coefficients of variation indicate adequate precision both in the production of asphalt mixtures and the determination of bulk densities. The obtained values were all below the 1% mark, thus indicating the estimate is very precise, and subsequently, the variability in the bulk density results is low.

The final volumetric characteristic of the mixtures involves assessing their air voids content. The air voids content of asphalt mixtures has a significant influence on the strength and deformation properties. If the air voids content is too high, the mixtures can have, generally, worsen strength properties, such as stiffness modulus and water or frost resistance. Conversely, if the air voids content is too low, this can have implications such as problems with resistance to permanent deformation and also higher temperature susceptibility (El-Badawy and Abd El-Hakim, 2018).

Brown *et al.* (1997) in their evaluation of the performance of SMA type mixtures in the United States observe that early SMA mixtures from 1991 to 1993 were typically designed to have approximately 3.5% of air voids or less. Along with this observation, they also conclude that generally, the air voids content design is somewhat lower for the Northern States of US than that for the Southern ones, which can indicate that pavements which are subjected to lower temperatures for extended periods can benefit from lower air voids content to some extent. They also observe that SMA mixtures appear to be more resistant to cracking then dense mixtures.

The specification for these SMA 11 mixtures requires a minimum air voids content of 2% and a maximum of 5%, which was observed regardless of the bulk density method used.

In the PMB25 mixture, the lowest value of air voids content is obtained by the method B, reflecting the highest bulk density observed, followed closely by method A with a negligible difference and then, somewhat distantly, by methods C and D. A similar observation can be made for the PMB50 mixture, reflecting what is observed in the bulk densities relation. The main difference when it concerns the evaluation of the PMB50 mixture is that method A provides the lowest values of air voids content, albeit not that different from method B values.

The difference between the air voids content between both mixtures is somewhat significant in all methods, except for method C. All of them indicate that PMB50 has higher air voids content. Since the bulk densities were not significantly different, it can only be concluded that the determined maximum density has a significant impact in the air voids content determination, even though the difference of these values was of only 0.008 Mg/m^3. By observing the coefficient of variation percentages, the estimate can be considered reasonable since most are near the 20% range or below.

It can be concluded that methods A and B provide the more consistent values of air voids content, and thus, researchers should opt for these methods when determining bulk densities of asphalt mixtures and subsequently their air voids content.

Taking into account the results from laboratory tests performed on the asphalt binders, as well as the ones performed on the asphalt mixtures, and given that PMB25 presented lower cost and behaviour as reasonable as PMB50, the low-temperature mechanical tests were mainly performed in the PMB25 asphalt mixture (due to time constraints). Whenever the logistics and time allowed, the respective PMB50 asphalt mixture was to be mechanically tested as well.

4.4. Mechanical performance of the asphalt mixtures

4.4.1. Uniaxial tension stress test

In this test, a specimen is pulled with a constant strain rate at a constant temperature until failure. The rate defined by the standard corresponds to a tension rate of 1 mm/min. The results obtained from each temperature and specimen are shown in Tables 14 and 15.

Table 14 – Results of tensile strength βt, in MPa, from UTST for the PMB25 mixture

Specimen	Tensile strength βt (MPa)			
	-20 °C	-10 °C	+5 °C	+20 °C
UT251	5.363	5.476	3.689	0.422
UT252	6.041	5.645	3.129	0.566
UT253	4.907	6.902	3.832	0.543
mean value	5.437	6.008	3.550	0.510
standard deviation	0.466	0.636	0.303	0.063
coefficient of variation	9%	11%	9%	12%

Table 15 – Results of tensile strength βt, in MPa, from UTST for the PMB50 mixture

Specimen	Tensile strength βt (MPa)	
	+5 °C	+20 °C
UT501	3.653	0.498
UT502	3.338	0.572
UT503	3.346	0.531
mean value	3.445	0.539
standard deviation	0.147	0.025
coefficient of variation	4%	5%

An initial analysis of the variation between specimens of each mixture presented an odd value out of three studied at each temperature. By examining the coefficient of variation (CV) values, it is possible to observe that the results obtained are valid since a range of less than 10% is considered very good, and a range of 10 to 20% is considered good. The results obtained are around 10% for the PMB25 mixture and lower than 10% for the PMB50 mixture.

In the PMB25 mixture, the results also show that the tensile strength values increased as the temperature decreased until a maximum value of around 6.0 MPa was obtained at -10 °C. Then, the tensile strength slight decrease when the temperature was reduced to -20 °C. This behaviour is consistent with that seen in the literature (Pszczola and Szydlowski, 2018) and can be possibly explained by the occurrence of low-temperature cracking when thermal tensile stresses exceed the fracture strength of asphalt pavement (Stock and Arand, 1993).

Concerning the PMB50 mixture, a similar increase in the tensile strength while the temperature is reduced can be observed, although only two temperatures have been tested.

Comparing both mixtures, at the +20 °C temperature, PMB50 presents a slightly better behaviour, but the opposite happens at the +5 °C temperature, with PMB25 presenting a higher tensile strength. It can be inferred that at lower temperatures, PMB25 mixture will present better behaviour than PMB50, but further testing is required to confirm this hypothesis. Nevertheless, the tensile strength values of both mixtures are mainly similar at both temperatures.

Concerning the failure strain for PMB25 mixture, and by observing the variation between specimens values in Table 16 and Figure 56, once more it can be seen that at each temperature, there is always an odd value out of the three studied, as can be expected since tensile strain values match the tensile strength results. By examining the coefficient of variation values, it is also possible to observe that the results obtained are valid since a range of variation lower than 10% is considered very good, a range of 10 to 20% is considered good, and a range of 20 to 30% is considered acceptable. The obtained results generally varied around 20%, except for the tests performed at -20°C that are very good (CV of 9%).

Table 16 – Values of failure strain $\varepsilon_{failure}$, in ‰, from UTST for the PMB25 mixture

Specimen	Failure strain $\varepsilon_{failure}$ (‰)			
	-20 °C	-10 °C	+5 °C	+20 °C
UT251	0.36	0.46	2.92	10.00
UT252	0.41	0.58	1.76	11.38
UT253	0.33	0.79	2.98	7.51
mean value	0.37	0.61	2.55	9.63
standard deviation	0.03	0.14	0.56	1.60
coefficient of variation	9%	22%	22%	17%

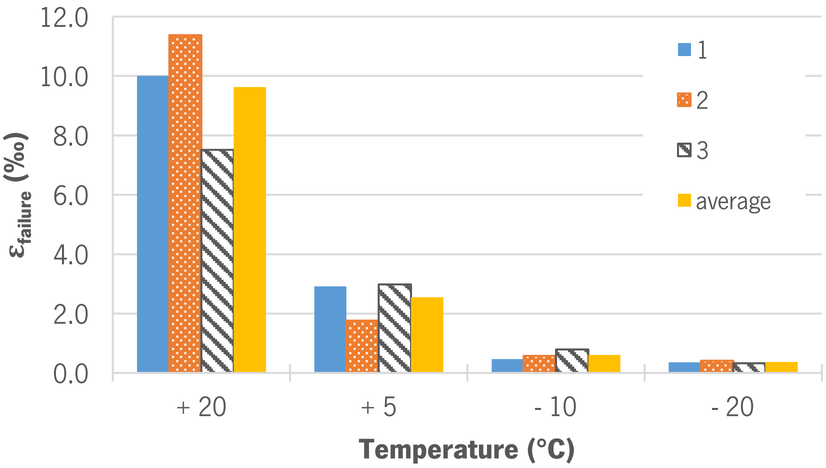


Figure 56 – Results of failure strain $\varepsilon_{failure}$, in ‰, from UTST for PMB25 mixture

The failure strain is shown to decrease with the reduction in the test temperature. The lowest studied temperature, -20°C, presents an elongation over twenty times shorter than that measured at the highest temperature of +20 °C for the PMB25 mixture. However, the difference in the failure strain between the -10 °C and -20 °C temperatures is low, and the mixture presents a brittle behaviour with very low strain failure values.

Regarding PMB50 mixture (Table 17), not much can be concluded for the elongation at +20 °C temperature due to the significant disparity of strain values obtained, which can be confirmed by the coefficient of variation of 46%. However, the strain variation at +5 °C temperature was very low (only 9%), and the PMB50 mixture presents a slightly higher failure strain than that of PMB25, although both mean values are similar.

Table 17 – Values of failure strain $\varepsilon_{failure}$, in ‰, from UTST for the PMB50 mixture

Specimen	Failure strain $\varepsilon_{failure}$ (‰)	
	+5 °C	+20 °C
UT501	3.39	2.86
UT502	2.81	10.46
UT503	2.85	6.69
mean value	3.02	6.67
standard deviation	0.26	3.10
coefficient of variation	9%	46%

4.4.2. Thermal stress restrained specimen test

As previously mentioned, the TSRST test was performed at three different cooling rates: dT = -2 °C/h, dT = -5 °C/h and dT = -10 °C/h. Figure 57 shows the results from the various tests in a graph. The tests were performed on two specimens for the dT = -2 °C/h and dT = -5 °C/h cooling rate, and on four specimens for the dT = -10 °C/h.

The testing machine CS7400S was only able to maintain a stable minimum low temperature of approximately -20 °C, which made impossible to reach the failure stress $\sigma_{cry,failure}$ and failure temperature $T_{failure}$ of the studied mixture. All the specimens behaved as expected from the test principle, although one showed considerable peaks in stress throughout the whole test (specimen A2511) at -2 °C/h cooling rate. However, the same problem did not occur in the other specimens tested at the same cooling rate. So, it is suspected that an issue either with the equipment or with the specimen resulted in this behaviour.

It can also be observed that the curve representing the cooling rates of -2 and -5 °C/h are indistinguishably the same and a lot softer than the -10 °C/h one, which is to be expected since they have a slower pace.

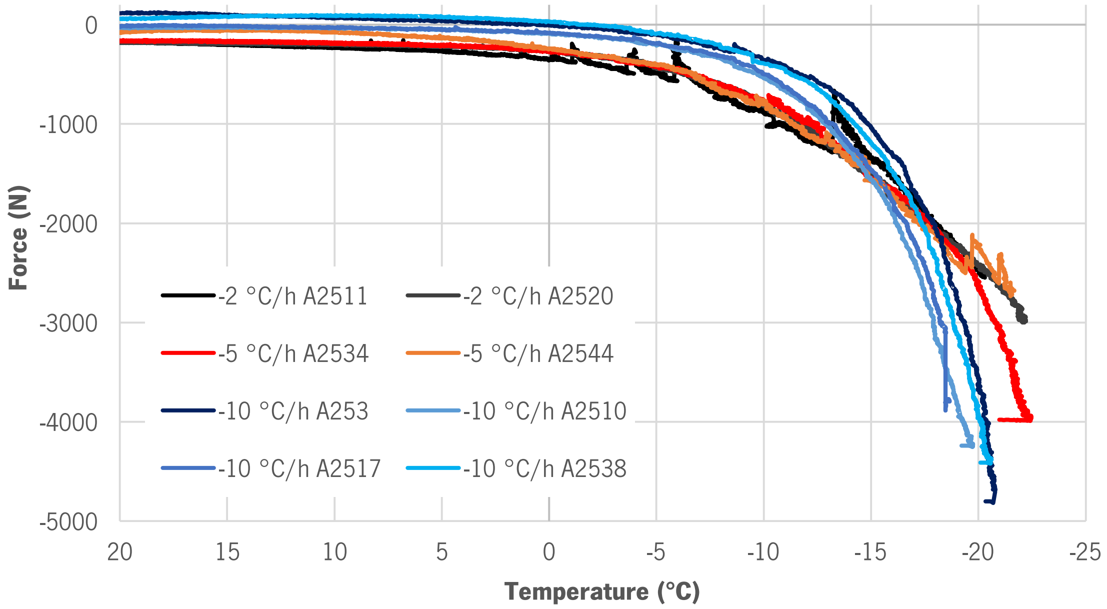


Figure 57 –Results of TSRST tests

As a comparison between the three cooling temperatures, the stress obtained once they first reached the temperature of -18 °C was registered and is presented in Table 18.

Table 18 – Stress (N) at -18 °C for the three cooling rates

Specimen	Cooling rate (°C/h)	Stress (N)			
		value	mean value	standard deviation	coefficient of variation
A2511	-2	1838	1941	103	5%
A2520		2044			
A2534	-5	1890	1958	68	3%
A2544		2027			
A253	-10	2171	2621	368	14%
A2510		3139			
A2517		2773			
A2538		2402			

The maximum stress (peak stress) obtained for the three cooling rates was also registered, along with the temperature at which that value occurred. Table 19 presents those results.

Table 19 – Peak stress (N) for the three cooling rates

Specimen	Cooling rate (°C/h)	Peak stress (N)				Temperatures at which it was obtained (°C)
		value	mean value	standard deviation	coefficient of variation	
A2511	-2	2376	2671	295	11%	-20.3
A2520		2966				-22.1
A2534	-5	3823	3236	588	18%	-22.4
A2544		2648				-21.5
A253	-10	4926	4370	387	9%	-20.7
A2510		4220				-19.7
A2517		3862				-18.5
A2538		4472				-20.6

By observing Table 18, it can be verified that the cooling rates of -2 and -5 °C/h present similar stresses at -18 °C, which is somewhat visible in Figure 57. However, once the peak values are analysed, a significant difference was noticed in Table 19. Thus, both cooling rates subject the pavement to very similar stresses right up until very low temperatures are reached. At these extremely low temperatures, a faster cooling rate causes higher stresses to the mixture, which in turn can result in cracking.

Regarding the cooling rate suggested by the standard (-10 °C/h), it can be observed that it subjects the mixture to significantly higher stresses than the slower cooling rates, both at -18 °C as well as at higher test temperatures. The same behaviour is observed for very low temperatures, around -20 °C, at which the mixture presents very high stresses in comparison with just 2 °C warmer test temperature. In summary, the -10 °C/h cooling rate most likely will result in the mixture failure at higher stress levels.

By comparing the three cooling rates, the effect of that parameter on the resulting values is evident. The faster the cooling rate, the higher the thermal stresses the mixture will present. This aspect is vital in regards to the correct definition of the cooling rate in the design stage, as to accurately reflect the actual conditions observed in the pavement. Certain regions can have more propensity to faster cooling for different reasons, and also this rapid cooling can occur more often. Thus, the climate of the region where the pavement will be executed should be carefully studied in order to design the asphalt mixture properly.

The validity of the data can be ascertained by observing the coefficients of variation. They all present values lower than 20%, which indicates the data values are reliable.

In conclusion, even though it was not possible to obtain the failure temperature of the mixture, those temperatures are lower than -20 °C, and it is not predictable that those temperatures will be observed in Portugal, given the local climate. Thus, the laboratory tests with the testing machine used will be sufficient to validate the low-temperature performance of this mixture in Portugal.

However, in order for Portuguese companies to be able to compete in the international market, particularly in Northern and Eastern countries, it would be of interest that this research work can be deepened in order to obtain the failure temperature and correspondent stress since lower failure temperatures from TSRST indicate that low-temperature cracking should be reduced (Nicholls, 2017).

4.4.3. Relaxation test

The initial input values for the relaxation test are shown in Table 20. The initial imposed stress derives from the UTST and should not be greater than 75% of the obtained tensile strength βt. The relaxation time is the time when the stress is reduced to 36.8 ± 0.1% (1/e = 1/2.718 = 0.368) of its initial value. Relaxation modulus is a main viscoelastic parameter of an asphalt mixture and is used as an essential basis for evaluating and analysing the performance and predicting the long-term stability of a pavement (Sun *et al.*, 2018).

Table 20 – Input values for the RT

Parameter	-20 °C	-10 °C	+5 °C	+20 °C
75% Peak stress from UTST (N)	-6243.1	-7038.2	-4281.9	-651.5
Corresponding input strain (mm)	0.042	0.067	0.130	0.580

These values reflect the expected stress level is the pavement. The difference in stress levels between +20 °C and +5 °C is considerable, and the mixtures are expected to be subjected to high strain levels. Between -10 °C and -20 °C even though the stress from UTST was less on the lower temperature, the measured strain did not correspond totally to the expected behaviour, having a lower relaxation value than that observed at -10 °C temperature. The summary of the results obtained from the RT tests is shown in Table 21.

All specimens behaved as predicted, as can be observed in the evolution of stress versus time shown in Figures 58 to 60.

Table 21 – Relaxation test results

Parameter	-20 °C		-10 °C	+5 °C		+20 °C
	RT1	RT2	RT3	RT4	RT5	RT6
Relaxation stress (N)	did not reach	did not reach	-2492.0	-1973.6	-1598.3	-749.9
Relaxation time t_{rel} (s)	did not reach	did not reach	2206	45	49	33
Relaxation time t_{rel} (hh:mm:ss)	did not reach	did not reach	00:36:46	00:00:45	00:00:49	00:00:33
Test stopping time (s)	172991	172991	-	-	-	-
Test stopping time (hh:mm:ss)	48:03:11	48:03:11	-	-	-	-
Stress at end of test (N)	-2778.7	-2420.5	-	-	-	-

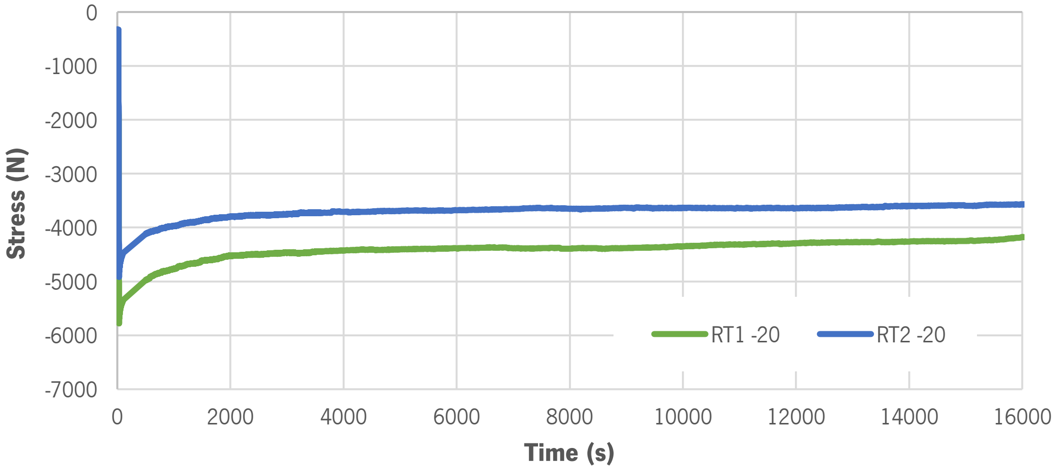


Figure 58 – Evolution of stress (N) with time (s) of both specimens tested at -20 °C

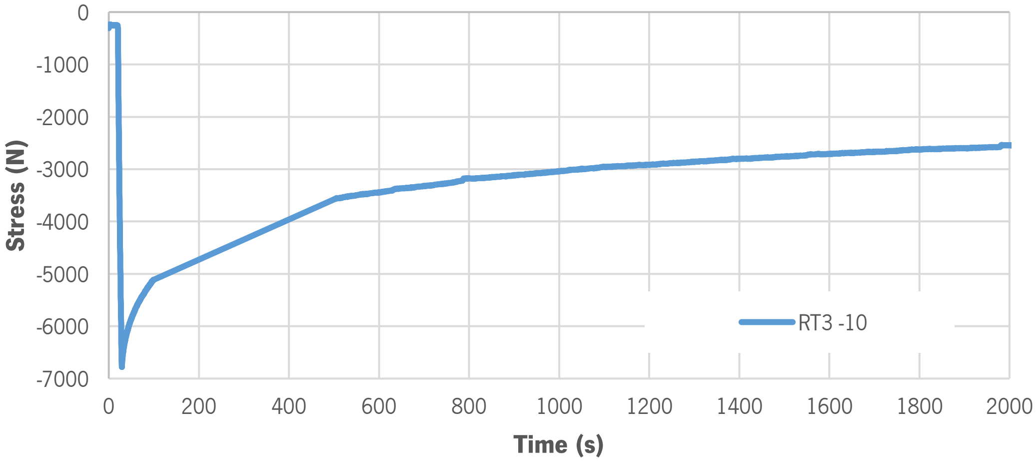


Figure 59 – Evolution of stress (N) with time (s) of the specimen tested at -10 °C

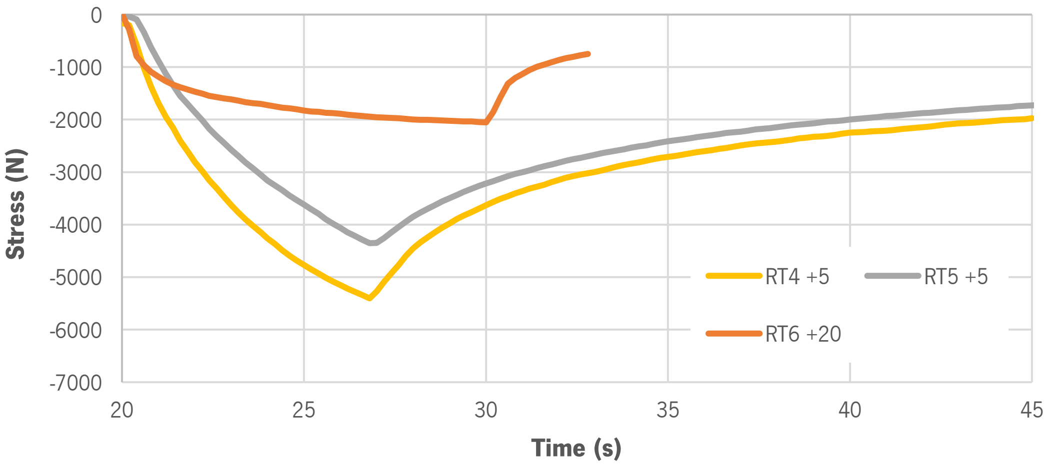


Figure 60 – Evolution of stress (N) with time (s) of the specimen tested at +5 °C and +20 °C

It can be observed that for higher temperatures, the asphalt mixture reached the relaxation time very quickly, under a minute. This result is consistent with the observed experience in the literature that the rheological properties of the asphalt mixtures are affected by temperature, vehicle load and vehicle speed. Once pavements are subjected to stress, it is gradually dissipated over time, and relaxation occurs. When the temperature is high, the stress accumulated in the pavement will dissipate quickly because of a stronger relaxation ability, which is verified in this study.

For the -10 °C temperature, the relaxation time was reached after approximately 36 minutes. However, for the -20 °C temperature, both specimens were not able to reach the relaxation time in the 48 hours proposed by the standard. This test should be concluded when stress level falls below 36% of its initial value (2125.9 for RT1; 1809.4 for RT2). The final stress level for RT1 was 2778.7 N, while for RT2 was 2420.5 N, and both are near the pre-established value of 36%. Nevertheless, by observing their curves in the stress versus time graph in Figure 58, it is predictable that they would take a long time to reach the relaxation time since both curves present a steady but slow behaviour.

According to Sun *et al.* (2018), when the temperature is low, the stress in the pavement dissipates slowly because of poor relaxation ability, which is confirmed by this study. When this occurs, cracks in the pavement may appear, a product of the final stress being more significant than the ultimate strength of the mixture.

Overall, this mixture presents a remarkable behaviour at low and medium temperatures when it relates to relaxation. Some slow relaxation issues of the mixture may only occur at very low temperatures (-20 °C) if the pavement is subjected to these temperatures for an extended period.

4.4.4. Tensile creep test

A creep test (sometimes referred to as a stress-relaxation test) is used to determine the amount of deformation a material experiences over time while under a continuous tensile load at a constant temperature. The results from the tensile creep tests at different temperatures are shown in Figure 61.

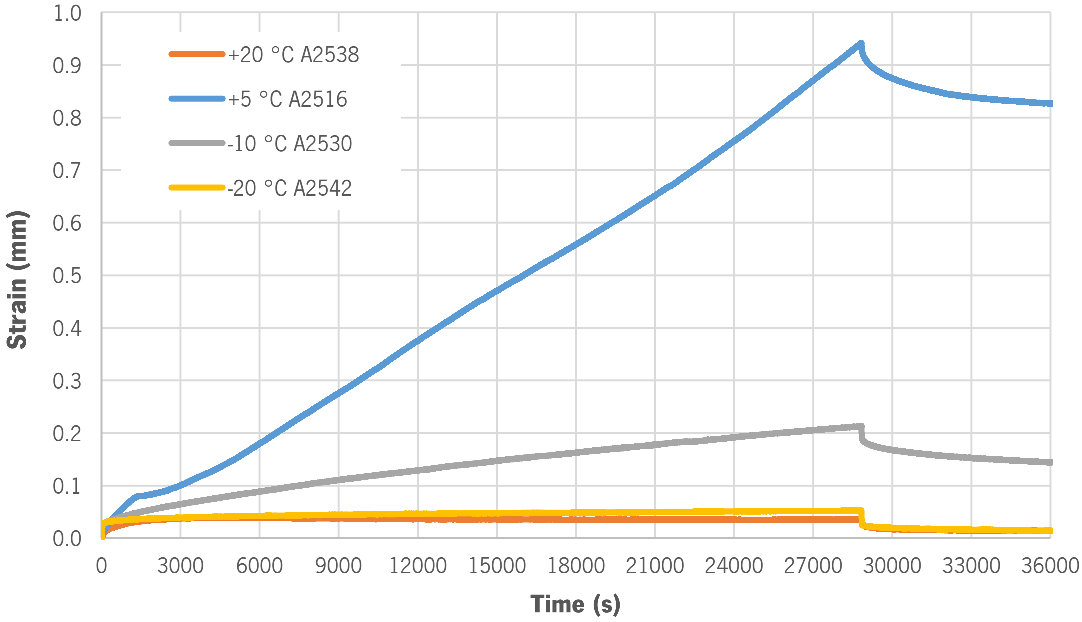


Figure 61 – Results of TCT tests

As can be observed from the graph in Figure 61, both the limit temperatures of -20 °C and +20 °C demonstrate a similar behaviour in this test, with almost corresponding values of strain evolution with time. They both recovered almost the whole deformation achieved previously. It is essential to understand that this is only conceivable because the imposed stress level, defined in the standard, is much lower at higher temperatures.

The temperature of -10 °C presented a higher deformation value than that observed in the previous two temperatures and recovered part of its maximum deformation. Its peak of deformation was obtained at 0.208 mm and recovered 0.064 mm, which presents an approximate recovery of 30% of the initial deformation. The stress level imposed in the standard at this temperature originated a worse behaviour of the mixture at this temperature.

Nevertheless, the worst performance was observed at the temperature of +5 °C, with a considerable deformation under the standard imposed stress level, and only recovering a tiny proportion of that deformation. The mixture deformed 0.939 mm and recovered 0.112 mm, which results in a recovery of only 12% of the initial deformation.

These results indicate that at high temperatures, as well as very low temperatures, this asphalt mixture most likely will not present deformation issues. The same cannot be said for the intermediate temperatures of +5 °C and -10 °C, particularly at +5 °C, considering the stress values imposed in the standard. In this temperature range, the mixture only recovered 12% of the initial deformation, which can indicate that deformation can occur during the lifetime of the pavement, depending on the conditions it would be subjected. This test simulates the effects caused in the pavement when a load is applied by vehicles or thermal stresses and then is removed when those conditions change. If the asphalt mixture is not able to recover from the load promptly, that will result in deformation damage to the pavement as more loads are applied. These effects of transitory loading and unloading of asphalt mixtures cause deformations which can, over time, result in rutting or cracking of the pavement (Hischke, 2019).

Further analysis in order to fit a creep model to the observed characteristics would be of interest. SABITA (2015) indicates that an apparent correlation exists between elastic recovery and deformation resistance. Those authors mentioned that an increase in elastic recovery provides better performance for deformation, as well as the increase of the cohesive strength.

A model describing the viscoelastic properties derived from the results in this test would also be of interest. Many models have been studied to describe these properties, such as the Burgers model, Huet–Sayegh model to describe the viscoelastic component, Perzyna-type model to describe the viscoplastic component, among many others. A comparison between those different models can further augment the knowledge in this field, in order to show their differences and determine their accuracy. For example, Hornych *et al.* (2012) observed that the viscoelastic strains calculated with the model were more extensive than the experimental viscoelastic strains obtained during the unloading phase of the creep test. Pszczoła and Judycki (2009) also observed that the elastic modulus and viscosity coefficient increased with the decrease in temperature, which indicates an increased stiffness of the studied mixtures.

4.4.5. Uniaxial cyclic tensile stress test

Table 22 below presents the input values for the base stress $F_{cry}(T)$, the stress caused by the traffic load ΔF, as well as the peak stress F_{tot}. These values are intended to simulate the dynamic loading condition caused by traffic, which is given by the traffic load ΔF in combination with constant stress, given by the base stress $F_{cry}(T)$, which symbolizes the expected cryogenic (thermal) stress in the pavement. This cryogenic component is derived from the TSRST test, in which the stress at the UCTST test temperature is obtained from the curve stress vs temperature. The sum of both these stresses results in the maximum stress the specimen is subjected to, the peak stress F_{tot}. These values intend to reflect the tension/compression load patterns observed at the bottom of the asphalt pavement layer where cyclic loading is applied in terms of repetitive load cycles comprising tensile and compressive loading (Mollenhauer and Wistuba, 2012).

Table 22 – Input values for UCTST

Parameter	-20 °C	-10 °C	+5 °C	+20 °C
Base stress $F_{cry}(T)$ (N)	3910	473	37	4
Traffic load ΔF (N)	2560	2560	2560	2560
Peak stress F_{tot} (N)	6470	3033	2597	2564

Another consideration is the test frequency. As previously stated, a value of 10 Hz was selected in this work. The test frequency is meant to simulate the actual loading rate the pavement is subjected to due to the various traffic speeds. Saal and Pell (1960) analysed the influence the test frequency can have in cyclic controlled tests, and they concluded it could be considerable since the fatigue life of an asphalt mixture at a frequency of 13 Hz was found to be significantly lower than for a frequency of 50 Hz. Thus, it may be of interest to continue the research carried out in this work with different test frequencies in the future, after adjusting the testing device to perform additional tests.

The input values used in the test reflect the stresses caused in the pavement due to the decrease in temperature. As can be observed, the difference in cryogenic stress between +20 °C and +5 °C is negligible, but it is increased when the temperature decreases to -10 °C. Between -10 °C and -20 °C, a jump in cryogenic stress can be observed, which may indicate that the pavement with this mixture will not suffer significant thermal stresses due to low temperature until it reaches very low temperatures (near -20 °C). The summary of the results obtained from the UCTST tests is shown in Table 23.

A criterion of a maximum of 3.0 million cycles was established for the specimen to reach failure. This criterion was based on a restriction of time for the laboratory tests to be performed. It results in continuous testing for about three and a half days. This issue, along with other constraints, limited the number of specimens that could be tested in this work.

Table 23 – Results of the UCTST

Temperature (°C)	-20 °C	-10 °C	-10 °C	+5 °C	+5 °C	+20 °C
Specimen	UC1	UC1	UC2	UC1	UC2	UC1
Type of failure	did not reach failure	did not reach failure	did not reach failure	fracture	fracture	fracture
Number of load applications $N_{failure}$	1.5 million cycles	3.0 million cycles	2.4 million cycles	10 000 cycles	15 000 cycles	451 cycles

Fatigue behaviour can be characterised by relating the strain of a mixture to the number of load applications to failure (Hassan *et al.*, 2014).

The specimens behaved accordingly to the expected test principle. An example of their behaviour is shown in Figure 62. The strain shows the deformation of the specimen, and the stress shows the applied load.

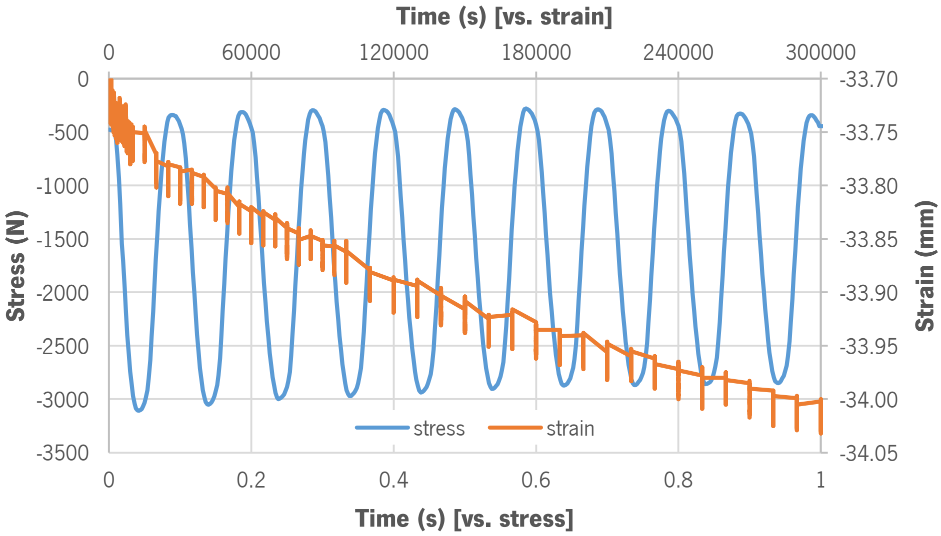


Figure 62 – Behaviour of test specimen at -10 °C during the initial cycles of UCTST

The three specimens tested at higher test temperatures (+20 °C and +5 °C) reached failure. The test at +20 °C took only 451 cycles to reach failure, which corresponds to an elongation of 3.5 mm before it breaks. Both specimens at +5 °C temperature reached failure, but due to a problem in the recording equipment, it was only able to record 10 000 cycles. The behaviour of this specimen showed an almost linear progressive curve regarding the strains recorded by the three LVDT. The other specimen reached failure presumably at 15 000 cycles, although the specimen showed an unusual behaviour concerning the strains recorded (Figure 63). It can be seen that the three LVDTs reached the maximum range of deformation, which is approximately 5 mm.

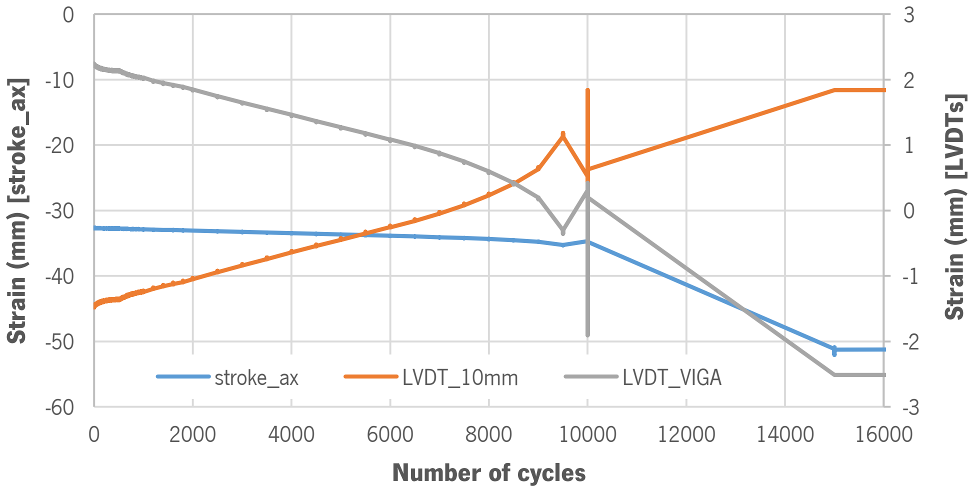


Figure 63 – Unusual behaviour of specimen 2 at +5 °C in UCTST

The rest of the specimens were tested at -10 °C and -20 °C temperatures without reaching failure. Specimen 1 of -10 °C temperature reached the pre-established 3.0 million cycles criterion, obtaining an elongation of 0.259 mm at the end of the test, while specimen 2 reached 2.4 million cycles. Since specimen 1 was already tested, it was decided to end the test. At 2.4 million cycles, specimen 2 reached an elongation of 0.275 mm, slightly higher than that of specimen 1. Both these specimens presented a linear progressive strain behaviour.

Finally, the specimen tested at -20 °C was tested until 1.5 million cycles. At this point, it presented an elongation of only 0.054 mm. Its strain behaviour was slow and progressed linearly. Based on the previous tests at -10 °C and their respective elongations, it was decided to stop the test at half of the pre-established criterion of the number of cycles since the progress of strain was very slow and presumably, the specimen would not reach failure at 3.0 million cycles.

Mollenhauer and Wistuba (2012) also reflect on the appearance of the surfaces of the broken specimens halves. These depend on the mixture properties and test conditions, particularly on test temperature. The fracture may be observed to run through the binder mastic, which can indicate failure by cohesion, through the binder-aggregate interface, which can indicate failure by adhesion, or directly through the aggregate, which can indicate the overstepping of the strength provided by the aggregate. The latter is mainly observed at low test temperatures. Since the specimens at low temperatures did not reach failure, it was not possible to visually verify the type of failure. Figure 64 presents a comparison of the specimens after the break for the higher test temperatures that reached failure.

Figure 64 – (a) Specimen failure at +20 °C; (b) Specimen failure at +5 °C

In Figure 64, it is possible to observe that the failure area through the aggregates increased when the temperature decreased from +20 °C to +5 °C. This result is consistent with the literature review and demonstrates the excellent cohesion of the asphalt mastic with a polymer-modified binder.

The observed behaviour of the specimens at low temperatures and the fact they did not reach failure under extensive quantities of cycles can indicate this mixture will have excellent properties to resist fatigue at low temperatures.

5. CONCLUSIONS AND FUTURE WORK

"We make our world significant by the courage of our questions and by the depth of our answers."

Carl Sagan

5.1. Conclusions

The research investigation described in this dissertation focused on the evaluation of the influence of the modification of asphalt mixtures in their behaviour at low temperatures. A conventional binder of 50/70 penetration grade was used as a base and modified with two percentages of SBS polymer, 2.5% and 5.0%. The three binders were then characterized regarding their softening point, penetration, viscosity, resilience and other rheological properties, elastic recovery and force ductility. Two SMA 11 asphalt mixtures were then produced with both of the polymer-modified bitumens, which were cut into specimens. These asphalt mixtures were then characterized and mechanically tested. The mechanical tests were based in the European Standard 12697-46, including the UTST, TSRST, RT, TCT and UCTST tests.

Concerning the objective of evaluating the types of binders, it can be stated that these objectives were successfully reached. The penetration values of both PMBs (with 2.5% and 5.0% SBS) were similar and lower than that of base bitumen, which indicates the higher viscosity of the PMBs. The softening point presents a much higher value for both PMB in comparison to the base bitumen, which indicates that these PMB are less susceptible to temperature and substantially more stable to permanent deformation.

The rheological characteristics present almost the same behaviour for both PMBs, which indicates that the difference in the percentage of polymer in these two binders almost does not impact their rheological behaviour at medium to high temperatures. Another sign of the excellent permanent deformation performance of these two PMBs is presented from a combination of a higher complex modulus and a lower phase angle at high temperatures in comparison to those of base bitumen 50/70.

In the elastic recovery and force ductility tests, these two PMBs revealed slight differences in their performance. PMB25 showed a slightly better recovery performance than PMB50 at +20 °C. At lower temperatures (+5 °C), PMB25 presented a more significant difference from PMB50. PMB50 showed a more rigid behaviour at both temperatures, which is evident by the higher force required to stretch the samples. Concerning the force ductility tests, both PMBs presented a very similar behaviour at +5 °C, even though they could not reach the total elongation imposed in the standard before breaking. Nevertheless, they did present high ductility in this test, which can be a good sign of a desirable performance concerning low-temperature cracking resistance.

Regarding the maximum density, bulk density and air voids content of the studied mixtures, they presented a low value of air voids, although within limits established by the specification for these SMA 11 mixtures. By comparing the different methods proposed in the standard, it was possible to observe the influence they can have on the final air voids content estimate. Generally, methods A and B presented the highest bulk densities with not much difference between them, which in turn leads to the lowest air voids contents. The lowest bulk densities were given by method D, with the highest air voids content.

The air voids content is particularly relevant when designing and characterizing an asphalt mixture. Too low air voids content can lead to problems of higher susceptibility to rutting. The determination of the maximum density is also worth noticing. Even though the maximum densities of both mixtures were almost the same and the bulk densities did not differ that much between them, the effect the maximum density had in the final air voids content calculation was considerable. Thus, the final values of air voids content in PMB50 was considerably higher than those found in PMB25 mixture, even though they presented similar values of bulk densities.

Because of time constraints, it was decided to proceed with the low-temperature mechanical tests with one of the mixtures, testing only six specimens of the other mixture. The PMB25 mixture was the selection made. Since both PMBs presented an identical performance in many tests, the selection of the asphalt mixture was mostly based on its final cost and elastic recovery at +5 °C. It is believed that elastic recovery can reduce bitumen pavement's low temperature cracking (Zhang *et al.*, 2019).

The UTST test on PMB25 mixture presented a behaviour consistent with that observed in the literature: as the temperature decreases, the tensile strength values increase until they reached their peak value, and then they start to decrease again. This result can be explained by the occurrence of low-temperature cracking when thermal tensile stresses exceed the fracture strength of an asphalt mixture. Six specimens of PMB50 mixture were tested at higher temperatures, and even though they presented a slightly higher tensile strength at +20 °C, their tensile strength at +5 °C was lower than that of PMB25 mixture. However, the tensile strength of both mixtures was generally similar. Moreover, it was concluded that the elastic recovery at low temperatures could be a suitable parameter for an initial evaluation of binders designed for asphalt mixtures subjected to low temperatures.

In the TSRST test, even though it was not possible to reach the failure temperature and stress, due to equipment restrictions, other conclusions were ascertained, mainly related to the influence of the cooling rate on the test results. Slower cooling rates present much lower stress values. The cooling rate indicated by the standard shows the most conservative values, with higher stresses, thus justifying its recommendation. This option can act both as a benefit, by ensuring the asphalt mixture can withstand higher thermal stresses, and as a hindrance, because it may lead to overdesigning the mixture with unnecessary costs. Another note of worth is the change in measured stresses at low temperatures: at around -20 °C the difference in stresses regarding the cooling rates was much more significant than at only 2 °C warmer. At these extremely low temperatures, a faster cooling rate causes much higher thermal stresses to the mixture, which in turn can result in thermal cracking.

The RT test main objective is to simulate the stress dissipation of an asphalt mixture in the pavement over time. It was observed that for the higher temperatures, the asphalt mixture showed an excellent performance since it reached the relaxation time very quickly. This behaviour at high temperatures is consistent with what is observed in the literature since when the temperature is high, the stress accumulated in the pavement will dissipate quickly because of a stronger relaxation ability. At a low temperature of -10 °C, the mixture continued to exhibit a suitable performance, dissipating the imposed stress in approximately 30 minutes. Some issues in the mixture may only be inferred at very low temperatures (-20 °C) since the mixture was not able to dissipate the applied stress in 48 hours, although it dissipated a significant portion of that stress. This result can indicate that cracking may occur if the pavement is subjected to these very low temperatures for an extended period. Despite this, it can be inferred that thermal cracking problems may not be severe because the mixture can dissipate most of the applied stress.

The TCT test presented a very particular behaviour, because the asphalt mixture performed almost the same at the highest (+20 °C) and the lowest (-20 °C) temperatures, recovering a significant amount of the imposed strain. At -10 °C, the mixture recovered approximately 30% of the initial deformation but recovered only 12% at +5 °C. This test indicates that this asphalt mixture most likely will not present deformation issues at high or very low temperatures.

The last mechanical test, UCTST, intends to evaluate the mixture with regards to fatigue, by subjecting it to repeated cycles of loading and unloading designed to simulate both the thermal stress and traffic loads applied in the pavement. At the higher temperatures, +20 °C and +5 °C, the specimens cracked, which can indicate that the mixture may not present a desirable performance at those higher temperatures.

Conversely, at lower temperatures, the asphalt mixture behaved remarkably well since it did not reach failure at the pre-established criterion of 3.0 million cycles which can indicate that it may not suffer the effects of fatigue cracking under the standard test conditions.

Cracking is one of the main issues that can occur at low temperatures. This problem arises from the tensile stresses developed in the pavement as the temperature falls. Since the asphalt is, to a greater or lesser degree, restrained, and if those stresses reach a critical level, cracks may form. These cryogenic cracks can be initiated by one particularly low temperature or by multiple warming and cooling cycles, with the latter being exacerbated by fatigue from traffic loadings (Nicholls, 2017).

Overall, it is considered that the mixtures produced and evaluated in this research work performed very satisfactorily and that the knowledge regarding low-temperature laboratory tests and their derived conclusions will deepen the understanding of these conditions and their effect and influence on pavements durability.

5.2. Limitations and future work

Concerning the bonding agent, some considerable challenges could be placed. These pertain mostly to its adhesiveness to the metal plates, because it adheres well to the specimens. In future studies, it is proposed to analyse the stress and strain this bonding agent can withstand while under low-temperature conditions. It is also proposed to test the bonding agent by performing a pull-out test or by other means to analyse the adhesive properties of the bonding agent in detail.

Some limitations arising from this study are the extensive amount of testing time and respective pretesting preparation, such as the curing time of the bonding agent, the conditioning of testing temperatures in the chamber and, consequently, the temperature stabilization in the specimen. Some other issues also appear concerning the load cell of the equipment, which further delayed the tests. All these problems were a severe difficulty to test the PMB50 mixture properly. Thus, it is proposed that these tests will be performed in the future to compare both mixtures in more detail.

One main drawback concerning the TSRST is caused by constraints of the device used in this work since it was not able to reach temperatures lower than -20 °C, which prevents the tests from reaching certain levels of thermal stress and temperature. It is proposed that the equipment may be outfitted with a refrigeration technology that can further lower the temperature in the chamber. This process will make it possible for the TSRST to yield the expected results.

It would be of interest to study the rheological properties at low temperatures and analyse if this can be related to any of the mechanical tests performed. Mollenhauer and Tušar (2016) propose conducting the DSR tests at low temperatures, and that they may better represent the cracking temperatures of the asphalt mixtures. Bending beam rheometer test on asphalt binders can be performed for a better comparison between the asphalt binder and mixture performance.

Another comparison that may be of interest would be to produce an asphalt mixture with the base binder 50/70, to ascertain the improvement the polymer produces in the mixture at low temperatures.

Another significant laboratory test that could not be performed was the Fraass breaking point test. It indicates the temperature at which bitumen first becomes brittle by the appearance of cracks. This test is usually executed when the low-temperature properties of asphalt binders are being evaluated. Thus, it is suggested that this test may be performed in future studies.

Another possible future investigation of interest would concern the effect of ageing in the low-temperature performance of asphalt mixtures. Because the low-temperature cracking resistance of asphalt pavement is reduced during service life and also by ageing effects, it would be essential to determine this effect on asphalt mixtures.

Finally, *in situ* tests with an experimental trial would be of considerable interest to determine the true relation of the laboratory tests with real everyday low-temperature conditions, although those conditions are difficult to be met in Portugal.

REFERENCES

Airey, G.D. (2001). Viscosity-Temperature Effects of Polymer Modification as Depicted by Heukelom's Bitumen Test Data Chart, *International Journal of Pavement Engineering*, Vol. 2 (4).

Airey, G.D. (2002). Rheological evaluation of ethylene vinyl acetate polymer modified bitumens, *Construction and Building Materials*, Vol. 16 (8), pp. 473-487.

Airey, G.D. (2003). Rheological properties of styrene butadiene styrene polymer modified road bitumens, *Fuel*, Vol. 82 (14), pp. 1709-1719.

Alataş, T., Yilmaz, M. (2017). Low temperature properties of hot mix asphalts prepared with different polymer modified binders, *Journal of the Croatian Association of Civil Engineers*, Vol. 69, pp. 93-104.

Almusawi, A., Sengoz, B., Topal, A., One, J. (2019). Comparison between Zero Shear Viscosity and Steady Shear Flow Methods to determine mixing and compaction temperatures of PMB, *Eurasian Journal of Civil Engineering and Architecture*, Vol. 3 (2), pp. 1-8.

Asphalt Institute (2019). *Individual Asphalt Binder Tests*, Lexington, Asphalt Institute; http://www.asphaltinstitute.org/laboratory/testing-services/individual-asphalt-binder-tests/; Acedido em 4/7/2019.

Asphalt Institute (2020). *Determining Lab Mixing and Compaction Temperatures for Binders*, Lexington, Asphalt Institute; http://www.asphaltinstitute.org/engineering/determining-lab-mixing-compaction-temperatures-binders/; Acedido em 13/1/2020.

Becker, Y., Méndez, Maryro P., e Rodríguez, Yajaira (2001). Polymer modified asphalt, *Vision Tecnologica*, Vol. 9 (1).

Behnood, A., Ameri, M. (2012). Experimental investigation of stone matrix asphalt mixtures containing steel slag, *Scientia Iranica*, Vol. 19 (5), pp. 1214-1219.

Bernucci, L., Motta, L., Ceratti, J., Soares, J. (2010). *Pavimentação asfáltica*, Rio de Janeiro.

Blazejowski, K. (2011). *Stone Matrix Asphalt theory and practice*, CRC Press Taylor & Francis Group.

Boutevin, B., Pietrasanta, Y., Robin, J.J. (1989). Bitumen-polymer blends for coatings applied to roads and public constructions, *Progress in Organic Coatings*, Vol. 17 (3), pp. 221-249.

Branco, F., Pereira, P., Picado Santos, L. (2011). *Pavimentos Rodoviários*, Edições Almedina.

Brandão, M.F.C.M. (2015). *Estudo de betumes modificados recuperados de misturas produzidas pelo método húmido e pelo método seco*.

Brown, E., Mallick, R., Haddock, J., Bukowski, J. (1997). Performance of stone matrix asphalt (SMA) mixtures in the United States, *Journal of the Association of Asphalt Paving Technologists*.

Burguete, L.M.F. (2013). *Contribuição para o estudo do comportamento de misturas betuminosas incorporando nanomateriais*, Instituto Superior Técnico.

CEN (2012). *EN 12697-46 Bituminous mixtures - Test methods for hot mix asphalt - Part 46: Low temperature cracking and properties by uniaxial tension tests*, European Committee for Standardization, Brussels.

Das, P., Tasdemir, Y., Birgisson, B. (2012). Low temperature cracking performance of WMA with the use of the Superpave indirect tensile test, *Construction and Building Materials*, pp. 643-649.

Domínguez, J.C. (2018). Rheology and curing process of thermosets, *Thermosets: Structure, Properties, and Applications*, p. 115-146, Elsevier.

EAPA (2018). *Heavy duty surfaces The Arguments for SMA*, European Asphalt Pavement Association, Brussels, Belgium.

EAPA (2019). *EAPA. European Asphalt Pavement Association*, Brussels, European Asphalt Pavement Association; https://eapa.org/; Acedido em 9/7/2019.

Ebewele, R.O. (2000). *Polymer Science and Technology*, CRC Press.

EC (2019). *Environment*, European Commission; https://ec.europa.eu/environment/nature/natura2000/biogeog_regions/continental/index_en.htm; Acedido em 14/10/2019.

El-Badawy, S., Abd El-Hakim, R. (2018). *Recent Developments in Pavement Design, Modeling and Performance*, Proceedings of the 2nd GeoMEast International Congress and Exhibition on Sustainable Civil Infrastructures, Egypt.

Encyclopaedia Britannica (2019). *Polymer*, https://www.britannica.com/science/polymer; Acedido em 12/12/2019.

Fernandes, S., Pereira, S., Silva, H., Oliveira, J., Palha, C. (2016). *Avaliação de métodos de caracterização de betumes modificados*, 1° Congresso de Ensaios e Experimentação em Engenharia Civil, Relacre, IST, Lisboa.

Gawel, I., Stepkowski, R., Czechowski, F. (2006). Molecular Interactions between Rubber and Asphalt, *Industrial & Engineering Chemistry Research*, Vol. 45 (9), pp. 3044-3049.

Hassan, N., Airey, G., Putra Jaya, R., Mashros, N., A. Aziz, M.M. (2014). A Review of Crumb Rubber Modification in Dry Mixed Rubberised Asphalt Mixtures, *Jurnal Teknologi*, Vol. 70.

Hassan, N., Airey, G., Yusoff, N., Hainin, M., Putrajaya, R., Abdullah, M., Aziz, M. (2015). Microstructural characterisation of dry mixed rubberised asphalt mixtures, *Construction and Building Materials*, Vol. 82, pp. 173-183.

Hischke, G. (2019). *Understanding Creep in Asphalt Concrete*, Illinois Asphalt Paving Association (IAPA).

Hornych, P., Blanc, J., Gabet, T., a, P., Piau, J., Di Benedetto, H. (2012). Creep tests on bituminous mixtures and modelling, *Road Materials and Pavement Design*.

Isacsson, U., Zeng, H. (1997). Relationships between bitumen chemistry and low temperature behaviour of asphalt, *Construction and Building Materials*, Vol. 11 (2), pp. 83-91.

Judycki, J. (2014). Influence of low-temperature physical hardening on stiffness and tensile strength of asphalt concrete and stone mastic asphalt, *Construction and Building Materials*, Vol. 61, pp. 191-199.

Kim, K.W., Hussein, M.E. (1997). Variation of fracture toughness of asphalt concrete under low temperatures, *Construction and Building Materials*, Vol. 11 (7-8), pp. 403-411.

Lesueur, D. (2009). The Colloidal Structure of Bitumen: Consequences on the Rheology and on the Mechanisms of Bitumen Modification, *Advances in Colloid and Interface Science*, Vol. 145 (1-2), pp. 42-82.

Mandal, T., Sylla, R., Bahia, H., Barmand, S. (2015). Effect of cross-linking agents on the rheological properties of polymer-modified bitumen, *Road Materials and Pavement Design*.

Miranda, H.M.B., Batista, F.A., Antunes, M.d.L., Neves, J. (2019). A new SMA mix design approach for optimisation of stone-on-stone effect, *Road Materials and Pavement Design*, Vol. 20.

Mollenhauer, K., Tušar, M. (2016). *FunDBitS : functional durability-related bitumen specification. Deliverable D.2c, correlations between bitumen and asphalt properties : low temperature cracking*, Conférence Européenne des Directeurs des Routes = Conference of European Directors of Roads (CEDR), p. 18, Bruxelles.

Mollenhauer, K., Wistuba, M. (2012). Evaluation of hot-mix asphalt susceptibility to temperature-induced top-down fatigue cracking by means of Uniaxial Cyclic Tensile Stress Test, *Road Materials and Pavement Design*, Vol. 13 (1), pp. 171-190.

Nicholls, C. (2017). *Asphalt Mixture Specification and Testing*, CRC Press.

Paliukait, M., Vaitkusa, A., Zofkab, A. (2014). *Evaluation of bitumen fractional composition depending on the crude oil type and production technology.*, 9th International Conference "Environmental Engineering" Selected Papers, p. 22–23, Vilnius, Lithuania.

Pangarova, D., Nikolov, A. (2016). *Study of the low temperature characteristics of binders and the asphalt mixtures produced with them*, 6th Eurasphalt & Eurobitume Congress, Prague.

Pavement Interactive (2019). *Pavement Interactive: Free online knowledge for the paving industry*, Pavement Interactive,; https://pavementinteractive.org/; Acedido em 13/6/2019.

Polacco, G., Kříž, P., Filippi, S., Stastna, J., Biondi, D., Zanzotto, L. (2008). Rheological properties of asphalt/SBS/clay blends, *European Polymer Journal*, Vol. 44 (11), pp. 3512-3521.

Porto, M., Caputo, P., Loise, V., Eskandarsefat, S., Teltayev, B., Rossi, C.O. (2019). Bitumen and Bitumen Modification: A Review on Latest Advances, *Applied Sciences*.

Pszczola, M., Jaczewski, M., Szydlowski, C. (2019a). Assessment of Thermal Stresses in Asphalt Mixtures at Low Temperatures Using the Tensile Creep Test and the Bending Beam Creep Test, *Applied Sciences*, Vol. 9 (5).

Pszczoła, M., Judycki, J. (2009). *Testing of low temperature behaviour of asphalt mixtures in bending creep test*, 7th International RILEM Symposium ATCBM09 on Advanced Testing and Characterization of Bituminous Materials, Rhodes, Greece.

Pszczola, M., Judycki, J., Ryś, D. (2016). Evaluation of Pavement Temperatures in Poland During Winter Conditions, *Transportation Research Procedia*, Vol. 14, pp. 738-747.

Pszczola, M., Szydlowski, C. (2018). Influence of Bitumen Type and Asphalt Mixture Composition on Low-Temperature Strength Properties According to Various Test Methods, *Materials*.

Pszczola, M., Szydlowski, C., Jaczewski, M. (2019b). Influence of cooling rate and additives on low-temperature properties of asphalt mixtures in the TSRST, *Construction and Building Materials*, Vol. 204, pp. 399-409.

Quirk, R.P., Kinning, D.J., Fetters, L.J. (1989). 1 - Block Copolymers, *Comprehensive Polymer Science and Supplements*, Vol. 7, pp. 1-26.

Read, J., Witheoak, D. (2003). *The Shell Bitumen Handbook*, Thomas Telford Publishing, London, UK.

Remišová, E., Holý, M. (2017). *Changes of Properties of Bitumen Binders by Additives Application*, IOP Conference Series: Materials Science and Engineering, IOP Publishing Ltd.

Saal, R., Pell, P. (1960). *Fatigue of bituminous mixes*, Conference on Flow, Fatigue and Failure of the British Society of Rheology.

SABITA (2015). *Technical Guideline : The Use Of Modified Bituminous Binder In Road Construction*, Southern African Bitumen Association (Sabita).

Saboo, N., Kumar, P. (2016). Optimum Blending Requirements for EVA Modified Binder, *Transportation Research Procedia*, Vol. 17, pp. 98-106.

Scheirs, J. (2003). *Modern Styrenic Polymers: Polystyrenes and Styrenic Copolymers*, John Wiley & Sons.

Senço, W. (2001). *Manual de Técnicas de Pavimentação*, Pini Ltda.

Sengoz, B., Isikyakar, G. (2008). Evaluation of the properties and microstructure of SBS and EVA polymer modified bitumen, *Construction and Building Materials*, Vol. 22 (9), pp. 1897-1905.

Shekar, P.C., H., R.B., Vishwas, J.S. (2018). Experimental investigation of Stone Mastic Asphalt by varying mix design., *International Research Journal of Engineering and Technology (IRJET)*, Vol. 5 (5), pp. 300–305.

Shenoy, A. (2001). Determination of the Temperature for Mixing Aggregates with Polymer-Modified Asphalts, *International Journal of Pavement Engineering*, Vol. 2 (1), pp. 33-47.

SHI (2019). *Science Matters: The Case of Plastics*, Philadelphia, Science History Institute; https://www.sciencehistory.org/the-history-and-future-of-plastics; Acedido em 11/11/2019.

Shinohara, K.J., Momm, L., Leandro, G.C., Barra, B.S., Pérez, Y.A.G. (2011). Avaliação do efeito da adição do polímero EVA nas propriedades do ligante asfáltico 50/70, *Universidade Federal de Santa Catarina*.

Silva, H.M.R.D., Oliveira, J., Ferreira, C.I.G., Peralta, E.J.F. (2009). *Evaluation of the rheological behaviour of Warm Mix Asphalt (WMA) modified binders*, 7th International RILEM, Taylor & Francis, p. 661-673, Rhodes.

Špaček, P., Hegr, Z., Beneš, J. (2017). Practical experiences with new types of highly modified asphalt binders, *IOP Conference Series: Materials Science and Engineering*, Vol. 236.

Stimilli, A., Virgili, A., Canestrari, F., Bahia, H.U. (2017). Estimation of low-temperature performance of recycled asphalt mixtures through relaxation modulus analysis, *Cold Regions Science and Technology*, Vol. 133, pp. 36-45.

Stock, A.F., Arand, W. (1993). *Low temperature cracking in polymer modified binders*, Asphalt Paving Technology 1993, Austin, Texas.

Sun, Y., Gu, Z., Wang, J., Fang, C., Yuan, X. (2018). Study on Relaxation Damage Properties of High Viscosity Asphalt Sand under Uniaxial Compression, *Advances in Civil Engineering* (Pavement Analysis and Design by Multiphysics).

The Constructor (2020). *Types of Pavement – Flexible Pavements and Rigid Pavements*, The Constructor. Civil Engineering Home; https://theconstructor.org/transportation/types-of-pavement-flexible-and-rigid-pavement/9570/; Acedido em 12/1/2020.

Torres, H. (2014). *Utilização de betumes modificados na regeneração de misturas com elevadas percentagens de reciclagem*.

Toth, C., Petho, L., Geiger, A., Soós, Z. (2016). *Performance assessment of hot mix asphalt with chemically stabilized rubber bitumen*, JO - Proceedings of 6th Eurasphalt & Eurobitume Congress 2016.

Vaitkus, A., Vorobjovas, V., Žiliūtė, L. (2009). *The research on the use of warm mix asphalt for asphalt Pavement structures*, XXVII International Baltic Road Conference, Riga, Latvia.

Van Amsterdam, E. (2000). *Construction materials for civil engineering*, Juta and Company Ltd.

Vonk, W., Scholten, E.J., Korenstra, J. (2010). *Novel Class of SBS Polymers for Enhanced Effectiveness in Bitumen Modification*, Australian Asphalt Paving Association Thirteenth International Flexible Pavements Conference, Queensland, Australia.

Walker, D. (2019). *The benefits of modified asphalts*, Asphalt: The Magazine of the Asphalt Institute; http://asphaltmagazine.com/the-benefits-of-modified-asphalts/; Acedido em 8/9/2019.

Wang, T.-h., Su, L.-j., Zhai, J.-y. (2014). A case study on diurnal and seasonal variation in pavement temperature, *International Journal of Pavement Engineering*, Vol. 15 (5).

WorldAtlas (2019). *Europe Weather*, https://www.worldatlas.com/webimage/countrys/euweather.htm; Acedido em 11/10/2019.

Zaniewski, J.P., Pumphrey, M.E. (2004). *Evaluation of Performance Graded Asphalt Binder Equipment And Testing Protocol*, Asphalt Technology Program.

Zhang, B., Chen, H., Zhang, H., Kuang, D., Wu, J., Zhang, X. (2019). A Study on Physical and Rheological Properties of Rubberized Bitumen Modified by Different Methods, *Materials*.