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**EVALUATION OF PERFORMANCE-BASED CRITERIA FOR SELECTION OF  
MATERIALS FOR SURFACE TREATMENTS**

**FORTALEZA**

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GLEDSON SILVA MESQUITA JÚNIOR

*EVALUATION OF CRITERIA FOR SELECTION OF MATERIALS FOR SURFACE  
TREATMENTS BASED ON PERFORMANCE*

Dissertação de Mestrado apresentada ao Programa de Pós-Graduação em Engenharia de Transportes da Universidade Federal do Ceará, como requisito parcial à obtenção do título de Mestre em Engenharia de Transportes. Área de concentração: Infraestrutura de Transportes.

Orientadora: Profa. Dra. Suelly Helena de Araújo Barroso.

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## RESUMO

Os Tratamentos Superficiais por Penetração (TSP) são utilizados com frequência nos revestimentos asfálticos brasileiros, com destaque para a Região Nordeste, de Rodovias de Baixo Volume de Tráfego (RBVT). Esta dissertação tem como objetivo propor critérios para a seleção de agregados e ligantes utilizados em TSPs visando-se alcançar melhor desempenho em relação aos fenômenos da exsudação e da perda agregado. Para tanto, esse estudo foi subdividido em três partes que foram organizadas em formato de artigos. No primeiro artigo, foram avaliadas as mudanças que ocorrem, durante a vida de serviço de diferentes emulsões asfálticas comumente utilizadas na execução dos revestimentos de TSP. Para isso, foram utilizados métodos laboratoriais de envelhecimento para condicionar as amostras - ultravioleta (UV) e Pressure aging vessel (PAV). Nesse primeiro artigo, foram avaliadas as características reológicas dos materiais e como elas se relacionam com o desempenho dos revestimentos. Foi observado que os efeitos do envelhecimento UV são diferentes dos efeitos do PAV, dependendo do material, e que essas mudanças apresentam impactos no desempenho dos TSP. O segundo artigo abordou a ligação entre agregado e ligante focando nos fenômenos de adesividade e coesividade. Os resíduos de emulsões envelhecidos foram avaliados com o uso do Bitumen Bond Strength (BBS), teste de adesividade direcionado aos ligantes asfálticos. A aderência desses materiais ao agregado após o envelhecimento foi então observada. Concluiu-se com o segundo artigo que os ligantes tendem a necessitar de uma tensão de arracamento maior com o envelhecimento e que os efeitos do envelhecimento UV e do PAV impactam na adesividade, coesividade e no dano por umidade. O terceiro artigo focou na perda de agregado, o maior fator de falha em TSPs, considerando rodovias do estado do Ceará. As propriedades de forma dos agregados e a uniformidade foram as variáveis estudadas e seus impactos no desempenho foram avaliados sob a perspectiva desse defeito. Para tanto, agregados com variação nesses parâmetros foram selecionados, e corpos de prova moldados em laboratório foram testados a partir do Wet Track Abrasion Test (WTAT). Foi observado que a organização dos agregados na superfície do revestimento, aliado a alta uniformidade e baixa lamelaridade, é relevante na obtenção de melhor desempenho em relação à perda de agregado. Em geral, neste trabalho pode-se concluir que as diversas propriedades encontradas nos agregados e nos ligantes asfálticos podem refletir no desempenho dos TSPs. Em relação aos ligantes, essas características ainda podem apresentar grande variação durante a vida de serviço devido ao envelhecimento. É importante, dessa forma, considerar esses efeitos em projetos, assim como meios de evitar a perda de qualidade dos materiais durante a vida de serviço.

**Palavras-chave:** tratamento superficial; emulsões asfálticas; envelhecimento; adesividade; perda de agregado;

## ABSTRACT

Surface treatments are often used in Brazilian pavements. Among those, chip seals (CS) are commonly used in the Low Volume Roads (LVR) in the Northeast Region. This Master's Thesis aims to propose criteria for selection of aggregates and binders for CS Coats aiming to achieve best performance regarding functional problems such as aggregate loss and bleeding. For this purpose, this research was subdivided into three parts that were organized in paper format. In the first paper, changes that occur during service life in features of the asphalt emulsions used in CS treatments were evaluated. In this regard, two methods of laboratory aging were evaluated: ultraviolet (UV) and Pressure aging vessel (PAV). In this first paper, rheological parameters of emulsion residue were analyzed, as well as their relation to the performance of coats. It was observed that the effects of UV aging are different from those found in PAV, depending on the material. It was also observed that these effects reflect in implications in the performance of coats. The second paper addresses the binder-aggregate bond and the phenomena of adhesiveness and cohesiveness. The aged residues of emulsion were tested with the Bitumen Bond Strength (BBS), adherence test recommended for asphalt binders. The adherence of these materials to the aggregate after aging was then observed. It was noticed that aged binders tend to present higher Pull-off tensile due to the effects of UV and PAV in adhesiveness/cohesiveness, and in moisture damage. The third paper focused on aggregate loss as it is the major failure factor in CS, considering pavements in the state of Ceara. Aggregate shape and uniformity were defined as the parameters and their influence on performance was evaluated from the perspective of aggregate loss distress. For this purpose, aggregates with variations in these parameters were selected and samples of specimen were constructed in laboratory and tested by using the Wet Track Abrasion Test (WTAT). It was observed that the organization of the particles of aggregate on the surface of the coat, summed to both high uniformity and low flatness/elongation, is relevant to the acquisition of better performance concerning aggregate loss. In this research, it is possible to conclude that properties found in the aggregates and in the asphalt binders might present effects on the performance of CS pavements. In relation to the binder, these properties also might present notorious variation during service life due to aging. Based on that, it is relevant to consider these effects in projects, as well as techniques to avoid the loss of quality of the materials during service life.

**Keywords:** chip seal; asphalt emulsions; aging; adhesiveness; aggregate Loss

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## LIST OF ABBREVIATIONS

(%) $P_{2EM}$	Percentage of aggregates larger than 1.4 M
(%) $P_{EM}$	Percentage of aggregates lower than 0.7 M
$ G^* $	Complex modulus
AASHTO	American Association of State Highway and Transportation Officials
ABS	Asphalt bond strength
AE	Asphalt emulsions
AIMS2	Aggregate imaging measurement system
ASTM	American Society for Tasting and Materials
BBS	Bitumen bond strength
CNT	Confederação Nacional do Transporte
COR	Curing on rock
CS	Chip seal
CV	Coefficient of variation
DCS	Double chip seal
DER	Departamento Estadual de Rodovias
DIP	Digital image processing
DNIT	Departamento Nacional de Infraestrutura de Transportes
DSR	Dynamic shear rheometer
F and E	Flatness and Elongation
FOT	Flip-over test
FTIR	Fourier transform infrared
$I_{Aro}$	Aromatics index
$I_c$	Carbonyl index
$I_s$	Sulfoxide index
$J_{nr}$	Non-recoverable compliance
LVR	Low volume roads
LWT	Loaded wheel track
MSCR	Multiple stress creep recovery
PATTI	Pneumatic adhesion tensile testing instrument
PAV	Pressure aging vessel
PDI	Processamento Digital de Imagens
POTS	Pull-off tensile strength

PUC	Performance-based Uniformity Coefficient
R	Recovery
R <sub>agg</sub>	Rate of aggregate
RBVT	Rodovia de Baixo Volume de Tráfego
R <sub>em</sub>	Rate of emulsion
ROR	Residue on rock
RPOTS	Bond strength ratio
RS	Rapid-setting
RTFOT	Rolling thin film oven test
SCS	Single chip seal
TCS	Triple chip seal
TS	Tratamento superficial
UV	Ultraviolet
WTAT	Wet track abrasion test
μ	Arithmetic mean

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## 1 INTRODUCTION

### 1.1 Contextualizing

Emerging countries often have a need for cost-effective solutions to improve the quality of transport systems. In these cases, infrastructure investments are usually not enough to address structural problems and to attend to the demand concerning growth. In Brazil, for example, data show that 22.4% of national asphalt pavements are in poor traffic conditions (DNIT, 2018). In addition, the majority of roads operates without a surface course and did not even receive any intervention to enable traffic, resulting in approximately 78,5% of network composed by unpaved roads (CNT, 2019). Among this percentage, most roads are classified as Low Volume Roads (LVR).

In Brazil, especially in Northeast Region the use of thin layers of surface course is a common practice for increasing the number of paved LVR. Surface treatments represent a type of coat applied with high frequency in such cases. Among that, it is possible to mention the Seal Coats. This technique simply consists of the application of a layer of bituminous binder followed by a layer of aggregate and compaction (LARSEN, 1985).

In general, the service level of Seal Coats might be related to features found in the materials used. Examples are the shape and size of particles of aggregate, as well as the viscosity and stiffness of the binder. Some construction factors should also be considered, such as application rates of aggregate and binder and type of compaction used. It is important to consider that standardization used in Brazil generally do not indicate how these factors should be addressed in the design of seal coats, which encourages the construction of coats based on empirical criteria.

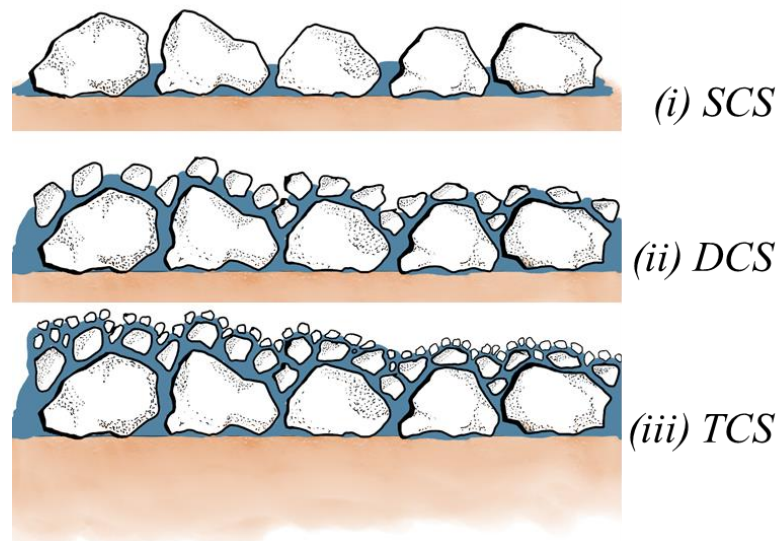
In Brazil, surface treatments are frequently the designation given to courses of Chip Seals constructed with up to three layers of aggregate. National standards (DNIT, 2006) categorize these services in the group of bituminous surface courses built by penetration of binder. For purpose of arrangement, regarding this research, these types of pavements will be referred as Chip Seals (CS) pavements, and other types of Surface Treatments will be referred as Seal Coats. In relation to the national classification of CS-type pavements, this is usually determined by the number of layers of binder-aggregate applied:



- i. Single Chip Seal (SCS): Seal Coats composed by a single layer of aggregate;
- ii. Double Chip Seal (DCS): Seal Coats composed by two layers of binder and aggregate; or two applications of SCS;
- iii. Triple Chip Seal (TCS): Seal Coats composed by three layers of binder and aggregate.

Figure 1 illustrates a scheme of each type of CS based on the number of layers. This classification may be different depending on the state or country. Also, there are other types of Seal Coats that use the CS technique, such as Racked-in Seal and Cape Seal. However, these pavements are not as used in national practice as CS. National practice usually recommends that aggregates in upper layers must have lower size, to fit voids and increase the quality of macrotexture. Further details about gradation and other information of aggregates used in CS-type pavements can be obtained by contacting local agencies and DERs, the state departments of transportation and infrastructure of Brazil.

Figure 1 – Classification of CS based on number of layers.



Source: author (2021)

In the State of Ceará, DCS and TCS represent together approximately 58% of surface courses applied on highways (SOP-CE, 2019). For construction purpose, the local design standards DERT ES – P 10/00 and DERT ES – P 11/00 (DER-CE, 2000) are frequently used in this state. National standards DNIT-ES 146/2012 and DNIT-ES 147/2012 (DNIT, 2012) are also commonly used. It is important to consider that although these standards address the same service, they provide different and sometimes not-explicit recommendations, leading certain judgments to the operator. As a consequence of that, lots of CS Coats present failure before

achieving the service life. This problem is also present in other states that use Seal Coats as a surface course. In many cases these states do not have a local standard to perform this service.

This technique has been reviewed in many countries where Seal Coats are often used such as the United States, New Zealand, and Australia. In these cases, the results were better concerning performance of pavements (LEE and KIM, 2009; TRANSIT IN NEW ZEALAND, 2005). For these countries, the most common to guide the construction of new CS-type pavements is the use of manual of best practices which specifies the correct usage for each material and equipment. In general, this method addresses to better developed highway network, in which Seal Coats are used as a mean of conservation of other types of surface courses. However, the use of this technique in LVR is also common.

Studies frequently focus on phenomena that cause low efficiency of coatings and on how to establish criteria to avoid this situation. It is understood that the main causes of failure on Seal Coats are due to bleeding and/or aggregate loss (SILVA, BARROSO, and KIM, 2018). Thus, standards and manuals for this service must include criteria in order to evaluate and make decisions based on these defects.

National specifications for this service in general present an empirical procedure and do not consider these criteria in its conception, thus the methods for evaluating these defects are often not utilized and chip seals commonly operate below its potential.

## **1.2 Background and Research Problem**

Based on the considerations made in the previous section, it is possible to infer that design and execution standards used in Brazil for Seal Coats present several deficiencies that may directly affect the efficiency of pavements that need to be considered. The main problem studied in this master thesis is the low efficiency of national practice regarding Seal Coats.

Studies of materials and techniques which focus on LVR are a necessity, since investments on these types of roads are directly related to development of places where the access to basic facilities is limited. In fact, it is expressive the percentage of highways in Brazil that presents construction problems and irregularities. Among these, most are not even able to receive traffic. Considering the majority of these roads have never received any kind of asphaltic layer in its structure, thin layers of surface courses are an alternative that contribute to increase their quality at a low cost.

It is important to consider that although Seal Coats are frequently presented as the most appropriate option for improving quality of unpaved roads, their construction must be carried out responsibly to avoid unnecessary waste of resources. Studies which focus on efficiency of pavements of this type are presented as a solution for both proper selection of materials and definition of the most viable construction techniques.

### **1.3 Research objective**

It was defined as the general objective to propose criteria for selection of aggregates and binder for CS Coats aiming to achieve best performance regarding functional problems such as aggregate loss and bleeding.

#### ***1.3.1 Specific objectives***

Based on the general objective defined previously, it was also established the following specific objectives.

- i. Analyze the effects that aging mechanisms present on rheological parameters of asphalt binders and on the performance of CS coats.
- ii. Investigate how adhesion and cohesion evolve based on the aging of binder and which variables might be related to failures on this interface.
- iii. Evaluate the impact that changes in aggregate properties (shape and uniformity) have in both: the arrangement of the aggregates in the surface of CS coats and the performance concerning the problem of aggregate loss.

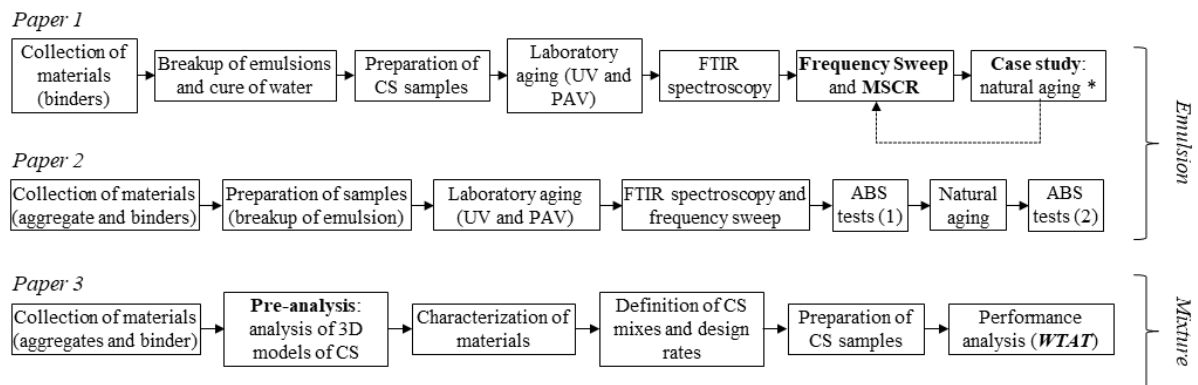
### **1.4 Considerations about Method and Thesis Organization**

Based on the objectives previously defined, the experimental plan was sectioned into three parts, each one referring to a specific objective. In addition, the proposed method consists of three phases, subdivided so that each one refers to a specific part of the experimental plan, as summarized in Figure 2.

Within this context, it was defined to present each part of the experimental plan in a separate section, in paper format. Thus, this master thesis is made up of three papers: (i) the first one, refers to the study of rheology of emulsion residue after aging; (ii) the second, focuses on the evolution of the adhesiveness/cohesiveness in the binder-aggregate interface; and (iii) the third one, focuses on the phenomenon of aggregate loss. Also, it is presented a section of

conclusions in which there are technical contributions to a national manual of the best practices based on material features concerning aggregate loss and bleeding.

Figure 2 – Flow chart overview of the experimental plan



Source: author (2021)

Thus, this thesis is proposed to have five chapters:

- **CHAPTER 1: INTRODUCTION** – This chapter presents the context of research and definition of the research problem. Research objectives and a summary of the structure of the experimental plan are also presented.
- **CHAPTER 2: RHEOLOGY OF BINDER: EVALUATION OF EMULSIONS USED IN SURFACE TREATMENT IN DIFFERENT STAGES OF AGING** – This chapter, presented in paper format, focuses on the study of residue of emulsions and their properties after aging. Several rheological parameters of this material are studied and how it relates concerning the performance of CS-type coatings.
- **CHAPTER 3: BINDER- AGGREGATE BOND: ANALYSIS OF BINDER-AGGREGATE INTERFACE OF CHIP SEAL BINDERS IN DIFFERENT STAGES OF AGING USING BITUMEN BOND STRENGTH**– This chapter, presented in paper format, is focused on the study of adhesion between aggregates and binders used in CS-type coats. Aging effects are also considered in this analysis.
- **CHAPTER 4: PERFORMANCE OF CS: ANALYSIS OF AGGREGATE LOSS IN SURFACE TREATMENTS FOCUSING ON UNIFORMITY AND SHAPE PROPERTIES** – This chapter, presented in paper format, addresses the phenomenon of aggregate loss in CS-

type coatings. Factors that might correlate to better or worse performance considering the occurrence of this failure are analyzed.

- CHAPTER 5: CONCLUSIONS – This chapter presents general conclusions obtained after analyzing features of materials (binder and aggregate) used in CS-type pavements and how they might relate to performance. In addition, suggestions for a manual of best practices addressing Seal Coats are presented.

### **1.5 Considerations about Literature Review**

Concerning the proposed organization this document is subdivided into papers and thematic areas, the same organizational pattern was used for literature review. Thus, the literature review was subdivided and presented in separate parts for each paper.

## **2 RHEOLOGY OF BINDER: EVALUATION OF EMULSIONS USED IN SURFACE TREATMENT IN DIFFERENT STAGES OF AGING**

### **2.1 Abstract**

This paper aims to evaluate effects that aging mechanisms present on rheological properties of asphalt binders used in chip seal (CS) pavements in Brazil. Considering that most of these treatments are conducted with the use of rapid-setting emulsions, this part of the research focuses on rheological parameters and aging effects of the inner asphalt residue of this material. Therefore, three types of binder were used: two rapid-setting emulsions (unmodified and polymeric) and a conventional asphalt cement binder. These materials were subjected to laboratory-induced aging using ultraviolet (UV) radiation and PAV. After that, the samples were evaluated with two different tests: (i) firstly, the frequency sweep in Dynamic Shear Rheometer (DSR), in which it was obtained the master curves of the complex modulus ( $|G^*|$ ) and the phase angle ( $\delta$ ) of these materials; (ii) secondly, the Multiple Stress Creep Recovery (MSCR) was carried out. Also, it was evaluated the Fourier-Transform Infrared (FTIR) spectroscopy to obtain aging parameters. The results suggest that the effect of UV in the aging of binders are not constant. The decrease of  $J_{nr}$ , for example, was better explained by an exponential curve. Regarding to performance, the unmodified emulsion presented higher tendency to develop bleeding than the polymeric emulsion. This parameter was above the limits in the first 100 hours of UV aging, suggesting a predisposition of the binders to distresses of bleeding in the beginning of service life.

### **2.2 General Considerations**

Asphalt Emulsions (AE) are primarily materials composed of Asphalt Cement and water. This composition is recommended for surface courses in which application and construction are carried out at lower temperatures. Among the benefits of the use of this material, it is possible to highlight the workability and thus the reduction of energy consumption in its application. This results in reduced costs and less damage to the environment.

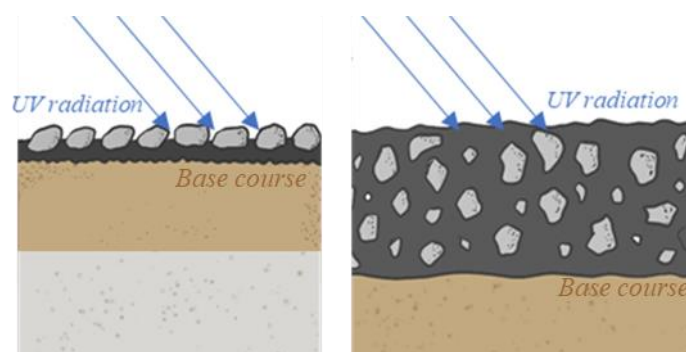
In Brazil, emulsions are manufactured with attributes that are better suited to each type of project. Examples of use of emulsions are prime coats, micro surfacing, warm mixtures, and surface treatments. Chip Seals (CS), the focus of this research, in some locations of Brazil represents more than half of the surface courses found in paved network, which demonstrates the significance of this technique for national infrastructure.

In emulsions, the bituminous material is dispersed in water in the form of microscopic droplets and surrounded by an emulsifier agent, a chemical component that can be cationic or anionic depending on the charge of the polar part of the molecule which is used for keeping the stabilization of the system. The destabilization of this system leads to the union of asphalt particles and water evaporation, which are respectively defined as emulsion breaking (or rupture) and cure. Emulsions are manufactured and classified based on the time required for this process.

Pavements constructed with CS techniques frequently make use of rapid-setting (RS) emulsions. In such coats, the first material to be applied is the emulsion, followed by a layer of aggregates which is then compacted. This process is intended to be carried out promptly so that the asphalt binder can adhere to the aggregate surface before the emulsion breaks. After the emulsion breaks, it is important to wait a period for curing the water, which may vary depending on the type of emulsion used and the climatic conditions of the site. However, this is expected to happen as quickly as possible.

Climatic conditions also have effects on service life of CS pavements. Among the factors that can affect the characteristics of materials and its durability are heating, UV radiation, rain, snow, and factors that increase the oxidation rate. In general, methods to test aging of binder in asphalt mixtures are based on the process of heating and do not consider the influence of UV radiation. Since this factor strongly affects the surface of pavements, it is important to consider how this is reflected in the aging of surface treatments. Considering that this type of coat is thin and directly exposed to the atmosphere. It is expected that the binder within these layers reach service life in a different period when compared to other asphalt layers. Figure 3 shows a demonstrative scheme of how this can affect the surface of pavements, especially thin layers such as CS-type coats.

Figure 3 – Scheme of a CS pavement affected by UV radiation.



Source: author (2021)

This chapter addresses the first specific objective of this master thesis, which is to investigate the effects that aging mechanisms present on rheological parameters of asphalt binders and, consequently, on the performance of CS coats.

## **2.3 Literature Review**

### **2.3.1 Asphalt emulsions**

Asphalt Emulsions are applicable in most asphalt paving processes, especially those in which it is desirable to achieve lower viscosity than pure asphalt cement at lower temperatures. This material is composed of 5-55  $\mu\text{m}$  asphalt particles dispersed in water (RONALD AND LUIS, 2016). Since the aqueous phase represents the medium where asphalt binder is dispersed, the reduction in viscosity can be explained by the water/bitumen ratio. Another component of AE is the surfactant, or emulsifier, which concentrates at the surface of asphalt particles and is used to stabilize emulsions and prevent segregation of aqueous phase.

The system created by these three components is thermodynamically unstable since several mechanisms can cause the emulsion break, such as: addition of a mineral to the system; evaporation of water; and the coalescence of asphalt particles (RONALD AND LUIS, 2016). Generally, the time of breakup of an emulsion is related to the type and amount of emulsifier used in its composition. Rapid-setting emulsions, for example, tend to make use of lower emulsifier rates.

Another characteristic that also depends on the emulsifier is the emulsion charge, which can be cationic (positive charge) or anionic (negative charge). In Brazil, the production of cationic emulsions is notorious higher than the production of anionic emulsions. In fact, the normative is almost entirely directed to cationic emulsions (CERATTI, BERNUCCI and SOARES, 2015). This is due to the cationics which present a satisfactory adhesion with both alkaline and acid aggregates (ABEDA, 2010).

As mentioned previously, the national practice of CS commonly makes use of rapid-setting emulsions, for example, the “RR-2C” type, the Brazilian code for rapid-setting emulsions with 67% of asphalt content (SILVA, 2018; PEREIRA, 2013). This feature contributes to the fact that, once the road construction is completed, pavements are often quickly opened for traffic. Another consideration to be made about that property is that, as these systems are more unstable, emulsions used in CS treatments show sensitive performances concerning



the temperature of application and the duration of each constructive step. Thus, it is important that both the duration between the application of binder and aggregate and between that and compaction are defined.

Regarding temperature control, Silva (2018) observed that both heating temperature in the application and cure temperature influence the performance of coats. The author concluded that heating the emulsion (~58° C) before its application is indispensable and critical for good performance, especially for colder locations, and ensures better adhesion between binder and aggregate. Furthermore, Silva (2018) also concluded that when analysing ravelling, samples cured in an oven at 50° C showed lower loss than samples cured at room temperature (27° C for that study).

Thus, the conception of a method of laboratory evaluation of CS performance should consider the variation of temperature to be effective. Also, it is important to notice whether the emulsion itself varies in characteristics between curing conditions. The curing process depends on the weather conditions in which the emulsion is used. Taking into consideration that just the temperature used in this process might contribute to better or worse performance of a CS, it is also important to evaluate materials based on specific conditions of each location to achieve the best performance of these types of pavements.

Also, regarding specific test conditions, it is recommended that a procedure adopted to recover the binder residue must approximate the temperature of construction operations (TAKAMURA, 2015). There is a standardized method to perform this process in lab, such as ASTM D7497 (ASTM, 2016). However, since there is no specific standard to evaluate binder residue in Brazilian practice, it is relevant to mention that this method might not be the best to obtain asphalt residue from AE produced in Brazil.

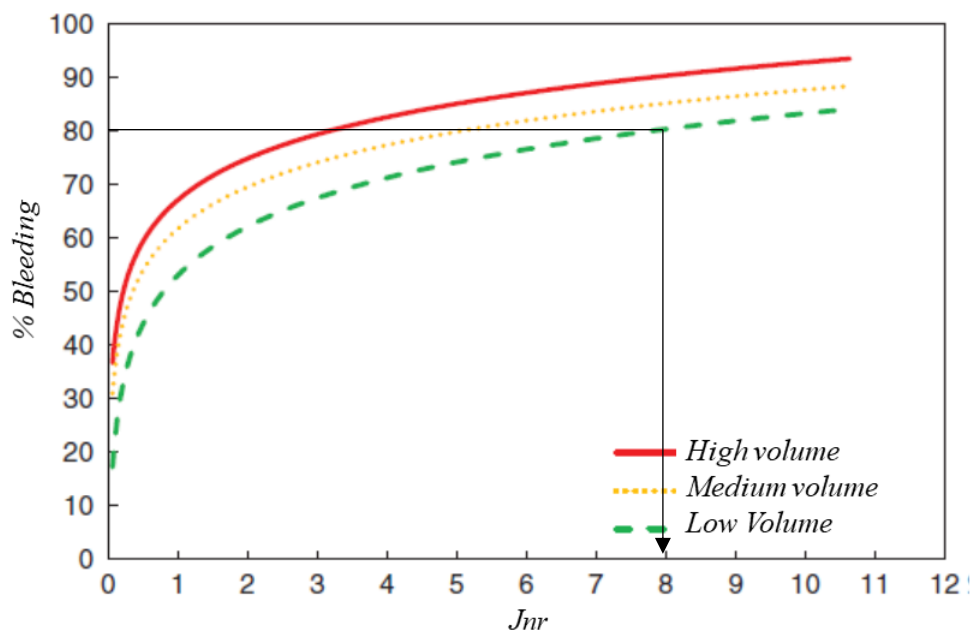
Excessive heating of ordinary asphalt materials might result in increase of stiffness (BERNUCCI *et al.*, 2008). When it comes to emulsions, changes in the characteristics of the binder are also expected due to evaporation of water present in the system (ABEDA, 2010). Thus, aging and change in water content should be both considered for this type of material.

Tests that are based on rheological properties of materials frequently are recommended to evaluate asphalt binders due to their thermoviscous behaviour. Among that, it is the Multiple Stress Creep Recovery (MSCR). In this test, materials are subjected to loading and unloading cycles and the binders' elastic and non-elastic responses are observed. Adams (2014) and Kim *et al.* (2017) recommend this test for emulsion residues to estimate CS performance regarding

the risk of bleeding. In a study that related bleeding and the non-recovery compliance ( $J_{nr}$ ) parameter, Kim *et al.* (2017) established a tendency curve from which it is possible to estimate bleeding.

Figure 4 shows the curves suggested by the authors for the three traffic levels studied in the aforementioned research (high, medium, and low). For technical purposes, it was defined that 80% of bleeding represents the service limit for the test used. Adams (2014), in a test with several types of emulsion, explains that the 80% value would be ideal for commercial purposes as it is highly likely that materials which did not meet this criterion would surely not be ideal for CS. However, it is recommended lower  $J_{nr}$  to ensure better performance. From this criterion, it was possible to define, based on traffic level, a  $J_{nr}$  limit value for acceptance of emulsions.

Figure 4 –  $J_{nr} \times$  Bleeding curve considering 3 traffic levels



Source: adapted from Adams (2014)

Hanz, Johannes and Bahia (2012) also presented a study about performance of CS taking into perspective parameters extracted from the MSCR test and similarly realized that a good way to estimate susceptibility of CS to failure by bleeding is to analyse the  $J_{nr}$  parameter. In this study, a group of emulsions used in CS construction was characterized, as well as performance tests were developed in samples of the CS itself regarding the causes of distresses. At the end of the study, materials were ranked, and it was possible to validate the premise that  $J_{nr}$  is effective in explaining the bleeding phenomenon.

In order to analyse aggregate loss, it was observed that it is possible to estimate a relation between this distress and rheological properties of binder. Kim *et al.* (2017), for example, observed that lower  $G^*$  is better for resisting aggregate loss. Also, it was observed that the phase angle is not the best property to estimate binder performance in relation to aggregate loss. These parameters can be obtained when testing the emulsion residue in a dynamic shear rheometer in a frequency sweep (KIM *et al.*, 2017). Taking into consideration that, like bleeding, this is one of the distresses that is a common CS failure. It is important to make use of tests that can be reproduced in materials to estimate performance before construction.

Another recommended test is the adherence test, evaluated by using the Bitumen Bond Strength (BBS) test (ADAMS, 2014). This test can indicate both the tensile necessary to induce failure in the binder-aggregate bond and whether this failure is cohesive or adhesive.

### 2.3.2 Aging

Aging is one of the factors that directly affect the performance of CS and other types of asphalt pavements. With aging, some mechanisms, such as oxidation of asphalt and changes in the chemical composition may affect the rheological properties of bitumen. In general, it is possible that aging in asphalt results in hardening, which can contribute to distresses on pavements (HUANG *et al.*, 1996). Thus, most of the service life of pavements is associated with the ability of asphalt binders to resist aging.

Environmental elements must be considered in the study of aging of asphalt materials. Heating is one of the factors that most impact service life of pavements. For example, asphalt mixtures that need to be heated before construction can change its properties in this phase. This is commonly defined as short-term aging. Alternatively, long-term aging is related to service life of mixtures. The Rolling Thin Film Oven Test (RTFOT) simulates hardening which asphalt binder undergoes during mixing and compaction (AIREY, 2003). The test makes use of thin films (~1.25 mm) of bitumen at high temperature (163 °C).

The Pressure Aging Vessel (PAV) procedure is often used to evaluate long-term aging. In this method, a 140 mm pan is used in which bitumen is placed at high temperatures (up to 100° C) and pressurised with air to 2.07 MPa for 20 hours (AIREY, 2003).

Although using high temperatures to reach the characteristics of material after mixing can be accurate, the same might not be used to simulate aging that occurs during its service life. Pavements are susceptible to environmental conditions that are particular for each location,

such as rain seasons, ultraviolet (UV) radiation and temperature. The usual tests to simulate aging frequently do not take into value these factors, especially for coatings such as CS. Furthermore, the temperature applied on the PAV is much higher than the real field temperatures.

In this perspective, there are relevant literature which mention the specific influence of UV aging on asphalt binders using natural aging and/or accelerate aging on a weathering chamber. Oliveira (2015) evaluated aging for binders used in the state of Ceará considering natural exposure. Among the tested samples, there is alternative materials used for priming. FTIR spectroscopy showed increase of compounds such as carbonyls and sulfoxide.

Banja *et al.* (2018) also used FTIR spectroscopy to evaluate changes in the composition of aged material but alternatively used a weathering chamber for that purpose. The analysis was made by evaluating band area and using an index that considered a particular functional group (carbonyl, sulfoxide or aromatic) and CH<sub>2</sub> (1454 cm<sup>-1</sup>) and CH<sub>3</sub> bands areas (1375 cm<sup>-1</sup>). UV radiation was at 340 nm, and time of exposure was of 10 to 250 hours. A decrease on aromatic index showed good relation to initial stages of aging.

Considering that there is no standard for use of weathering aging for asphalt binders, it is important to evaluate how conditions can be constructed to estimate how tests relate to real aging. Several research studies analysed this relation using accelerate methods such as RTFOT and PAV or monitoring natural aging. Nascimento and Faxina (2017) evaluated 5 types of binders comparing weathering aging and PAV resulting in similarities in some samples, depending on the type of modification present in each binder. It was observed that the aging process affected the stiffness of materials, especially at high temperatures.

Crucho *et al.* (2018) presented a method of UV aging which proposes to age materials using weathering chamber. In this method it is suggested that 7 years of pavement performance can be achieved by 30 days of aging for the conditions analysed. In this method it is considered annual solar radiation of each location to determine the test duration. Aging was made by using 280-315 nm UVB lamps and cycles of immersion in water.

## 2.4 Method

In the experimental plan developed in this chapter, three binders were selected for the tests: (i) unmodified emulsion (“RR-2C” type); (ii) polymeric emulsion (“RR-2C-E” type); and (iii) a conventional asphalt binder (50-70 mm of penetration). The classification “RR-2C” is

used as a Brazilian code of reference for cationic rapid-setting emulsions more often applied to CS-type coats. In the state of Ceará, for example, this is the type of emulsion used in most of the constructed treatments. In the polymeric emulsions, the code “E” refers to the addition of polymer in the emulsion. For organization, the Brazilian code is going to be referred to identify the materials used in this paper in the further sections.

It was also previously analysed that these emulsions meet the technical requirements, which are Saybolt-Furol viscosity at 50° C (ASTM D88) between 100 and 400 s and sieving (ASTM D244-09) up to 0.10 %. Also, regarding the conventional binder, this material was selected taking into account features of materials used in the manufacturing of these type of emulsions. In preliminary characterization this binder presented softening point of 48° C and penetration of 49 (0.1 mm) at room temperature (25° C).

#### ***2.4.1 Preparation of samples***

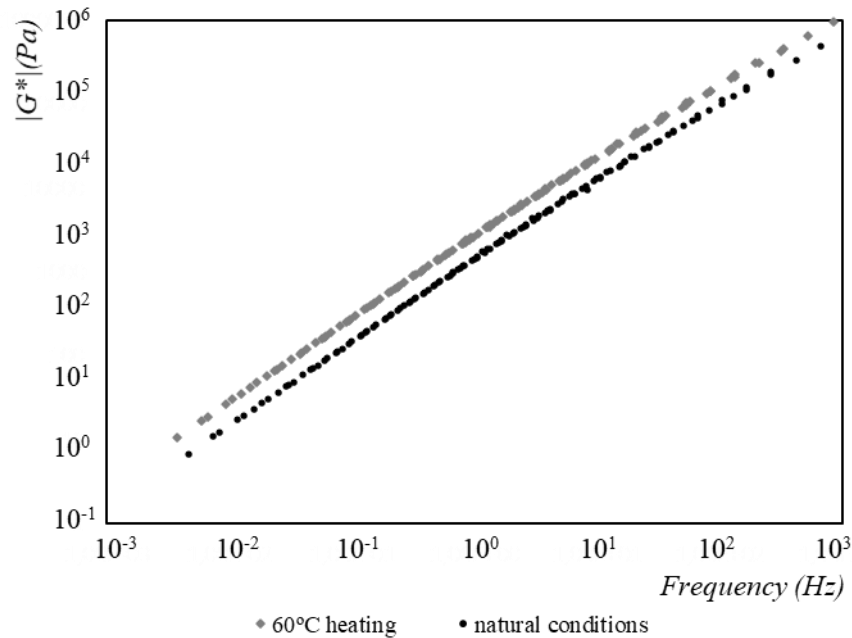
Before exposing materials to the conditions of aging in this study it was defined a procedure exclusively for emulsions to promote the break and cure without inducing aging. This procedure is necessary to submit only the asphalt content of emulsions to further tests.

Thus, to induce the breakup of emulsion and curing of water content, two methods were considered: (i) firstly, using an oven at 60° C for 24 h; and (ii) secondly, using room temperature and natural conditions for 24 h. The first one was based on current performance tests used for CS (LOIOLA, 2009; SILVA, 2018) which commonly make use of an oven before analysis, and the second one aimed to reproduce the conditions that emulsions are subjected to the field. This test was made with the binder of reference used in this research (emulsion RR-2C). For simulating the layer thickness, it was used a rate of emulsion of approximately 1.4 L/m<sup>2</sup>, which is the average rate applied in CS treatments in the state of Ceará.

Sample weights were checked before and after the 24-hour period. Both samples lost the same mass, indicating approximately the same loss in water content. The residual material in both cases corresponded to nearly 67 % of the original mass, which is within the expected asphalt residue for RR-2C emulsions. Frequency sweep testing was done in a DSR, and the master curves of materials were plotted. The analysis of the complex modulus ( $|G^*|$ ) obtained after frequency sweep testing did not provide conclusion that both materials are the same after the two procedures (Figure 5). This result was also in accordance with the discussion proposed by Silva (2018), which indicated that CS specimen presented differences in properties related to performance when tested after these conditions of cure. Thus, it was defined to proceed with

cure at environmental conditions and sunlight to prepare samples aiming to reduce the effects of heating.

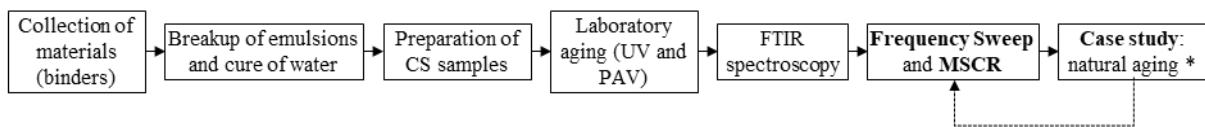
Figure 5 – Analysis of breakup procedures



#### 2.4.2 Experimental plan

The experimental plan developed for this part of research was subdivided into steps as summarized in Figure 6.

Figure 6 – Flow chart of experimental plan (chapter 2)



\* Natural aging of the unmodified emulsion

Source: author (2021)

The first step is the identification and collection of materials. The emulsions and the asphalt binder were obtained from the same supplier in the state of Ceará, where this research was developed. The following step refers to sample preparation. As described previously, it was chosen to carry the cure of emulsion at environmental conditions. Figure 7 shows one of the samples. A plastic film was also used to cover samples after this documentation to avoid contamination.

Figure 7 – Demonstrative of sample curing at environmental conditions



Source: author (2021)

Before the samples were submitted to the procedure of aging, the Fourier-Transform Infrared Spectroscopy (FTIR) was carried out. This test was preliminarily conducted to the residues immediately after the 24-hour period of cure and to the asphalt binder in its natural form after collection. After this procedure, the residues of emulsion and the asphalt binder were treated similarly in the following tests. For organization purposes, in this paper these three materials will be referred as “binder”.

The next step refers to the mechanism of aging applied to the binders. Two methods were considered: (i) firstly, the use of ultraviolet (UV) light and, (ii) secondly, the use of Pressure Aging Vessel (PAV) for aging. The second method is the one conventionally used for that purpose and it is described in the standard ASTM D6521 - 19 (ASTM, 2019).

In relation to the UV aging method, there are plenty variations of how to carry out its procedure. Based on that, a test routine was created specifically for this research. A weathering chamber test was used. The test parameters were defined as: frequency of 300-400 nm; temperature of 70° C (in the specimen); and room temperature of 35° C. These parameters were set based on the average annual climate conditions of the location where the research was developed and the functionality of the chamber. Also, it was made the monitoring of humidity level in the chamber at 50 %. It was chosen to carry out the experiment without cycles of water spraying, focusing only on the effects of UV incidence. Samples were identified using the classification presented in Table 1. Figure 8 shows the arrangement of the weathering chamber to the process of UV induced aging. Figure 8a presents the first display, in which it is shown

the actual values of parameters and settings. In Figure 8b, there is the second display, with time and total dosage of test (kJ/m<sup>2</sup>). Figure 8c shows two of the tested specimens inside the chamber.

Figure 8 – Weathering chamber used for UV induced aging



Source: author (2021)

Table 1 – Description of samples tested

<i>Sample</i>	<i>Description</i>
T0	Sample immediately after 24 h resting in natural conditions (sunlight) or in its natural form
T24	Sample after 24 h of UV aging
T48	Sample after 48 h of UV aging
T120	Sample after 120 h of UV aging
T240	Sample after 240 h of UV aging
TPAV	Sample after submitted to PAV

Source: author (2021)

Lastly a case study was developed: the unmodified emulsion was exposed to natural aging under the same environmental conditions used in the process of break and cure of the residue. This material was aged for 15 and 30 days and the same tests used to evaluate the materials aged following the other methods was used. The natural aging was compared to the UV-induced aging in order to propose a correlation between the two methods.

FTIR was performed for each aging duration and the results were compared with the material prior to aging. This test was used to investigate the level of aging of each material. After the proceedings of aging, frequency sweep testing was carried out. Also, each sample was tested for MSCR, considering this test is used to estimate failure in CS concerning the distress



of bleeding. The following sections discuss the results obtained with the tests defined in this experimental plan.

## 2.5 Results

### 2.5.1 FTIR spectroscopy

Results of FTIR spectroscopy show that there is a noticeable change in the material after the UV aging process and after PAV. It is important to consider that after UV induced aging the surface of binder film presented a more aged aspect than the inner part of the sample. To avoid problems when comparing samples, the content of specimens was mixed and homogenized after aging. However, it should be relevant to evaluate separately the composition of the surface of these materials.

Figure 9 presents curves of absorbance of the three binders at four stages: (i) natural, (ii) after 120 hours of UV aging, (iii) after 240 hours of UV aging and (iv) after PAV. Figure 9a presents the spectroscopy for the samples of the conventional asphalt binder. In the top of the graph, it is shown the curve of conventional aged material (TPAV). Following the order, it is shown the materials T240 and T120, respectively, and T0, in the bottom. There was an increase in the concentration of functional groups of products of oxidation and aging such as carbonyls ( $1700\text{ cm}^{-1}$ ), sulfoxides ( $1032\text{ cm}^{-1}$ ) and aromatics ( $1600\text{ cm}^{-1}$ ). The band area was used to evaluate the formation of these products. The TPAV sample presented the highest areas among the four samples. Although less intense, the UV aged materials (T240 and T120) also presented a tendency of formation on these bands.

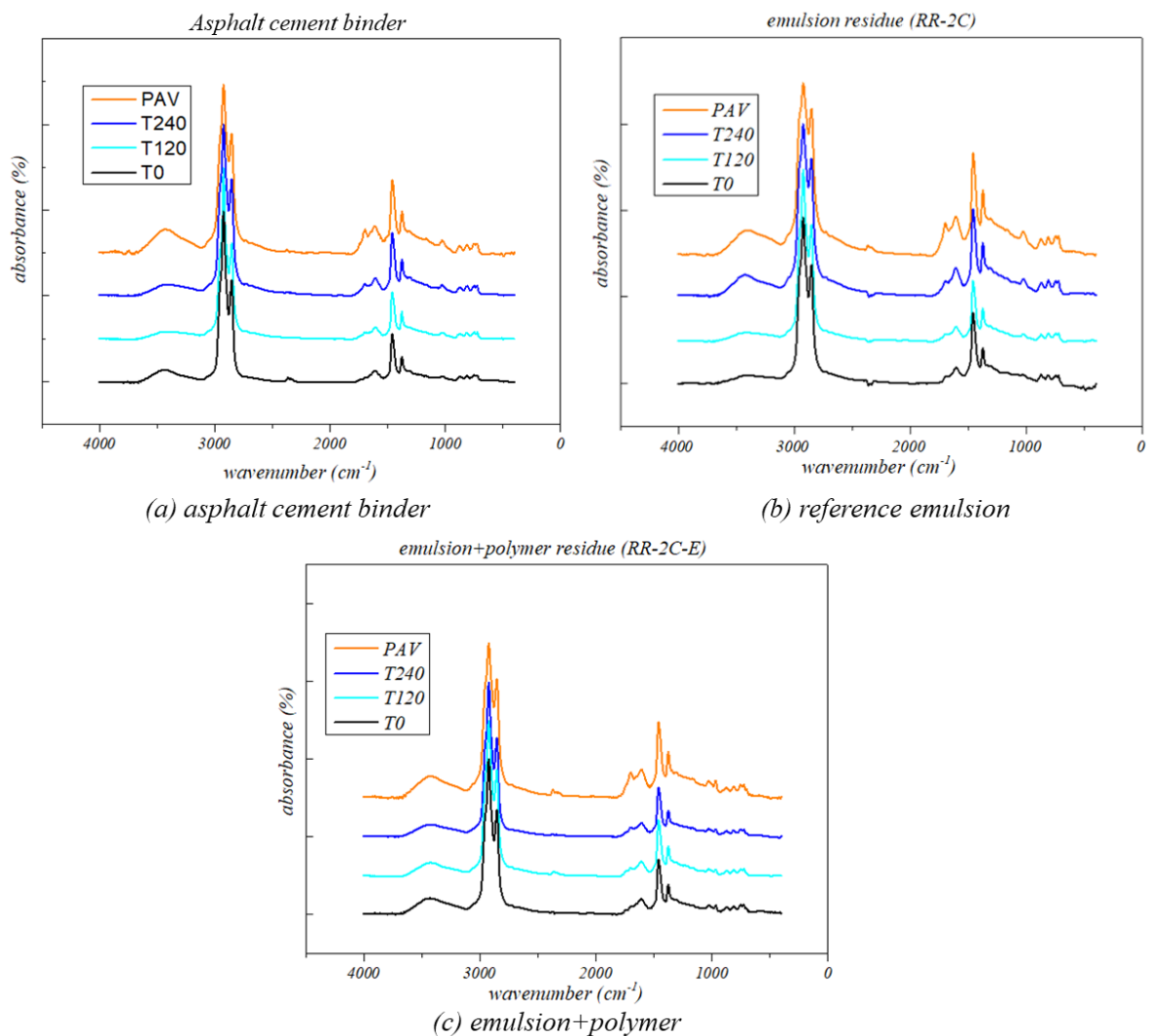
In order to enhance the evaluation of formation of bands, it was calculated an index of carbonyls ( $I_c$ ), sulfoxide ( $I_s$ ) and aromatic ( $I_{aro}$ ) in the samples (BANJA *et al.*, 2018). Based on these parameters, the formation of sulfoxide in T120 was near to T240 and TPAV, considering the increase of  $I_s$  was approximately 0.01. In contrast, there is indication that the formation of carbonyls is more intense in the TPAV sample, as shown by the increase of  $I_c$  from 0.00 to 0.12. The indexes calculated are summarized in Table 2.

In relation to the residue of the emulsions, the unmodified emulsion RR-2C presented formation of bands similar to the asphalt binder. The curves of spectroscopy are presented in Figure 9b. There is more indication of formation of carbonyls in the TPAV sample than in the other samples. In fact, the index  $I_c$  was 0.14 in this sample and around 0.03 in the others. It is possible to highlight that this index presented its highest values in the sample TPAV both in the asphalt binder and in the residue of emulsion RR-2C. This parameter also varied only 0.02

between the two binders. In relation to  $I_s$ , there is a linear tendency of growth between the samples, increasing from 0.02 to 0.08.

The spectroscopy of the residue of the polymeric emulsion presents formation of functional groups created in the oxidation of binders when both methods of aging were considered. In relation to the indexes, the growth of  $I_c$  was also more intense when the sample was subjected to PAV, although in different proportion of what was obtained in the other binders. In addition, there is indication of formation of a band in  $966\text{ cm}^{-1}$ , resulting from the presence of polymer in this sample.

Figure 9 – FTIR spectroscopy of samples



Source: author (2021)

Table 2 – Carbonyls, Sulfoxides and Aromatics indexes

	<i>asphalt cement binder</i>				<i>RR-2C</i>				<i>RR-2C-E (pol.)</i>			
	<i>T0</i>	<i>T120</i>	<i>T240</i>	<i>PAV</i>	<i>T0</i>	<i>T120</i>	<i>T240</i>	<i>PAV</i>	<i>T0</i>	<i>T120</i>	<i>T240</i>	<i>PAV</i>
<i>I<sub>c</sub></i>	0.00	0.03	0.05	0.12	0.03	0.03	0.03	0.14	0.02	0.02	0.04	0.09
<i>I<sub>s</sub></i>	0.04	0.05	0.06	0.07	0.02	0.04	0.06	0.08	0.05	0.04	0.05	0.06
<i>I<sub>ARO</sub></i>	0.24	0.23	0.24	0.30	0.10	0.13	0.14	0.16	0.25	0.24	0.23	0.28

Source: author (2021)

Based on both the spectroscopy and the indexes  $I_c$ ,  $I_s$  and  $I_{aro}$  calculated and summarized in Table 2, there is not an indication that the mechanisms involved in both the PAV aging and the proposed UV induced aging are similar. After 240 hours of UV aging, the samples did not present similarities to the conventional method in relation to these parameters. The index of carbonyls ( $I_c$ ) increased more strongly after PAV aging in all samples, which can be related to the conditions which the samples are submitted to and the intensity of this method of aging compared to the others. This method of aging is commonly used to simulate in laboratory the condition of binders in the end of service life. Considering the particularities of CS pavements, this stage possibly is reached in different conditions from other asphalt layers. It is expected that this stage is reached faster, considering the area of exposure significantly superior to ordinary wearing courses.

The following topics discuss about the impacts of aging in the complex modulus ( $|G^*|$ ) and in the non-recoverable compliance ( $J_{nr}$ ) of materials. Also, it is made a comparison between the results obtained in the stages of aging using UV and the binder after PAV procedure.

## 2.5.2 Analysis of Complex Modulus

### 2.5.2.1 Unmodified emulsion (RR-2C)

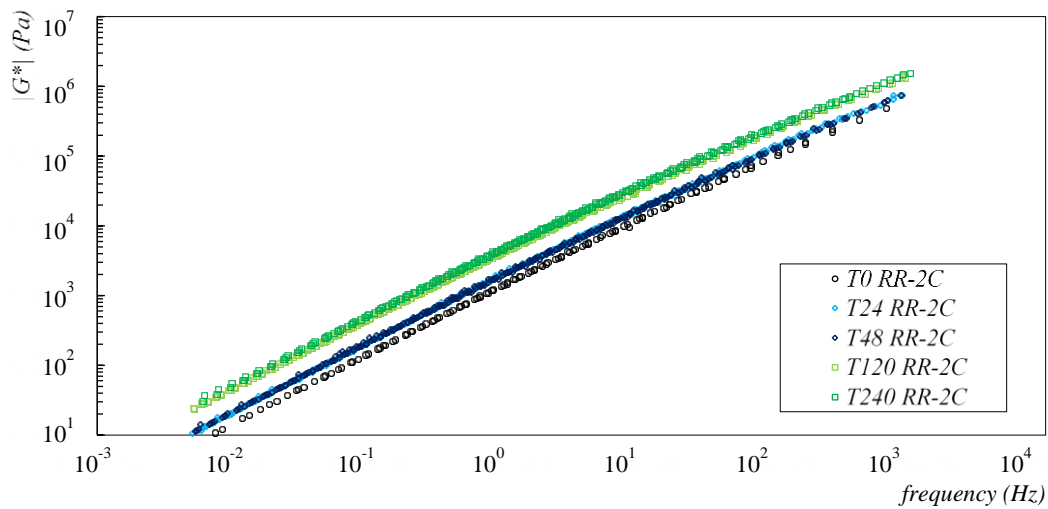
Figure 10 shows master curves of complex modulus ( $|G^*|$ ) obtained in frequency sweep tests of binder at the stages of aging analysed. The curves were plotted for a reference temperature of 60° C. Figure 10a presents curves of samples after UV induced aging from 0h to 240 h and Figure 10b compares the stiffness of the most aged sample in this method to PAV aging. The residue obtained after the breaking of the emulsion is presented in both graphs as T0.

In relation to  $|G^*|$ , it has occurred a displacement of the curves of the aged samples in upward direction, which means an increase in stiffness. Analysing T24 and T48, there was a higher displacement of the curve in the first 24 hours of aging than in the next 24 hours. A

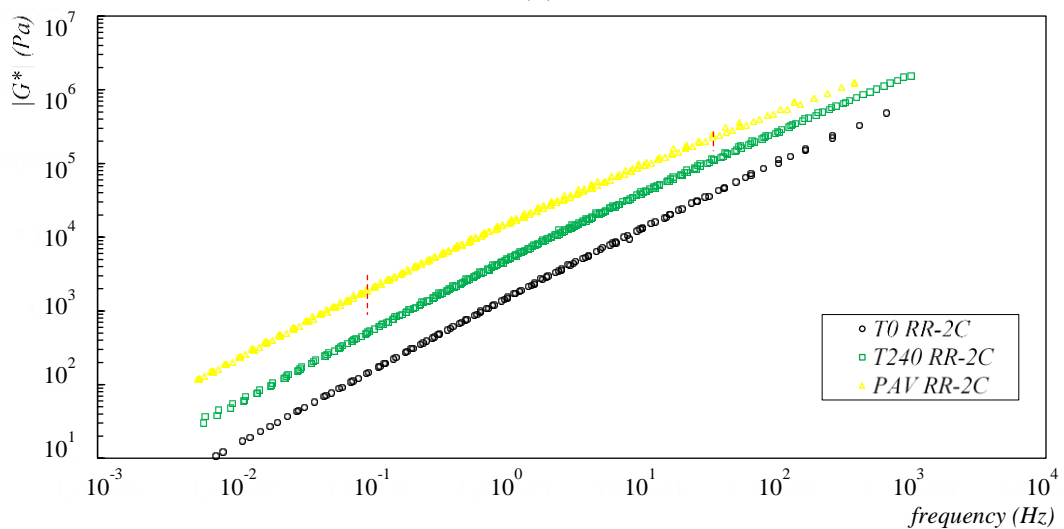
similar behaviour was obtained comparing the first and the next 120 hours of test considering the displacement between T0 and T120 was stronger than between T120 and T240. The curve T240 presented, as expected, higher displacement among the UV aged samples.

The PAV aged sample presented a higher displacement when compared to the binder after 240 hours of UV induced aging. Thus, there is indication that the mechanism of aging used in the PAV procedure is more intense than 240 hours of the UV procedure analysed. In addition, the increase of  $|G^*|$  was more intense in lower frequencies than in the higher frequencies, which indicates that the increase of stiffness is more intense when it comes to high temperatures.

Figure 10 – Master curves of  $|G^*|$  of residue after aging (RR-2C)



(a)



(b)

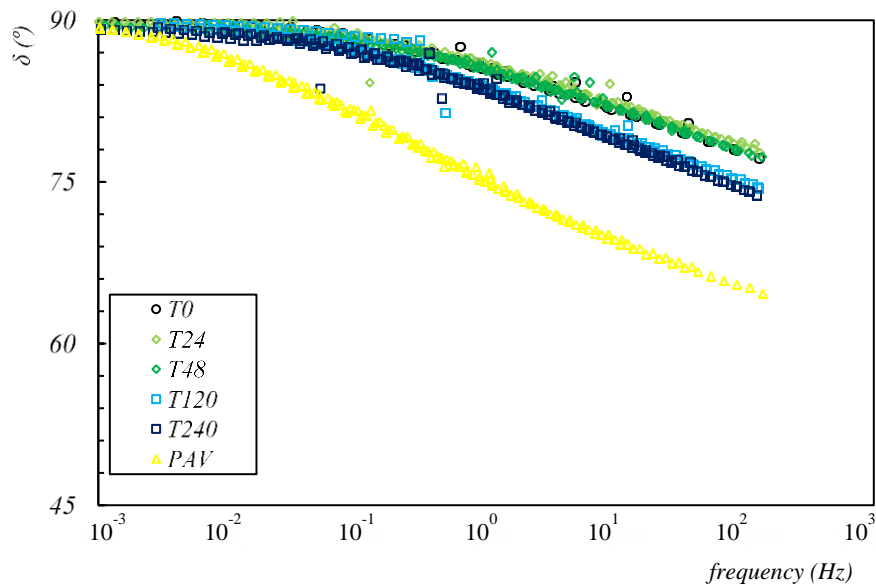
Source: author (2021)

To evaluate the average displacements and the aspect of curves, it was considered the value of  $|G^*|$  in the frequencies of 2 Hz, 4 Hz and 10 Hz. Higher frequencies (20 Hz and 50 Hz) were also considered in the evaluation of increase of stiffness. In relation to the T0, the increase of  $|G^*|$  in T24 was average 39 % for the mentioned frequencies. Between T0 and T120, the increment of  $|G^*|$  was around 180 %. In T240 this value was 212 %. This consideration corroborates the idea that the evolution of hardening due to UV induced aging is more intense in the first days.

When it comes to PAV aging the increase of stiffness was around 735 %. However, this value was considerably variable depending on the frequency. For example, for frequencies of 20 Hz and 50 Hz the increase of  $|G^*|$  was, respectively, 526 % and 447 %. Alternatively, for the frequency of 2 Hz the increase was 845 %. This sample presented the highest disparity in this value, which might be related to the mechanism applied in this method.

Figure 11 presents the master curves of phase angle ( $\delta$ ). The higher reduction of the phase angle, particularly in the sample tested in PAV indicates an increase in the elastic behaviour of the sample. This is probably related to the evolution of products of oxidation such as carbonyls.

Figure 11 - Master curves of  $\delta$  of residue after aging (RR-2C)



Source: author (2021)

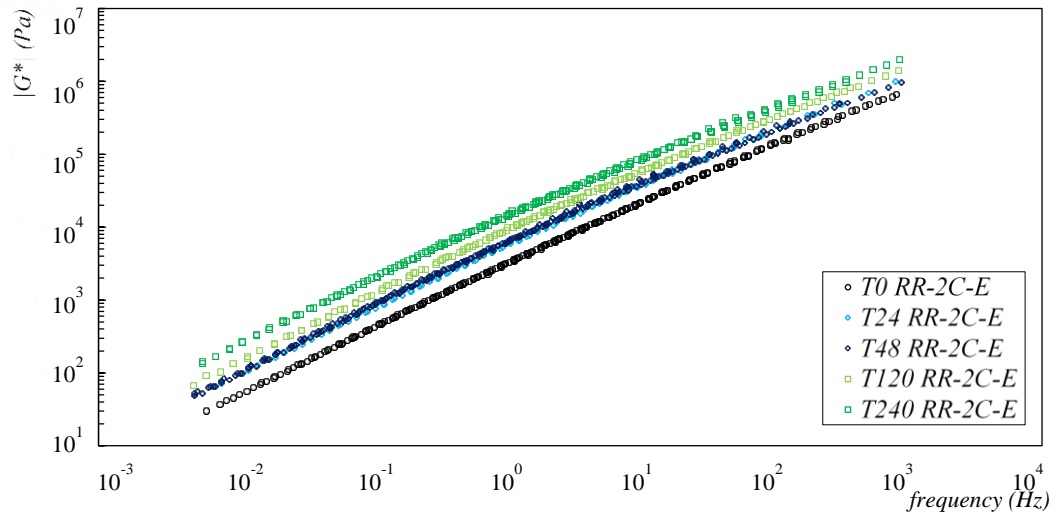
#### 2.5.2.2 Polymeric Emulsion (RR-2C-E)

The master curves of  $|G^*|$  of the emulsion residue modified with polymer presented the tendency of moving in the direction of higher stiffness after UV induced aging, as expected.

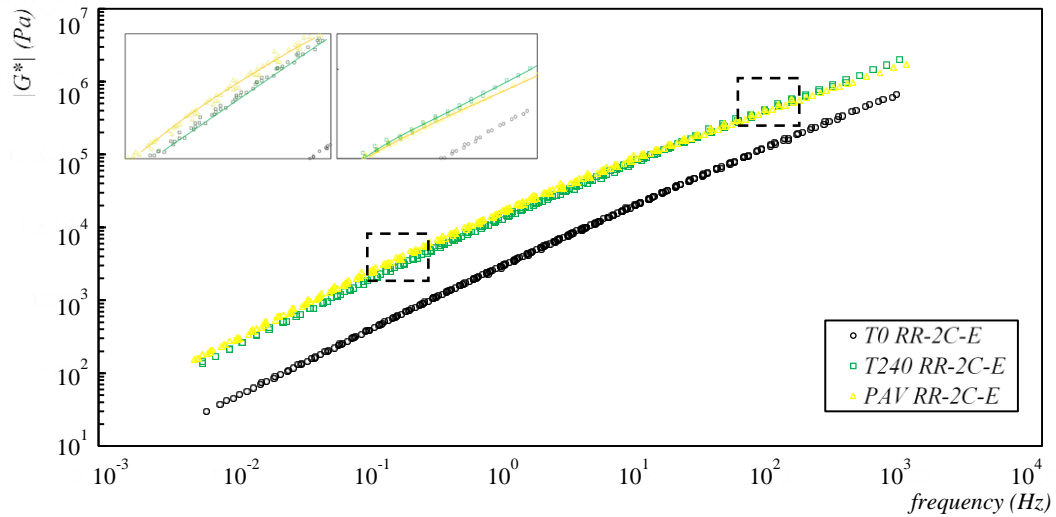
Figure 12 shows the master curves obtained with this material. Figure 12a presents the curves obtained for the material during UV induced aging between 0 and 240 hours (T0, T24, T48, T120 and T240). The displacement of the curve was evident after the first 24 hours of test. Considering the frequencies of 2 Hz, 4 Hz and 10 Hz the increment of  $|G^*|$  was around 172% in relation to the residue of emulsion after cure. However, little disparity was observed between T24 and T48, similar to the behaviour obtained with the conventional emulsion.

The average of increment of stiffness in T120 and in T240 were, respectively, 280 % and 410 %. This percentage indicates that, compared to the conventional emulsion, the second half of the aging process presented considerable contribution to hardening, possibly due to the activity of polymer inside the residue. In fact, comparing the conventional and the polymeric emulsions, the displacement between the curves T120 and T240 was higher in the second one.

The master curve of the PAV aged material (Figure 12b) presented stronger displacements for lower frequencies, which was also noticed in the residue of the conventional emulsion. In contrast, there is no indication that the material after PAV presented a higher increase of stiffness than after 240 hours of UV induced aging. In fact, for frequencies higher than 20 Hz, the curve T240 presents a tendency of going beyond and exhibit higher  $|G^*|$ , as shown in Figure 12b. Based on this perspective, it is possible to infer that the process of aging in the polymer might evolve differently from what is observed in asphalt materials, and that this might reflect in the aspect of the polymeric emulsion.

Figure 12 - Master curves of  $|G^*|$  of polymeric residue after aging (RR-2C-E)

(a)



(b)

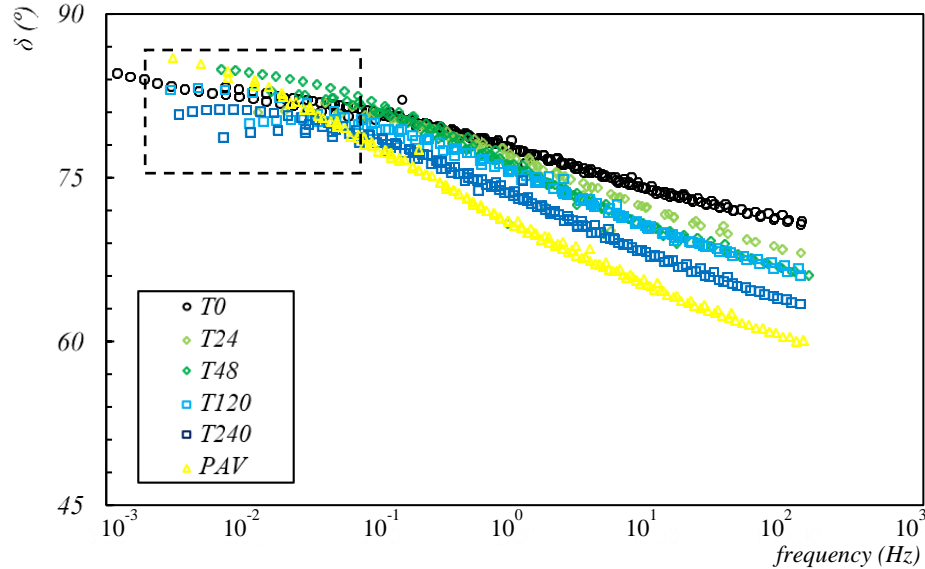
Source: author (2021)

Comparing PAV aging of the two residues of emulsion, there was significant effects in the stiffness. While there was average increase of 384 % of the complex modulus in the polymeric emulsion, this value was around 735 % in the unmodified emulsion considering the same frequencies. The explanation for this difference could be the activity of polymer inside the binder in materials of this type.

Similar behaviour was also observed in the phase angle ( $\delta$ ). Bringel (2007) suggested that one common improvement of asphalts modified with the addition of polymer (SBS) would be the improvement of elasticity. It was also observed that binders of this type presented elastic behaviour at high temperatures. Figure 13 presents the master curves of  $\delta$  of the polymeric residue. There was also tendency of decrease of the phase angle in this sample. The curves of

this residue presented an inconsistent behaviour in lower frequencies, possibly due to the content of polymer in the binder.

Figure 13 – Master curves of  $\delta$  of polymeric residue after aging (RR-2C-E)



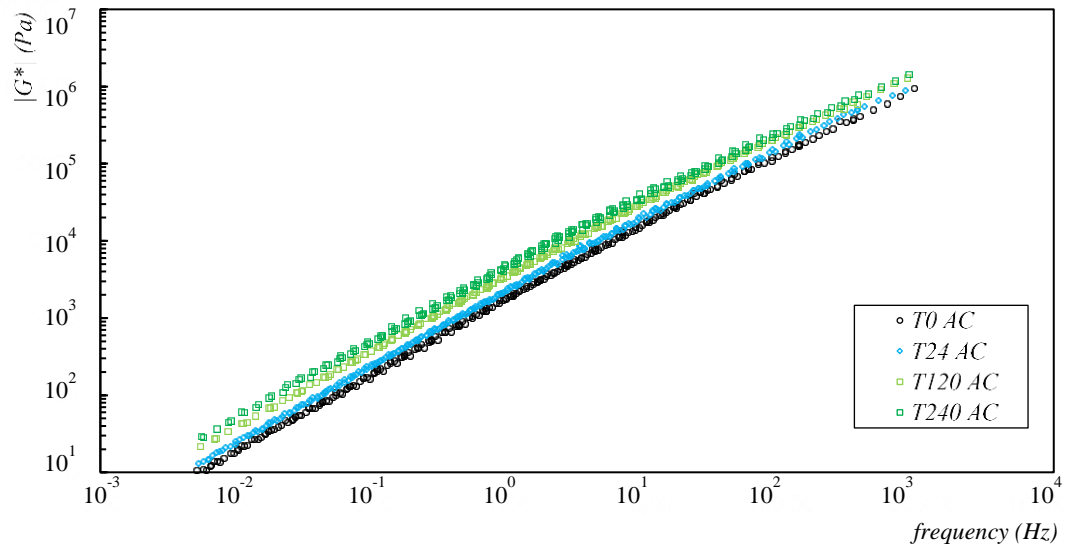
Source: author (2021)

### 2.5.2.3 Asphalt binder

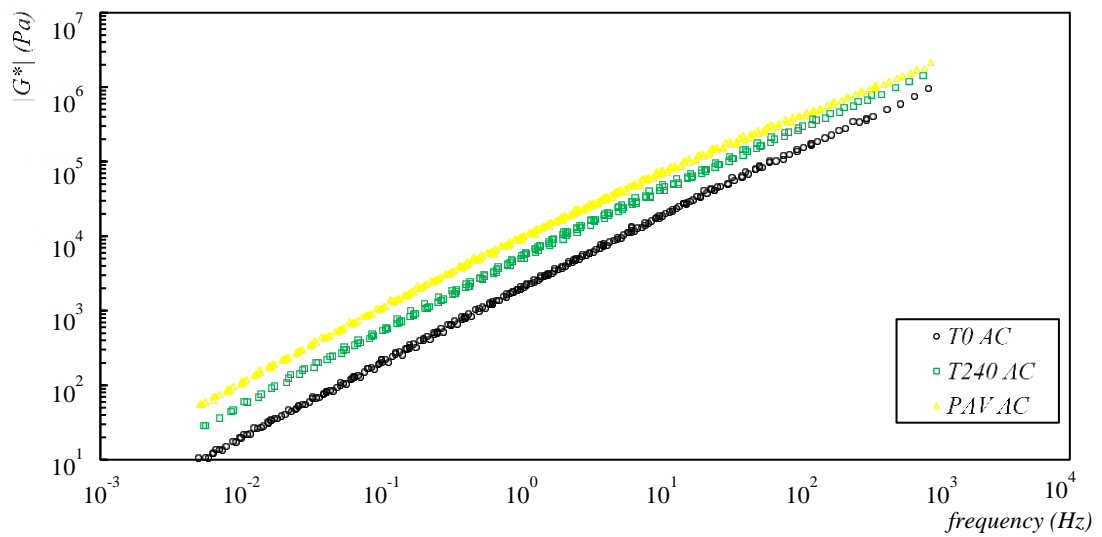
The third binder tested was the conventional asphalt binder. This material presented behaviour in some respects similar to the residue of the unmodified emulsion. Figure 14 presents master curves of complex modulus ( $|G^*|$ ) for this binder. In Figure 14a it is shown curves of samples tested during 240 hours of UV induced aging (T24, T120 and T240). The increment of stiffness in the first 24 hours of test was around 30 % analysing the  $|G^*|$  corresponding to frequencies in the range of 2 to 10 Hz. The maximum increase in the value of complex modulus was observed in T240, and it was average 315 % for the analysed frequencies.

Figure 14b shows the master curves of complex modulus of T0, T240 and TPAV. The stiffness of the sample obtained after PAV aging was higher among all the samples analysed. The increase of stiffness is stronger in lower frequencies.



Figure 14 – Master curve of  $|G^*|$  of asphalt cement after aging

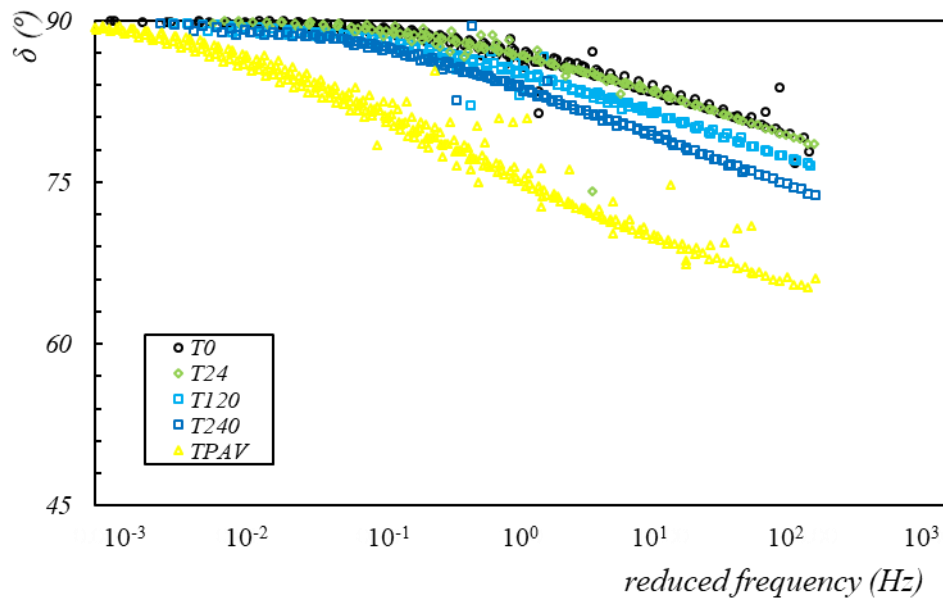
(a)



(b)

Source: author (2021)

Figure 15 presents master curves of phase angle ( $\delta$ ) of the asphalt cement. There is tendency of reduction of the phase angle, and consequently increase of elasticity of the asphalt cement similar to what was observed in the unmodified emulsion. As expected, the PAV aging developed the higher reduction in the phase angle.

Figure 15 – Master curves of  $\delta$  of asphalt cement after aging

Source: author (2021)

In general, samples tested after PAV induced aging presented the most significant reduction in the phase angle and consequently increment of elasticity. Also, the stiffness of these samples enhanced considerably. Regarding to lower frequencies this enhancement was even stronger, affecting the performance of binders at high temperatures. This behaviour is possibly related to the mechanism of the test which aims to simulate the end of service life.

### 2.5.3 MSCR $\times$ bleeding

The MSCR test was performed in the samples of emulsion residues after the cure of water. Table 3 summarizes the results obtained in these samples. The range of temperature used in this test was between 52 and 70° C in intervals of 6° C. The conventional emulsion (RR-2C) presented low recovery. In this sample, the maximum R obtained was 6.28 %, when the temperature was 52° C and at the stress level of 0,1 kPa. In contrast, the polymeric emulsion (RR-2C-E) presented maximum R of 52.96 % at the same conditions. Also, the recovery decreased as the temperature increased in both samples. The non-recoverable compliance presented opposite tendency. The  $J_{nr}$  increased in higher temperatures in both samples. Also, this value was superior in the conventional emulsion.

Table 3 – MSCR of the emulsion residues (52-70° C)

<i>T</i> (°C)	<i>stress</i>	<i>RR-2C</i>				<i>RR-2C-E (polymeric)</i>			
		<i>R</i> (%)	<i>J<sub>nr</sub></i> (kPa <sup>-1</sup> )	<i>R<sub>diff</sub></i> (%)	<i>J<sub>nr diff</sub></i> (%)	<i>R</i> (%)	<i>J<sub>nr</sub></i> (kPa <sup>-1</sup> )	<i>R<sub>diff</sub></i> (%)	<i>J<sub>nr diff</sub></i> (%)
52	0.1 kPa	6.28	1.14	66.9	8.2	52.69	0.25	49.11	73.83
52	3.2 kPa	2.08	1.23			26.82	0.44		
58	0.1 kPa	4.61	3.01	88.8	20.3	43.16	0.67	64.80	76.69
58	3.2 kPa	0.52	3.63			15.19	1.19		
64	0.1 kPa	1.72	8.23	91.4	2	32.52	1.78	77.66	59.74
64	3.2 kPa	0.15	8.39			7.27	2.85		
70	0.1 kPa	1.18	16.75	104.1	7.4	22.89	4.15	87.75	50.51
70	3.2 kPa	-0.05	17.99			2.80	6.24		

Source: author (2021)

Considering CS type coats, the parameter  $J_{nr}$  is often used in correlation to the effects of bleeding in the pavement (ADAMS, 2014; KIM *et al.*, 2017). This relation is represented in a criterion of maximum  $J_{nr}$  to accept the emulsion for service, based on the traffic of project. In this perspective, the RR-2C-E emulsion would present best performance in relation to this distress, and it would be appropriate to low traffic roads, since the parameter was below the criterion of 8 kPa<sup>-1</sup> in all temperatures evaluated. Considering temperatures up to 64° C the  $J_{nr}$  would attend the criterion of 3.25 kPa<sup>-1</sup>, for high traffic roads. Considering the RR-2C emulsion, the criterion would be attended only for temperatures below 58° C.

For effects of analysis, the evolution of the parameter  $J_{nr}$  was analysed after induced aging (PAV and UV) at the temperatures of 58, 64 and 70° C. The parameter was measured in the mentioned temperatures at the stress of 3.2 kPa and evaluated in relation to the type of method. The results were organized for each of the three binders analysed.

#### 2.5.3.1 $J_{nr}$ of unmodified emulsion (RR-2C)

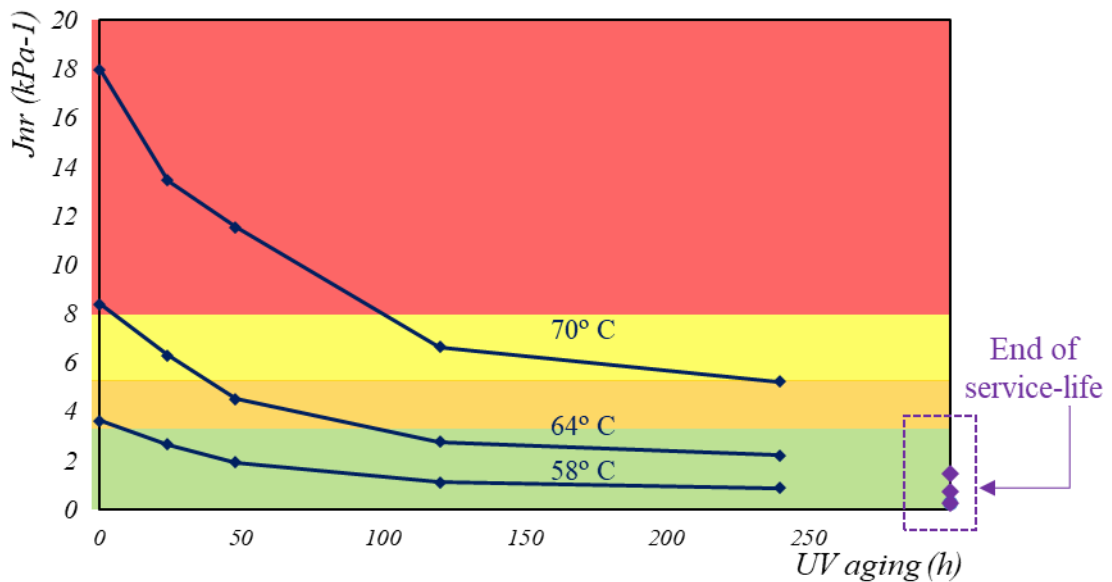
Table 4 presents values of  $J_{nr}$  for the unmodified emulsion. Before aging, the residue presented  $J_{nr}$  superior to the maximum limits for good performance at temperatures over 64° C. However, this parameter decreased with aging time. For example, after 120 hours of UV induced aging, the binder would attend the recommendation for low traffic roads for the considered conditions. The sample tested in PAV presented lower values of  $J_{nr}$  than the others. In fact, the binder attends to the criterion for high volume roads, since all considered values were inferior to 3.25 kPa<sup>-1</sup>. Figure 16 summarizes the results in a graph with the decrease of  $J_{nr}$  in each sample. The results obtained in the sample tested in PAV are identified as the end of service life. There is a tendency outlined by the measured values of  $J_{nr}$  in which the decrease is more intense closer to the beginning. Based on that, all curves would be out of the failing zone around 100 hours.

Table 4 –  $J_{nr}$  of the residue after induced aging (RR-2C)

	<i>RR-2C</i>					
	<i>T0</i>	<i>T24</i>	<i>T48</i>	<i>T120</i>	<i>T240</i>	<i>TPAV</i>
58 °C	3.63	2.65	1.93	1.10	0.86	0.25
64 °C	8.39	6.30	4.52	2.77	2.20	0.73
70 °C	17.99	13.45	11.52	6.63	5.23	1.44

■ High traffic      ■ Low traffic  
■ Medium traffic      ■ Fail

Source: author (2021)

Figure 16 – Aging curves of  $J_{nr}$  (unmodified emulsion – RR-2C)

Source: author (2021)

### 2.5.3.2 $J_{nr}$ of polymeric emulsion (RR-2C-E)

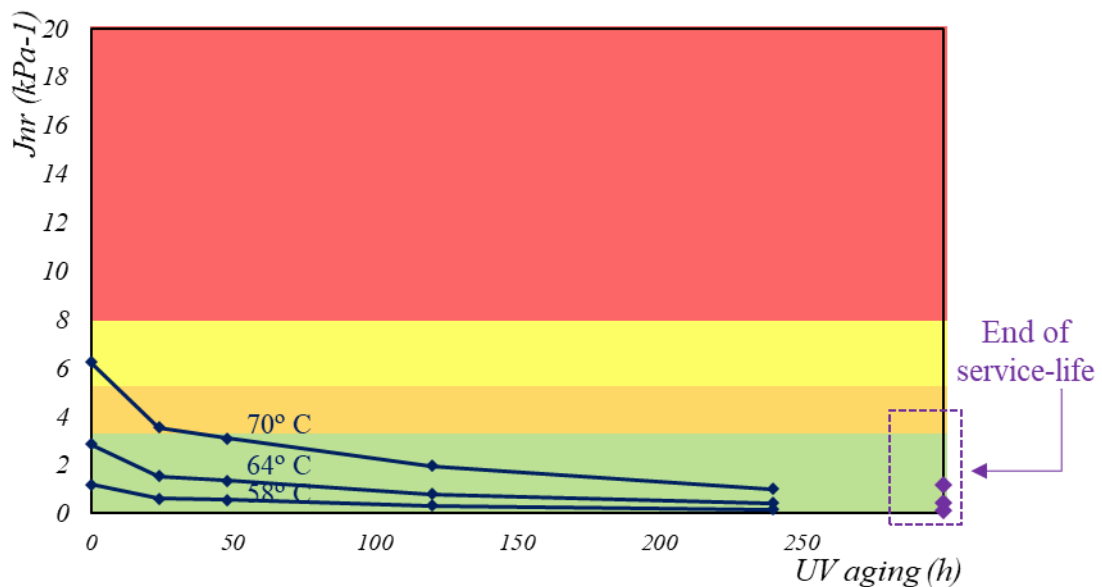
Differently from what was obtained in the tests with unmodified emulsion, the polymeric emulsion presented values under the limit for low traffic in all tests. However, similarly there was a tendency of drop of  $J_{nr}$  as the aging methods were proceed. Table 5 and Figure 17 present the values obtained in the tests of MSCR. All samples after 48 hours of UV aging presented  $J_{nr}$  lower than 3.25 kPa<sup>-1</sup>, corresponding to the limit for high volume roads.

Table 5 -  $J_{nr}$  of the polymeric residue after induced aging (RR-2C-E)

	<b>RR-2C (pol)</b>					
	<i>T0</i>	<i>T24</i>	<i>T48</i>	<i>T120</i>	<i>T240</i>	<i>TPAV</i>
58 °C	1.19	0.61	0.55	0.31	0.17	0.14
64 °C	2.85	1.53	1.36	0.79	0.42	0.44
70 °C	6.24	3.54	3.08	1.95	1.00	1.18

■ High traffic                      ■ Low traffic  
■ Medium traffic                      ■ Fail

Source: author (2021)

Figure 17 – Aging curves of  $J_{nr}$  (polymeric emulsion – RR-2C-E)

Source: author (2021)

### 2.5.3.3 $J_{nr}$ of asphalt cement binder

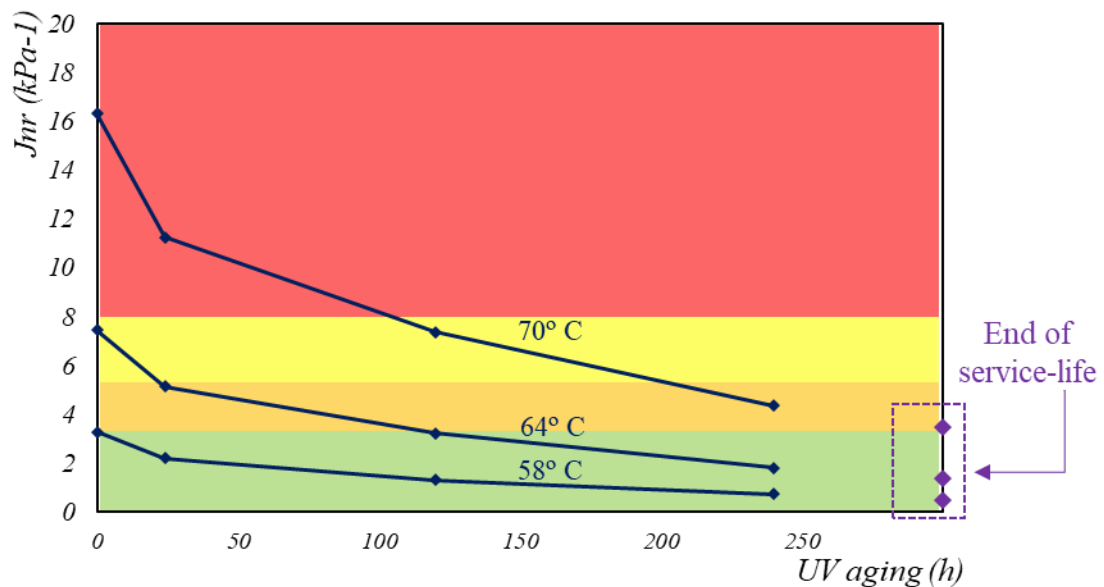
Table 6 summarizes the results of  $J_{nr}$  obtained in tests of MSCR on the asphalt cement binder. The evolution of  $J_{nr}$  observed in this material was similar to the unmodified emulsion. After approximately 100 hours of test, in both samples, this parameter reaches the limit established for low traffic roads in the most critical temperature, considering the curves of  $J_{nr}$  presented in Figure 18.

Table 6 –  $J_{nr}$  of asphalt cement after induced aging

	<i>Asphalt Cement</i>				
	<i>T0</i>	<i>T24</i>	<i>T120</i>	<i>T240</i>	<i>TPAV</i>
58 °C	3.27	2.20	1.32	0.73	0.49
64 °C	7.44	5.13	3.24	1.81	1.38
70 °C	16.30	11.24	7.36	4.37	3.48

■ High traffic      ■ Low traffic  
■ Medium traffic      ■ Fail

Source: author (2021)

Figure 18 – Aging curves of  $J_{nr}$  (asphalt cement)

Source: author (2021)

In the Northeast Region of Brazil pavements temperatures easily exceed 64° C. In this perspective, CS pavements constructed with unmodified emulsions would present poor bleeding performance. This criterion should be considered when selecting emulsions given the prevailing climatic configuration in locations with tropical climates. In addition, it is important to investigate how this is reflected in CS constructed by different specifications, especially when it comes to different type of emulsions. Taking into consideration that the relation between  $J_{nr}$  and bleeding is mostly based on correlation, it is possible that the limits for each level of traffic need be calibrated. Also, the analysis of  $J_{nr}$  curves had the assumption that the relation between this parameter and the distress of bleeding is sustained through time. Thus, it would be relevant to evaluate the evolution of this distress in road tests with controlled traffic.

In all tests, there was a tendency of  $J_{nr}$  decrease with time. The outline of this tendency could be well adjusted to an exponential behaviour. The R-square calculated using exponential trendlines were near 0.9 in all tests. The use of this type of model would be significant to estimate  $J_{nr}$  and possibly bleeding through service life of binders considering a relation between UV induced aging and natural aging.

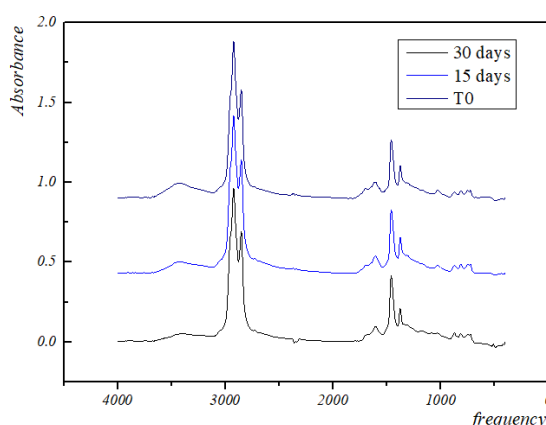
#### 2.5.4 Case study: natural aging of the unmodified emulsion

In the evaluation of natural aging in environmental conditions two samples of residue of the unmodified emulsion were tested aged for 15 and 30 days. FTIR spectroscopy, frequency sweep and MSCR tests were used to compare the evolution of aging parameters between natural aging and UV-induced aging using weathering chamber. The samples were aged between the months of August and September and no precipitation was registered.

##### 2.5.4.1 FTIR

Figure 19 presents the curves of absorbance obtained in the FTIR spectroscopy of the analysed samples. Results show that there is increase in the formation of sulfoxides after the two periods of aging. In relation to carbonyls and aromatics the tendency of growth was lower. The indexes  $I_c$ ,  $I_s$  and  $I_{Aro}$  were calculated and compared to the value of the indexes calculated for the unaged sample (T0). Table 7 presents the indexes calculated for T0 and for the aged samples.

Figure 19 - FTIR spectroscopy after natural aging (15 and 30 days)



Source: author (2021)

The index of sulfoxide ( $I_s$ ) presented a constant tendency of growth. After 15 days, this parameter increased 0.02 while, after 30 days, increased 0.04. This result suggests that the increase of  $I_s$  follows a linear correlation. In relation to the other indexes a different behaviour was observed. The index of carbonyls ( $I_c$ ) decreased after 15 days and increased after 30 days.

When it comes to the index of aromatics ( $I_{Aro}$ ) a decrease was observed between 15 and 30 days.

Table 7 - FTIR indexes after natural aging (15 and 30 days)

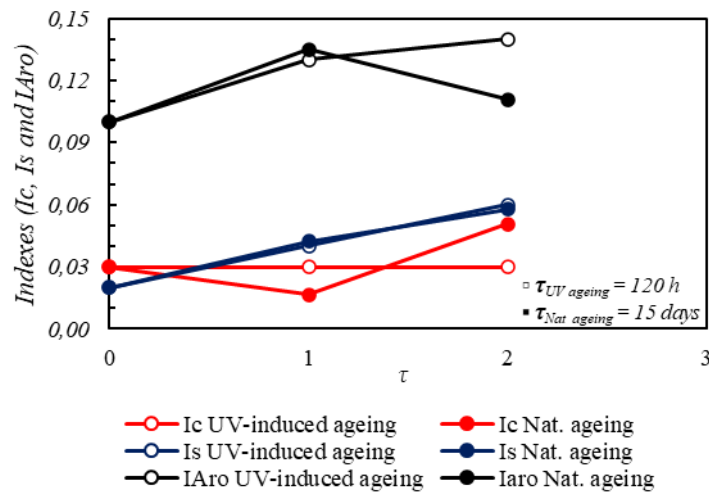
<i>Indexes</i>	<i>0 days</i>	<i>15 days</i>	<i>30 days</i>
$I_c$	0.03	0.02	0.05
$I_s$	0.02	0.04	0.06
$I_{Aro}$	0.10	0.13	0.11

Source: author (2021)

The variation tendency of the FTIR indexes obtained in the analysis of the UV-induced aging was compared to the behaviour obtained in the parameters of the natural aged samples. Figure 20 presents the comparison between the results obtained in each test in the perspective of each index. In this graph, the x-axis is the time constant ( $\tau$ ) that vary depending on the time spent in cycles of each aging method: for the UV-induced aging,  $1\tau = 120$  hours, while for the natural aging,  $1\tau = 15$  days.

Considering each index individually, the  $I_s$  presented the best approximation when correlating the two aging methods. In both analysis the increase of this parameter was nearly constant. In addition, considering the  $\tau$  defined, the rate of growth was strongly similar, suggesting that, after 120 hours of UV-aging, the formation of sulfoxides was approximately the same that after 15 days of natural aging. However, the same relation was not observed when analysing  $I_c$  and  $I_{Aro}$ . Concerning these indexes, the divergence of results was stronger when considering  $2\tau$ .

Figure 20 - Comparison between FTIR indexes between UV-induced aging and natural aging



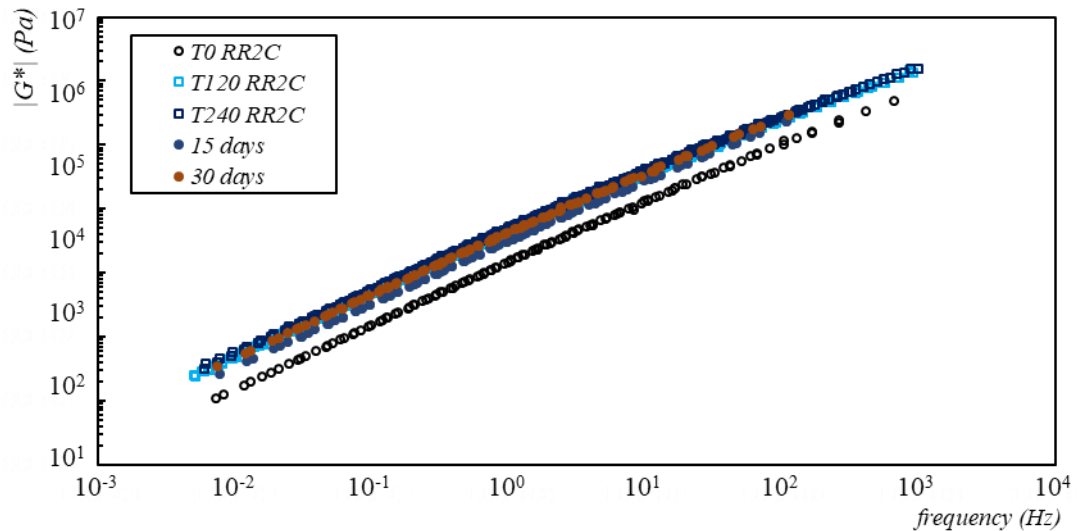
Source: author (2021)



### 2.5.4.2 Complex modulus ( $|G^*|$ )

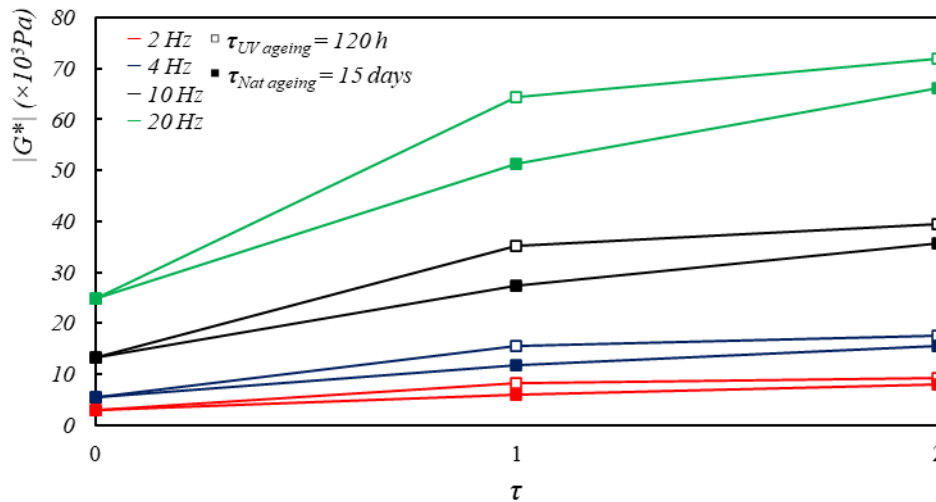
The master curves of  $|G^*|$  ( $T = 60^\circ \text{C}$ ) of the samples were calculated and plotted. Figure 21 presents the master curves obtained with experimental data for the two materials exposed to the natural aging (15 and 30 days). Also, there are the master curves of the materials aged in the UV method for 120 and 240 hours. In the four considered tests there is displacement of the curve to the direction of increase of stiffness, indicating aging.

Figure 21 - Mater curves of  $|G^*|$  after natural aging (15 and 30 days)



Source: author (2021)

In order to compare the two methods, the experimental data was used to elaborate corresponding sigmoidal models. This type of model was selected due to its high adaptability to the master curve of  $|G^*|$ . In addition, the models might provide the value of  $|G^*|$  for specific frequencies with higher accuracy. Figure 22 presents the increase of  $|G^*|$  of the residue after being exposed to the two aging methods. Four frequencies were considered for calculating this increment: 2, 4, 10 and 20 Hz. In relation to the analysed frequencies, the mean increase of  $|G^*|$  when  $\tau = 1$  was: 173 % for the UV-induced aging; and 108 % for the natural aging. When  $\tau = 2$ , the increase of  $|G^*|$  was: 207 % for the UV-induced aging; and 174 % for the natural aging. In the perspective of stiffness, there is indication that the residue aged for 30 days presented more similarities to the residue aged for 120 hours, when analysing the aging in weathering chamber.

Figure 22 - Comparison between  $|G^*|$  between UV-induced aging and natural aging

Source: author (2021)

#### 2.5.4.3 MSCR

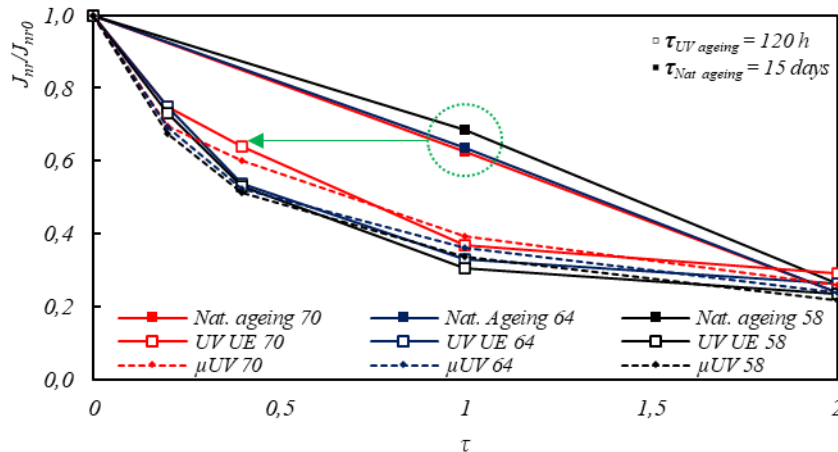
The MSCR test was carried out in each aged sample and the  $J_{nr}$  (at 3,2 kPa) of each sample was used as indication of the level of aging. As observed in the section 2.5.3.1 *Jnr of unmodified emulsion (RR-2C)*, this parameter presents a non-constant tendency of decrease when considering the UV aging. For effects of comparison, the decrease of  $J_{nr}$  calculated in the samples obtained after natural aging was analysed in relation to the UV aging method.

Figure 23 presents curves of decrease of  $J_{nr}$  obtained in relation to the method of aging. In the x-axis there is the time constant  $\tau$ , which corresponds to 120 hours for the UV method and 15 days for the natural method. In the y-axis there is the relation  $J_{nr}/J_{nr0}$  that indicates in each point the relation between  $J_{nr}$  and the  $J_{nr}$  when  $\tau = 0$ . Three temperatures are evaluated: 58, 64 and 70° C. In addition, three types of analysis are made: (i) the decrease of  $J_{nr}$  of the unmodified emulsion after natural aging; (ii) the decrease of  $J_{nr}$  of the unmodified emulsion after UV-induced aging; and (iii) the average decrease of  $J_{nr}$ , considering the three binders analysed in the section 2.5.3 *MSCR × bleeding*.

Considering the curves of mean behaviour of UV aged samples, the  $J_{nr}$  would decrease by half of the initial value after approximately 48 hours of aging. In fact, the results indicate that the reduction of this parameter through time is not constant and is better represented by an exponential curve (R-square > 0.9). When it comes to the MSCR tested after natural aging, it is not possible to conclude which type of behaviour can describe the decrease of  $J_{nr}$ . However, in this case the decrease after  $\tau$  was around the half of the obtained in  $2\tau$ . After 15 days of natural aging, for example, the  $J_{nr}$  would have decreased to a value around 0.65 of the initial

value, what would correspond to the period between 24 and 48 hours of the UV-induced aging. However, analysing the material aged for 30 days in environmental conditions, the  $J_{nr}$  presented a value closer to what was obtained after 240 hours of UV-induced aging.

Figure 23 - Comparison of  $J_{nr}$  between UV-induced ageing and natural aging



Source: author (2021)

More investigation about the accurate relation between the two mechanisms of aging is required. Also, it is important to consider that the material is more susceptible to external factors during the natural aging proceeding. However, the results indicate that the evolution of aging parameters might evolve differently depending on the aging method, especially in relation to the MSCR test.

## 2.6 Conclusions

This paper had as main objective to investigate the effects that aging mechanisms present on rheological parameters of asphalt binders, in particular, those that are related to the performance of CS coats. Based on that, we may highlight the following conclusions:

- i. The UV radiation considerably affects the properties of the emulsion residue. However, the evolution of most of the properties is not constant and the increase/decrease of the parameters throughout time of test could not fit a linear model.
- ii. The non-recoverable compliance ( $J_{nr}$ ) decreased in a form more similar to an exponential curve. Considering that, among the parameters obtained in the MSCR test, this is best recommended to explain the phenomenon of bleeding in chip seals, this type of model is an important direction to understand how this distress evolves throughout service life.

- iii. Regarding the relation between  $J_{nr}$  and bleeding, the unmodified binders presented values above the limits recommended for low volume roads, indicating a high risk of developing bleeding in the roads during the early life of pavements. On the other hand, the polymeric emulsion presented good performance, indicating inclination to better performance.
- iv. When the unmodified emulsion was exposed to natural conditions of aging, the material changed differently from what was obtained in the UV-induced aging using weathering chamber. However, it is possible to point some correlations in important parameters. For example, the index of sulfoxide (Is) and the  $J_{nr}$  presented after 30 days of natural aging values near to the expected for 240 hours of UV aging. In relation to the complex modulus ( $|G^*|$ ), the results obtained with this same material were more similar to 120 hours of UV aging.

It is important to mention that in Brazil the specifications of CS coats suggest that the distress of bleeding is less common than aggregate loss. Thus, it is important to understand how these achievements are reflected in the CS pavements constructed with these types of binder. Also, it would be relevant to carry out field tests to evaluate the evolution of bleeding when these materials are applied and then define limits of  $J_{nr}$  that are better adapted to coats designed by these specifications.

### **Acknowledgement**

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### **3 BINDER- AGGREGATE BOND: ANALYSIS OF BINDER-AGGREGATE INTERFACE OF CHIP SEAL BINDERS IN DIFFERENT STAGES OF AGING USING BITUMEN BOND STRENGTH**

#### **3.1 Abstract**

This paper aims to evaluate the evolution of the adhesion in the binder-aggregate interface during service life of binders used in chip seal treatments in Brazil. For this purpose, three materials were selected: two rapid-setting emulsions (unmodified and polymeric) largely recommended for the construction of these coats and a conventional asphalt cement binder of 50-70 penetration, similar to the binder used on the manufacturing of these emulsions. Samples of binder were aged in laboratory using two methods: (i) the ultraviolet (UV) induced aging, in weathering chamber; and (ii) the conventional PAV aging. The bitumen bond strength (BBS) test was used to evaluate adhesion/cohesion in the binder-aggregate interface. It was possible to conclude that both UV and PAV ageing mechanisms affect considerably the composition of the binders and consequently the parameters related to adhesiveness/cohesiveness and the occurrence of more adhesive failures.

#### **3.2 General Considerations**

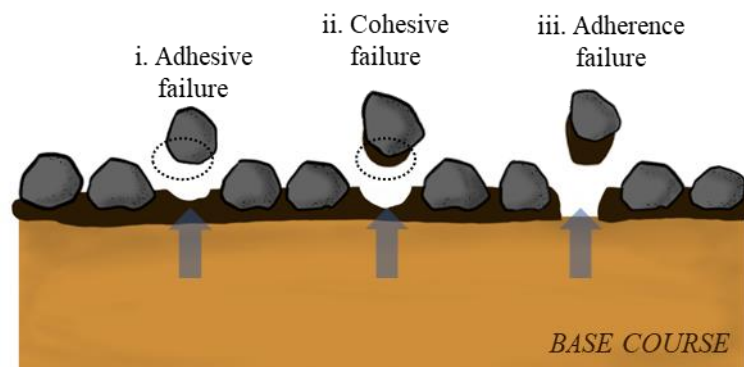
The bond between binder and aggregates has an important role in the performance of pavements. A high adhesion between the asphalt and the aggregates and a strong cohesion among molecules inside the binder affects the binder-aggregate interface which also correlates to the performance of pavement concerning common distresses. Both these mechanisms are commonly referred together as adhesiveness. In relation to chip seal (CS) treatments, these interactions are considered when analysing ravelling distresses.

Among the factors that affect the bond in binder-aggregate interface it is relevant to consider physical and chemical aspects of the materials that enhance both adhesion and cohesion. In relation to aggregates, shape properties - such as porosity and texture - and mineralogy are frequently mentioned as effective parameters that contribute to better adhesiveness. Alternatively, the composition of binders is known to improve adhesion, when there is good interaction between this material and the surface of aggregates, as well as cohesion, when there is a strong bond between the molecules.

These factors are examined when it comes to materials for chip seal pavements. In this type of coat, the bond between binder and aggregate is commonly associated to loss of

aggregate (ravelling) in the surface of the pavement, which is one of the most common distresses found in chip seal treatments. In pavements, this phenomenon occurs due to cohesive or adhesive failure. Figure 24 illustrates these two types of failure: (i) firstly, there is an adhesive failure, in which the aggregate detaches completely of the asphalt film; (ii) in the cohesive failure the aggregate detaches from the asphalt layer, but part of the binder content remains in the surface of the aggregate indicating that the rupture occurred in the asphalt film and (iii) depending on the thickness of the asphalt remaining on the surface of the aggregate, this rupture might cause the exposure of the base layer, one of the side effects of aggregate loss/ravelling.

Figure 24 – Types of failure in binder-aggregate bond in chip seals



Source: author (2021)

The distress of ravelling is known to occur during different stages of service life regarding coats. The binder-aggregate bond is often used to explain part of this phenomenon in the early life (MORAES *et al.* 2013), when the surface of the pavement is unaged and more vulnerable to distresses of this type. Besides that, the process of aging affects also other characteristics of the binder that might be related to the adhesion in the binder-aggregate interface. In this perspective, the objective of this paper is to investigate how adhesion and cohesion evolves based on the aging of binder and which variables might be related to failures on this interface.

### 3.3 Literature Review

In chip seal treatments, the bitumen layer is expected to provide adherence between the chips and the pavement surface, preventing distresses such as chip loss. The Bitumen Bond Strength (ABS/BBS) test (AASHTO TP 91-11) is used to estimate the minimum pull-off tensile strength (POTS) necessary to break the binder-aggregate bond. This test is adapted from the coating industry. The results obtained from ABS test can be used to: (a) rank more appropriate

materials for construction based on the performance of the bond obtained; and (b) propose criteria of minimum POTS necessary to select materials to operate under specific conditions of traffic.

### 3.3.1 BBS test

The BBS test is made by forcing the rupture of the bond made between binder and aggregate using a controlled tensile force. The equipment induces the tensile force needed in the system and a pull-out stub is used to adhere a solid substrate after conditioned and bond with the asphalt binder tested. This test is mentioned in the literature of pavements materials for different purposes. For example, it is appropriate to evaluate the bond necessary to avoid the distress of ravelling in CS pavements.

Among the advantages of using the BBS method to evaluate the binder-aggregate bond, it is the relative low time required to run the test. Also, various parameters can be analysed and adjusted in the test, such as: the equipment used in the test; the conditioning temperature; the conditioning time and the temperature of the test. When it comes to emulsions, the time for break and cure can also be adjusted to the type of analysis. Table 8 summarizes part of the literature that considered these parameters for similar purposes to the ones addressed in this paper.

Table 8 – Literature that mention the BBS test

Reference	Equipment	Curing* (emulsions)	Conditioning temperature	Time of cure (emulsion)	Conditioning time	Temperature of test
Moraes (2011)	PATTI	-	-	-	0, 6, 24, 48 and 96 hours	-
Hanz (2012)	pneumatic	ROR	-	6 hours	24 hours	~23 °C
Adams (2014)	PATTI	COR and ROR	15-25 °C	4 and 20 hours	-	15 and 25 °C
Moraes (2013)	PATTI	COR	70 °C	2, 4, 6, 12, 24, 48, 96, 192, 360, 720 and 1080 hours	-	25 °C
Ahmed (2017)	PosiTest AT-A	-	60 °C	-	24 hours	-
Silva (2018)	PosiTest AT-A	COR	50 and 25 °C	24 and 48 hours	-	25 °C

\* ROR: Residue on rock/ COR: Curing on rock  
Source: author (2021)

Adams (2014) analysed the relation between Vialit aggregate loss and bond strength for CS coats designed following US practice. The bond strength was analysed using the pneumatic

adhesion tensile testing instrument (PATTI) and the procedure used was the ASTM D4541 for emulsions at 15 and 25 °C. Also, two methods of curing were considered: (i) the curing on rock (COR), in which the emulsion is spread in the rock and the pull-out stub which is placed on the substrate before the evaporation of water content and (ii) the residue on rock (ROR), in which the residue is placed on the substrate after the breakup of emulsion. Among those methods, the COR showed better correlation with the Vialit test. The R-square obtained was 0.749 after 20 hours of dry curing. This correlation was used to propose limits of pull-off tensile strength for CS coats based on the level of traffic.

Concerning the Brazilian practice, Silva (2018) analysed the bond strength of emulsions incorporating the analysis of local coats. Similarly, the materials were analysed in terms of adhesiveness and these results were related to the distress of aggregate loss, using the Wet Track Abrasion Test (WTAT) adapted for CS samples. A hydraulic apparatus PosiTest AT-A was used in the BBS test. The samples were conditioned at 50 and 25 °C during 24 and 48 hours of curing. These tests shown that heating the emulsion reflected in the increase of the Pull-off tensile strength (POTS). The same tendency was also noted in the tests of aggregate loss using Wet Track Abrasion Test.

It is also important to consider that the conditioning time of the samples affects considerably the resulting bond strength. There are also differences when conditioning the materials in oven due to the oxidative effect that heating has on binders. Moraes and Bahia (2013) studied the relation between the bond strength and ravelling using aged emulsions. The samples were heated in oven at 70 °C for 2 to 1080 hours. The test used to investigate ravelling was the Sweep Test (ASTM D7000-11). It was observed that the bond strength in the first hours of test was particularly useful to explain the phenomenon of aggregate loss, which indicates that this test is useful to analyse performance of binders in the early life. Also referring to conditioning time, Moraes, Velasquez, and Bahia (2011) noted that the conditioning time was significant when it comes to moisture damage in tests carried with immersion in water depending on the nature of the substrate.

The apparatus used for BBS test consists of an instrument used to induce tensile and a pull-out stub. In relation to the mechanism used in the instrument, it can be pneumatic (PATTI) or hydraulic (PosiTest AT-A), depending on the model of the equipment. The geometry of the pull-out stub is also mentioned in standards of the test. Ahmed, Lee, and Williams (2017) investigated different geometries of stub to indicate which one is more accurate to explain the phenomenon of adhesiveness in asphalt mixtures. The authors found an indication that the stub



with edge 0.00 mm presented feasibility and it is less prone to cohesive failures than stubs with 0.40 and 0.80 mm.

### ***3.3.2 Factors associated to adhesion/cohesion***

The binder-aggregate bond is governed by characteristics found in both binders and aggregates. These characteristics may be related to the constitution of each material. For example, the acidity or alkalinity of aggregates can affect the adhesiveness to the binder (TARRER and WAGH, 1991). Due to the chemical composition, basic aggregates in general present better resistance to stripping of asphalt films than acid aggregates. Also, in relation to chemical composition, Lucas Júnior, Babadopulos and Soares (2018) suggest that aggregates with higher contents of  $\text{Fe}_2\text{O}_3$  and CaO improved the adhesion to asphalt films. This is also reflected in moisture damage. Bagampadde, Isacson and Kiggundu (2005) suggest that mixtures from aggregates containing alkali metallic elements tend to exhibit higher moisture sensitivity. Based on this, it is important to prefer aggregates with mineralogy that are less susceptible to present low adhesiveness. A possibility for aggregates that do not attend this criterion can be the use of additives. Moraes, Velasquez, and Bahia (2011) show that is possible to improve the moisture resistance of binder-aggregate systems with use of polymers, especially when using granitic or acidic aggregates. The chemical composition of binders is also significant in the binder-aggregate interface bond. In general, it is expected that sulfoxides and carboxylic acids present great affinity for aggregates while the same is not expected from aromatic hydrocarbons (Moraes, Velasquez, and Bahia, 2011).

### ***3.3.3 Curing and aging in Chip Seals***

In CS treatments, two processes modify considerably the performance of the binder. Firstly, the cure of water is the process of evaporation of water content of asphalt emulsions that happens in the first hours after the construction of the coats along with the breakup. Also, there is the process of aging that occurs due to the oxidative factors that the coats are exposed to such as heating, sunlight, and environmental conditions. The discussion about these processes is referred in the section 2.3.2 *Aging*.

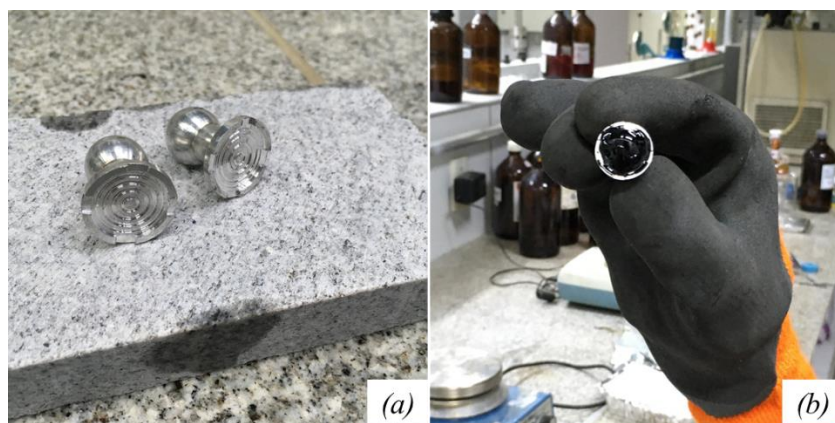
## **3.4 Method**

The samples of BBS test were prepared in accord to AASHTO TP 91-11. Three binders were selected for the test: (i) an unmodified rapid-setting emulsion, commonly recommended for CS treatments (RR-2C); (ii) a polymeric rapid-setting emulsion, recommended for CS

treatments (RR-2C-E) and (iii) a conventional asphalt binder of penetration 50-70 at room temperature (25 °C) similar to the material used to produce the emulsions. A granitic aggregate was selected as substrate. Aggregates of same origin are often used in construction of CS coats in the state of Ceará and it is also recommended by local research (PEREIRA, 2013; SILVA, 2018).

The equipment used to induce the pull-off tensile was the PosiTest AT-A. The rate of tensile was set as 0.7 MPa/s. The stub used was the standard 20 mm diameter. Figure 25a presents an example of stub used in this research and one of the aggregates used as substrate. Figure 25b illustrates the stub in the moment of the application of the binder and before being put on the substrate.

Figure 25 – Pull-out stub used in the BBS test



Source: author (2021)

#### 3.4.1 *Aging of samples*

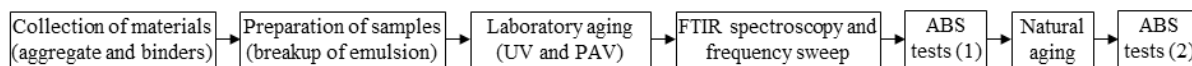
The materials were conditioned in induced aging proceedings (UV and PAV), as explained in the section 2.3.1 *Preparation of samples*. For effects of analysis, the standard procedure adopted for analysing the two residues after induced aging was similar to the one used to analyse the asphalt binder.

#### 3.4.2 *Experimental plan*

The experimental plan designed for this paper was divided in steps summarized in Figure 26. The first step is the collection of materials. The emulsions and the asphalt binder were obtained from the same supplier in the state of Ceará. The aggregate was also collected from a local supplier and cut following the specifications of substrate of the BBS test. The following steps are related to the preparation of samples for the BBS test. Firstly, it was carried

the tests with samples cured isolated. Then, it was carried the tests with emulsions cured on the substrate.

Figure 26 – Flow chart of the experimental plan (chapter 3)



Source: author (2021)

The emulsions were applied in a rate of 1.4 L/m<sup>2</sup> in a silicone recipient and then left for cure for 24 hours at environmental conditions. The samples of asphalt binder were prepared following the proportion of asphalt content of the emulsions. In the next step it was applied the aging mechanisms. Two methods were applied, one with the use of ultraviolet (UV) light in a weathering chamber and (ii) another with the use of the Pressure Aging Vessel (PAV) described in the standard ASTM D6521 – 19 (ASTM, 2019).

A test routine for the weathering chamber was used. The test parameters were defined as: frequency of 300-400 nm; temperature of 70 °C (in the specimen); and room temperature of 35 °C. Also, it was made the monitoring of moisture level in the chamber at 50 %. In this paper, samples were aged for 120 and 240 hours by this method.

In the next step, it was performed the tests of Fourier-Transform Infrared (FTIR) spectroscopy and a frequency sweep. The unaged binders were also included in these tests The BBS test was then carried in the samples following the defined specifications for the test.

In the second part, the unmodified emulsion was tested following the procedure recommended for asphalt emulsions, with the cure occurring in the substrate. The BBS test of this sample was carried right after cure and then, after aged for 120 hours at environmental conditions.

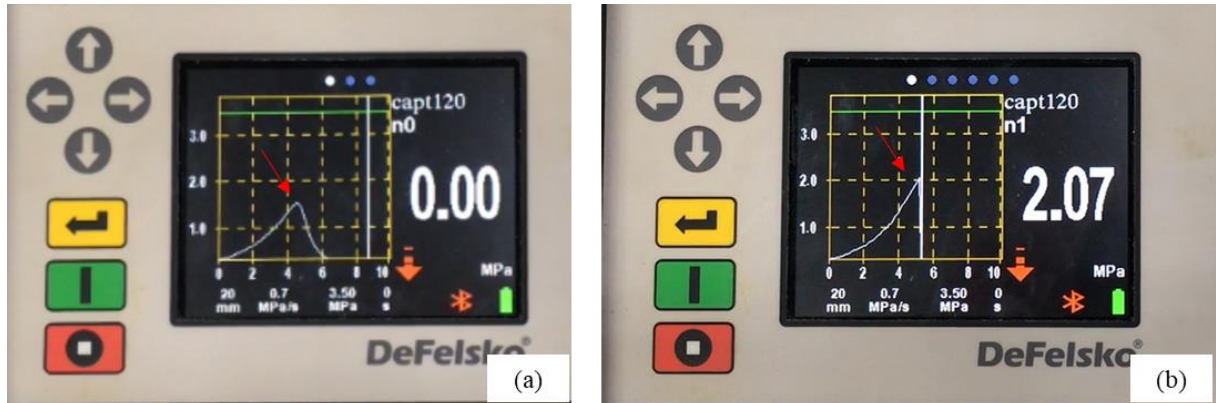
### 3.5 Results

The aged binder was tested before proceeding the BBS test. Frequency sweep and FTIR spectroscopy were carried out in these samples. For more information about the result of these tests one can refer to the items 2.5 *Results* and 2.5.2 *Analysis of Complex Modulus*.

In the BBS test performed with the hydraulic equipment the pull-off loading is applied in the interface in a rate defined by the user. In this test it was defined the loading rate of 0.7 MPa/s considering the indication that results are more coherent (SILVA, 2018). When the samples reach failure, two behaviours are observed. Firstly, failure can be reached when the

pull-off load is in the maximum value of test (Figure 27a). Also, the pull-off loading can perform a peak and then end the test in an inferior value (Figure 27b). In both cases the POTS was defined as the maximum value of the curve.

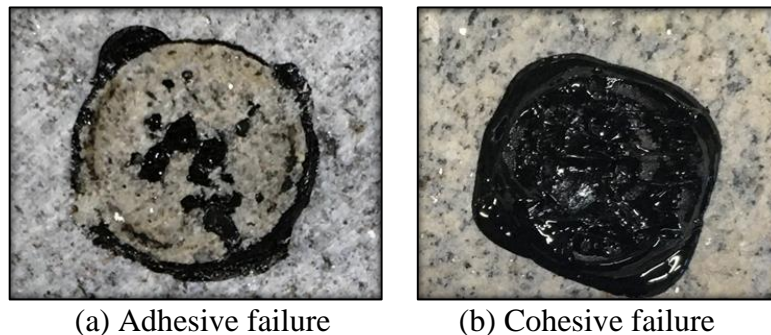
Figure 27 – Acquisition of maximum POTS



Source: author (2021)

Figure 28 illustrates two examples of tests performed with different samples. AASHTO TP 91-11 designates two types of failure. If the binder detaches completely from the substrate, the failure is adhesive (Figure 28a). When the binder remains in the substrate, the failure is cohesive (Figure 28b). It is also possible to occur a combination of the two types of failure, in which the binder remains partially in the substrate.

Figure 28 – Adhesive and cohesive failures



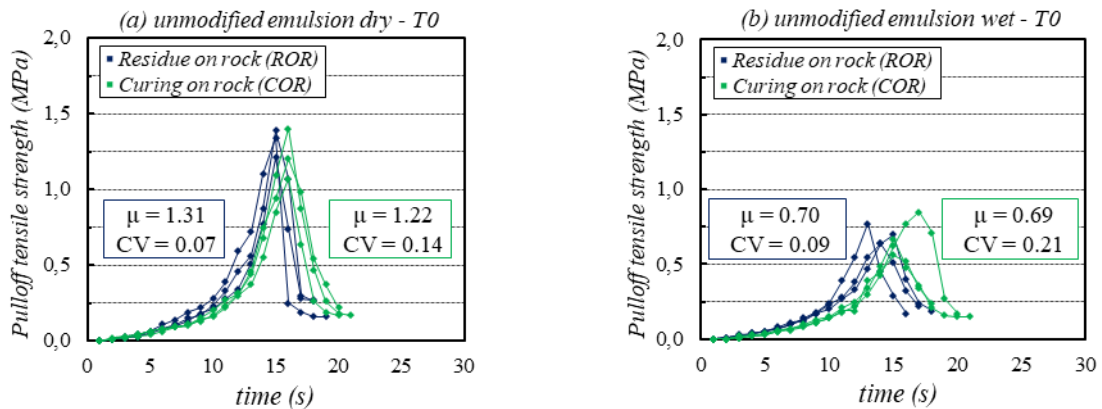
Source: author (2021)

### 3.5.1 Comparing residue on rock (ROR) and curing on rock (COR)

Considering the procedure of break and cure of asphalt emulsion, before testing the aged binders, the unmodified emulsion was tested following the methods defined as COR and ROR. The samples were tested dry and wet, after a period of immersion in water of 24 hours. The tensile strength was obtained in each second in the tests and a curve was plotted to outline the evolution of this parameter during tests for each sample. Figure 29 presents the curves obtained

in each test. Also, it is presented the arithmetic mean ( $\mu$ ) and the coefficient of variation (CV) of maximum POTS of each type of material. Figure 29a presents curves of samples that were tested in dry conditioning and Figure 29b presents curves of material tested after the period of 24 hours of immersion in water.

Figure 29 – BBS test of the unmodified emulsion before aging (ROR  $\times$  COR)



Source: author (2021)

In average, the POTS reached higher values in tests carried with dry conditioning. The sample of residue cured out of the substrate presented  $\mu = 1.31$ . Alternatively, the mean of the sample cured on the substrate was 1.22. Considering the dispersion of this parameter it is not possible to conclude that there is difference between the two samples. In fact, both the CV calculated indicate intervals that embraces both means. When it comes to the wet conditioning, the same behavior of the samples was observed. The mean of the ROR and COR samples were, respectively, 0.70 and 0.69, indicating a difference even lower than the obtained at dry conditioning.

Thus, it is possible to infer that the BBS procedure developed with the conditions established for this test is little influenced by divergences between the interface formed in samples tested by ROR or COR procedures.

### 3.5.2 Bond strength analysis

#### 3.5.2.1 Asphalt binder

Table 9 presents results obtained in BBS tests with the conventional asphalt binder in dry and wet conditioning. Considering the dry conditioning, all samples presented cohesive failures. It was observed that 120 hours of UV-induced aging reflected in a mean POTS of 2.11 MPa. After 240 hours this parameter was 2.12 MPa, not statistically different from 2.11 MPa. Based on that, it is possible to infer that the first 120 hours of aging in this method is responsible

for reaching the maximum increase of POTS, since changes were not observed in this parameter in the next 120 hours. Contrastingly, the sample aged in PAV presented the highest POTS (2.39 MPa). Thus, there is indication that the UV aging affects the material differently from the PAV aging.

When it comes to wet conditioning, a different behaviour was observed. In relation to the UV-induced aging, the increase of aging between 120 and 240 hours reflected in a divergence in the mean POTS. In these samples, the highest POTS was observed after 240 hours of UV ( $\mu = 1.24$  MPa). In relation to the type of failure, all samples presented a combination of adhesive and cohesive failure. The PAV-aged sample, however, presented the most adhesive behaviour among these samples.

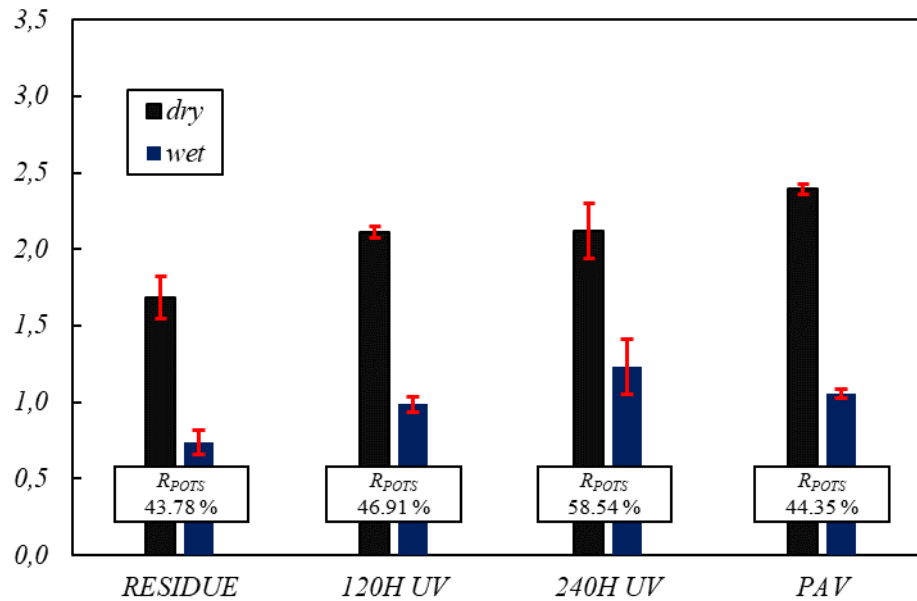
Table 9 – POTS of the conventional asphalt binder after aging

<i>conditioning</i>		$\mu$	<i>CV</i>	<i>max</i>	<i>min</i>	<i>failure type</i>
<i>DRY</i>	<i>unaged</i>	1.69	0.08	1.81	1.54	cohesive
	<i>120H UV</i>	2.11	0.02	2.14	2.07	cohesive
	<i>240H UV</i>	2.12	0.09	2.25	1.91	cohesive
	<i>PAV</i>	2.39	0.01	2.42	2.36	cohesive
<i>WET</i>	<i>unaged</i>	0.74	0.10	0.81	0.66	cohesive/adhesive
	<i>120H UV</i>	0.99	0.05	1.02	0.93	cohesive/adhesive
	<i>240H UV</i>	1.24	0.15	1.43	1.07	cohesive/adhesive
	<i>PAV</i>	1.06	0.03	1.09	1.03	mostly adhesive

Source: author (2021)

Figure 30 presents a comparison between dry and wet conditioning in the samples. Also, it is presented the interval defined by the standard deviation ( $\delta$ ) and the bond strength ratio ( $R_{POTS}$ ). The 240 hours UV aged presented both the largest variation of POTS and the highest  $R_{POTS}$  (58.54 %). Based on this, there is an indication that the UV-induced aging contributed to the adhesion, decreasing the moisture damage of the samples, while the same was not observed in the PAV aged samples.

Figure 30 – Comparing BBS test in dry and wet conditions (asphalt binder)



Source: author (2021)

Based on the results, it is possible to presume that, for the conventional asphalt binder, both aging mechanisms affected the bond strength of the binder increasing the POTS. However, the sample was affected differently depending on the mechanism of aging. In addition, the results obtained in moisture damage indicates that the aging mechanisms affects the wet conditioned samples in different proportion.

### 3.5.2.2 Unmodified emulsion

Similar to what was observed in the asphalt binder, in the unmodified emulsion the mechanisms of aging affected the bond strength increasing the POTS. Table 10 presents results of BBS obtained in tests with this material.

In dry conditioning, the mean POTS increased from 1.22 to 1.79 MPa after 120 hours of UV-induced aging. There was little difference observed between this sample and the one aged for 240 hours, the mean POTS obtained was 1.84 MPa. In the PAV aged sample, it was observed the highest value of POTS among samples tested in these conditions. In relation to failure type, only cohesive failure was obtained.

Alternatively, in the aged samples tested in wet conditions, the failure type was partially cohesive and adhesive. However, the unaged sample also presented cohesive failure. Among samples tested in these conditions, the highest POTS (1.26 MPa) was obtained after PAV aging, conflicting to what was obtained in the tests with the conventional asphalt binder.

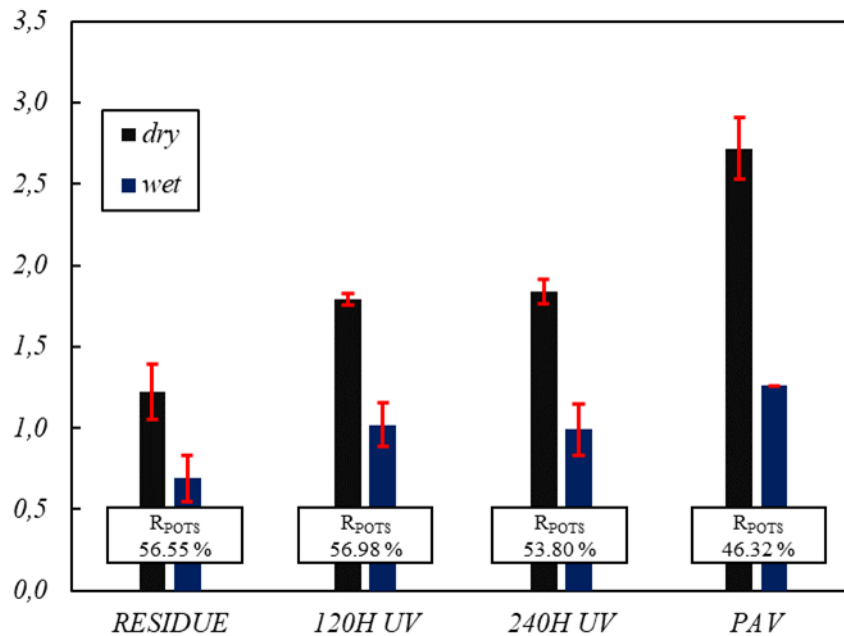
Table 10 - POTS of the unmodified emulsion after aging

<i>conditioning</i>		$\mu$	<i>CV</i>	<i>max</i>	<i>min</i>	<i>failure type</i>
<i>DRY</i>	<i>unaged</i>	1.22	0.14	1.40	1.07	<i>cohesive</i>
	<i>120H UV</i>	1.79	0.02	1.82	1.74	<i>cohesive</i>
	<i>240H UV</i>	1.84	0.04	1.91	1.78	<i>cohesive</i>
	<i>PAV</i>	2.72	0.07	2.86	2.50	<i>cohesive</i>
<i>WET</i>	<i>unaged</i>	0.69	0.21	0.85	0.56	<i>cohesive</i>
	<i>120H UV</i>	1.02	0.13	1.17	0.93	<i>cohesive/adhesive</i>
	<i>240H UV</i>	0.99	0.16	1.12	0.82	<i>cohesive/adhesive</i>
	<i>PAV</i>	1.26	0.00	1.26	1.25	<i>cohesive/adhesive</i>

Source: author (2021)

Figure 31 presents the evolution of POTS of samples in relation to the conditioning procedure. Also, it is presented the interval defined by the standard deviation and the  $R_{POTS}$  for each aging mechanism. In the PAV aged samples, the  $R_{POTS}$  was 46.32 %, the lowest observed in this material. Based on this observation it is possible to infer that the PAV mechanism affected more strongly the moisture damage.

Figure 31 - Comparing BBS test in dry and wet conditions (unmodified emulsion)



Source: author (2021)



### 3.5.2.3 Polymeric emulsion

The polymeric emulsion presented the most dissimilar results of BBS test. In addition, due to the high viscosity, the aged samples presented low workability to the temperatures used in the pull-out stub (150 ° C). Table 11 summarizes results obtained in tests carried out with this material.

Among dry conditioned tests, the highest mean POTS was obtained both in the 120 hours UV-aged and in the PAV aged samples (2.28 MPa). Differently from what was observed in other materials, there was a decrease between the results obtained from 120 to 240 hours. In contrast, when it comes to the wet conditioned tests, the highest POTS was observed in the 240 hours UV-aged sample.

In relation to the type of failure, all samples tested in dry conditioning presented cohesive failure. However, the wet conditioning reflected in mostly adhesive failures. Among these, the PAV-aged sample presented only adhesive failures.

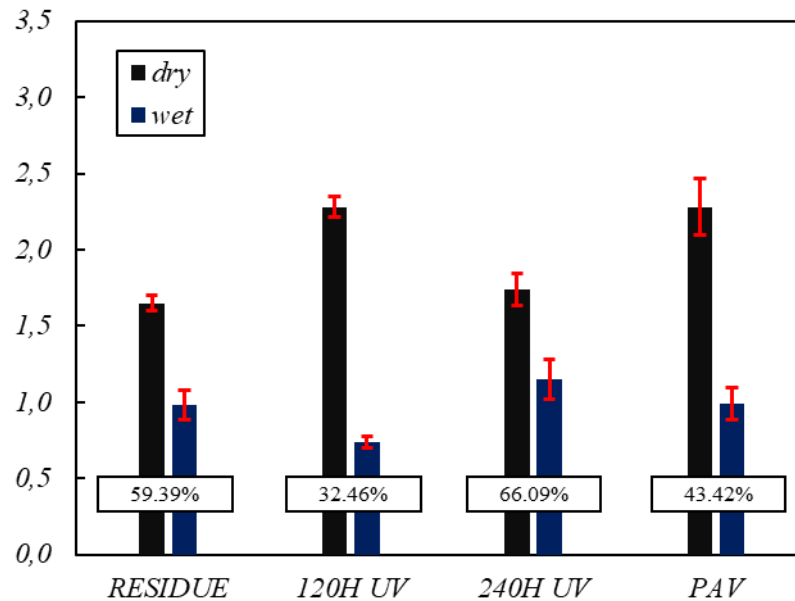
Table 11 - POTS of the polymeric emulsion after aging

<i>conditioning</i>		$\mu$	<i>CV</i>	<i>max</i>	<i>min</i>	<i>failure type</i>
DRY	<i>unaged</i>	1.65	0.03	1.71	1.64	cohesive
	120H UV	2.28	0.03	2.34	2.22	cohesive
	240H UV	1.74	0.06	1.85	1.63	cohesive
	PAV	2.28	0.08	2.5	2.13	cohesive
WET	<i>unaged</i>	0.98	0.10	1.05	1.02	cohesive/adhesive
	120H UV	0.74	0.05	0.76	0.07	mostly adhesive
	240H UV	1.15	0.11	1.24	1.01	mostly adhesive
	PAV	0.99	0.11	1.09	0.88	adhesive

Source: author (2021)

Figure 32 presents a comparison between dry and wet conditioning in the polymeric emulsion samples. Also, it is presented the interval defined by the standard deviation ( $\delta$ ) and the bond strength ratio ( $R_{POTS}$ ). The 240 hours UV aged presented the highest  $R_{POTS}$  (66.09 %). In contrast, the lowest ratio was observed in the 120 hours sample (32.46 %). This result might be related to the contribution of the polymer in the material.

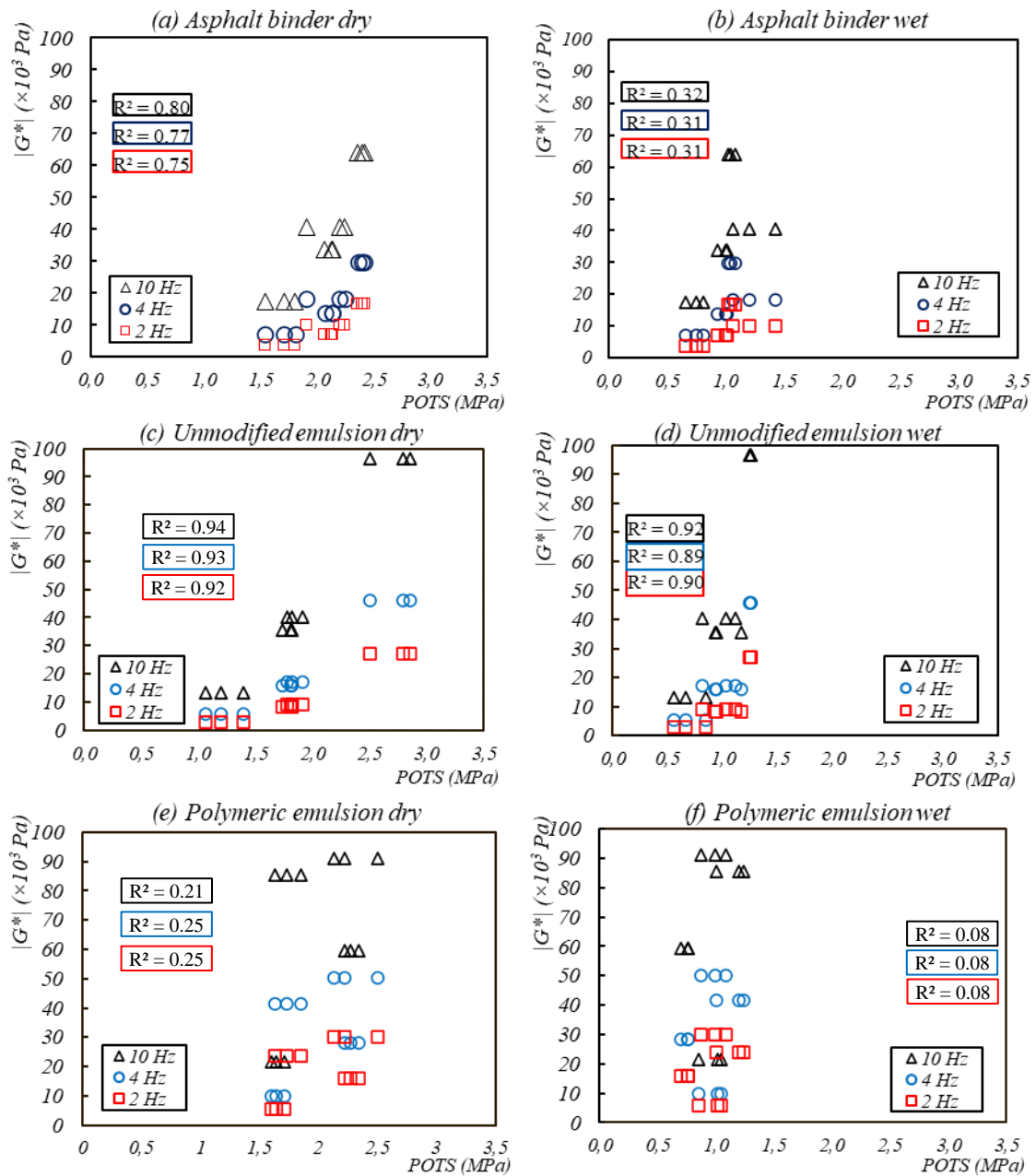
Figure 32 - Comparing BBS test in dry and wet conditions (polymeric emulsion)



Source: author (2021)

#### 3.5.2.4 Complex modulus

Considering the effects of stiffness over bond strength, the master curve of the complex modulus ( $|G^*|$ ) at 60 °C was analysed in contrast to the results of POTS obtained. Three frequencies were evaluated: (i) 2 Hz, (ii) 4 Hz and (iii) 10 Hz. Figure 33 presents the graph of the dispersion  $POTS \times |G^*|$  plotted for each material and conditioning method (dry or wet). The linear correlation of each dispersion was calculated and presented in the corresponding graph.

Figure 33 -  $|G^*| \times POTS$ 

Source: author (2021)

Figure 33a presents the graph  $POTS \times |G^*|$  for the conventional asphalt binder dry-conditioned. It was observed high correlation among these dispersions, with R-square over 0.75. The highest R-square was obtained when analysing stiffness corresponding to 10 Hz (0.80). When it comes to the analysis of this material in wet conditions, the correlation was expressively lower. For example, the correlation corresponding to the frequency of 10 Hz in this second analysis was 0.32.

The unmodified emulsion presented the highest correlation for both conditioning methods. Concerning dry conditioning, the highest correlation was observed in the frequency

of 10 Hz (0.94), similar to what was observed in the analysis of the conventional asphalt binder. High correlation was also observed in the same material when analysed in wet conditioning in the same frequency (0.92).

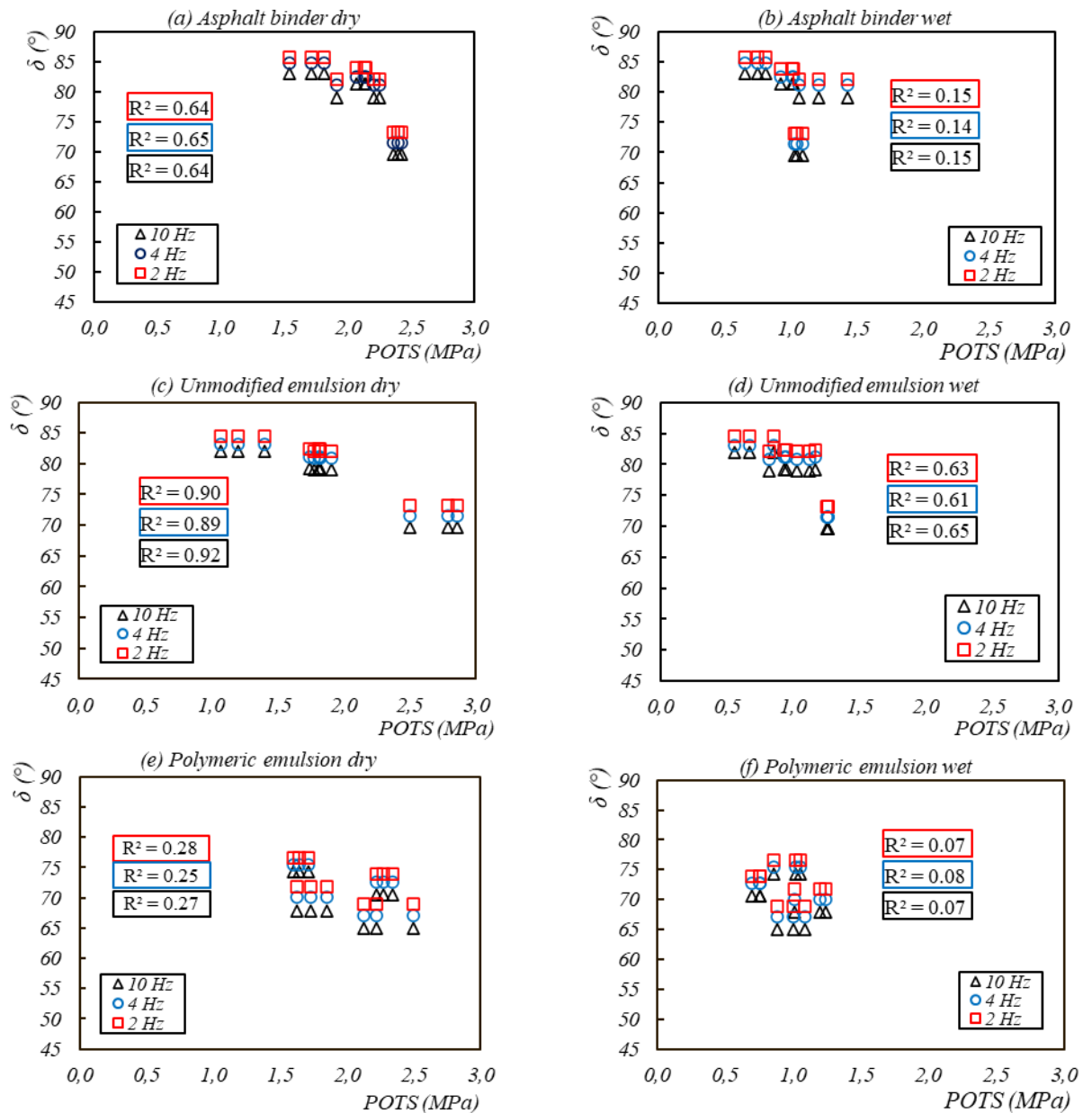
In relation to the polymeric emulsion, both analyses presented weak correlation with the values of R-square inferior to 0.25. Even in the analysis of the test carried under dry conditioning, the parameter  $|G^*|$  did not seem to be indicated to explain the phenomenon of adhesiveness. The analysis of the wet conditioned tests presented the least reliable correlation (Figure 33f).

Comparing the results obtained among the emulsion residues, it is possible to infer that the stiffness might be one good parameter to explain adhesiveness and cohesiveness when analysing unmodified materials. The same was not observed in the analysis of the polymeric residue, which might be an indicative that parameters related to the polymer behaviour would also interfere in this correlation. Also, it is important to highlight that at certain aging circumstances the polymeric emulsion presents low workability and cannot be easily tested in the BBS procedure as the unmodified emulsion.

In addition, it is important to highlight that although the  $|G^*|$  presented good correlation with the POTS obtained in the unmodified binders, this correlation is lower in materials tested under wet conditions, especially when it comes to the conventional asphalt binder. This gives indication that this material might be more susceptible to moisture damage.

#### *3.5.2.5 Phase angle*

The phase angle of the aged materials was also considered in the analysis. Figure 34 presents the relation between this parameter and the POTS obtained. The same frequencies mentioned in the complex modulus analysis were considered in this section.

Figure 34 – Phase angle  $\times$  POTS

Source: author (2021)

Figure 34a and Figure 34b present the graph corresponding to the analysis of the conventional asphalt binder respectively under dry and wet conditioning. The correlation was higher when analysing dry binder conditioning. The highest R-square value in this case was 0.65. When analysing the wet conditioning, however, this value decreased to lower than 0.15.

Concerning the unmodified emulsion, the R-square value was overall higher. For example, for the dry conditioned test, the R-square fluctuated around 0.89 and 0.92, as presented in Figure 34c. In Figure 34d it is also presented that this parameter varied between 0.61 and 0.65 considering wet conditioning.

The results obtained for the polymeric emulsion indicates that the phase angle is not efficient to explain the phenomenon of adhesiveness and cohesiveness. In the analysis of this material the R-square obtained was always lower than 0.28, which indicates low correlation in this type of analysis.

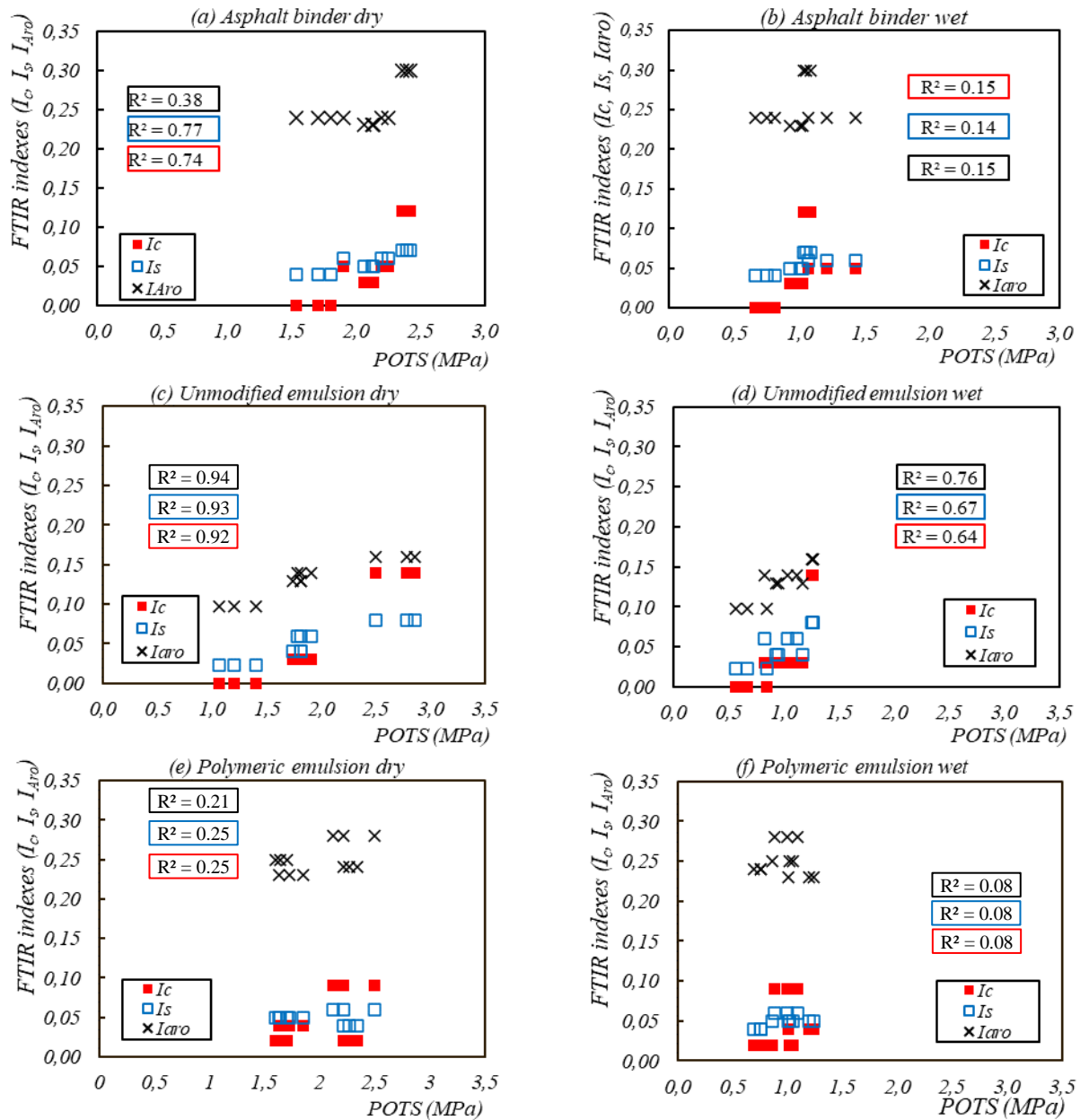
#### 3.5.2.6 FTIR spectroscopy

Parameters obtained from the FTIR spectroscopy were also considered in this analysis. Figure 35 presents a graph in which the POTS obtained in each test is correlated to the indexes ( $I_c$ ,  $I_s$  and  $I_{Aro}$ ) calculated for the aged materials.

Figure 35a presents the analysis of the conventional asphalt binder considering dry conditioning. Under these conditions the indexes  $I_s$  and  $I_c$  presented good correlation to the POTS, with R-square over 0.70. In contrast, the R-square calculated for the  $I_{Aro}$  was 0.38. Based on this, it is possible to conjecture that the formation of carbonyls and sulfoxides might be better related to the increase of POTS in these tests. In contrast, the good correlation was not obtained when analysing the wet conditioned tests. In this second analysis (Figure 35b), the maximum R-square calculated was nearly 0.15 among the three indexes.

The analysis of the unmodified emulsion produced the highest values of R-square concerning the FTIR indexes. In the dry conditioned tests (Figure 35c) this parameter was between 0.92 and 0.94, indicating high correlation. When it comes to the wet conditioning, this parameter decreased for between 0.64 and 0.76. It is relevant to highlight that in this case the index that presented better correlation was the  $I_{Aro}$ , contrasting to what was previously obtained.

Lastly, the polymeric emulsion was analysed. In this analysis low correlation was observed in both conditioning methods. The R-square obtained was below 0.25 considering dry conditions and below 0.8 considering wet conditions. Based on this it is not possible to affirm that the adhesiveness and cohesiveness of polymeric binders are related to the formation of carbonyls, sulfoxides and aromatic due to aging mechanisms.

Figure 35 – FTIR indexes ( $I_C$ ,  $I_S$  and  $I_{Aro}$ )  $\times$  POTS

Source: author (2021)

Adhesiveness and cohesiveness are two phenomena influenced by the molecules existing in the composition of the binders. Some of the products of oxidation might affect these properties. Sulfoxides and carboxylic acids, for example, are known to improve the affinity to certain types of aggregates (MORAES, VELASQUEZ, AND BAHIA, 2011). However, in some cases, this improvement is only possible when the effects of moisture are not considered. Taking as subject the unmodified asphalt binder, it is possible to observe that the indexes of sulfoxides and carbonyls presented relevant correlation to the POTS in dry conditioned tests. The same was not observed when the moisture effects were considered.

### 3.5.3 Comparing UV aging and PAV

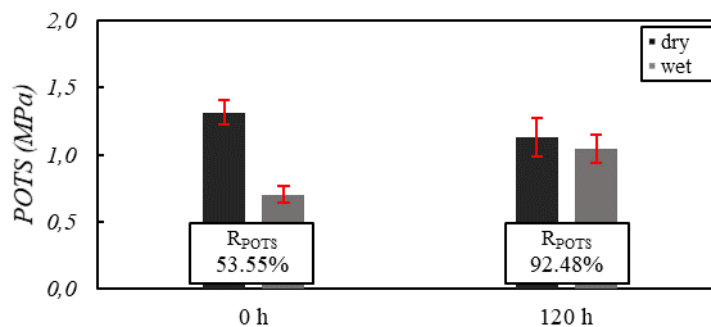
In the perspective of the aging method, two different behaviours were obtained. Firstly, in relation to the asphalt binder and the unmodified residue it was observed that between 120 and 240 hours of UV-induced aging the variation of POTS presented low sensitivity. Also, it was observed an increase in the mean POTS in the PAV aged binders. In contrast, the polymeric residue presented a divergent behaviour. The mean POTS decrease between 120 hours and 240 hours of UV aging and increased for the same value (2.28) in relation to PAV aging.

### 3.5.4 Natural cure and aging

Concerning the natural process of cure and aging, the unmodified emulsion was also tested for BBS after aged in natural conditions of sunlight and temperature for 120 hours. In this test, the break and cure of the emulsion occurred in the substrate simultaneously with the process of aging.

Figure 36 summarizes the results obtained using natural aging. Also it is presented the results obtained in the BBS test of the unmodified emulsion following the “curing on rock” procedure discussed in the item 3.5.1 *Comparing residue on rock (ROR) and curing on rock (COR) (0h)*. It was observed a decrease in the mean POTS between 0 and 120 hours of natural aging when considering the dry conditioning. When it comes to the wet conditioned tests, this parameter showed an increase. Also, the  $R_{POTS}$  in this sample presented the highest value, giving indication that the residue might preserve the attribute of good adhesiveness of the emulsion after this period of aging. This observation can be compared to the  $R_{POTS}$  obtained in the UV aged binders. In both unmodified materials this percentual was higher when compared to the PAV aging. One hypothesis for this result is that the UV radiation might be beneficial for adhesion and moisture damage.

Figure 36 – BBS of the unmodified emulsion aged in natural conditions

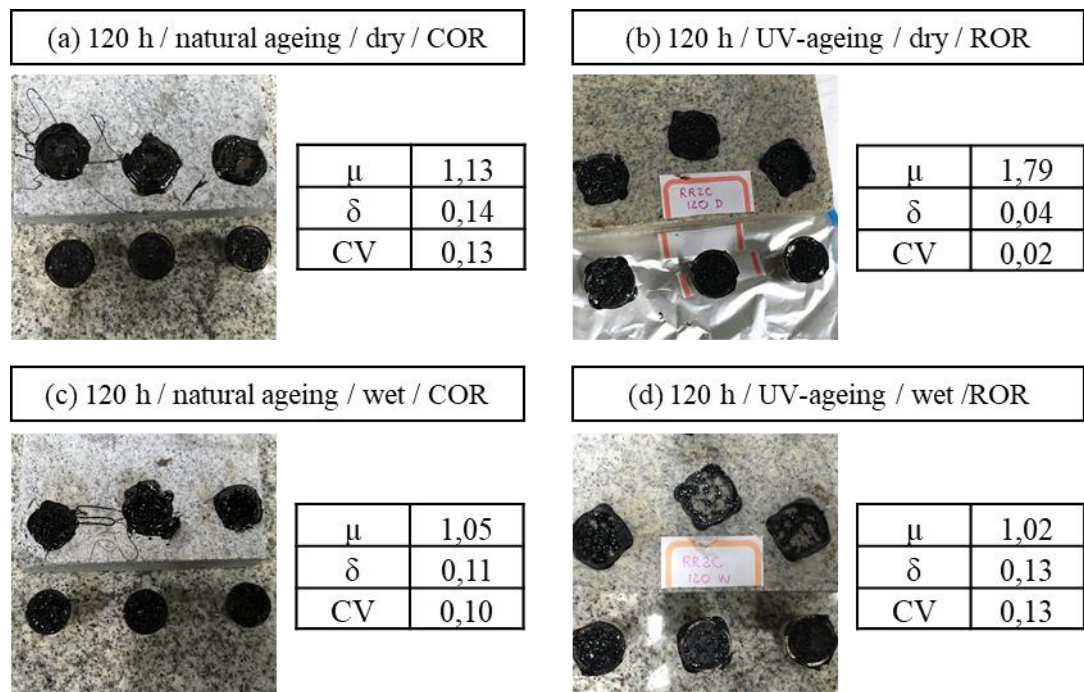


Source: author (2021)



For comparison purposes, these results were also related to what was obtained when the same material was aged in the UV controlled chamber separately from the substrate. Figure 37 presents the results obtained for each sample of the mentioned tests. In the sample aged in laboratory, there was a greater difference between the mean POTS obtained in the dry and wet conditioned tests (Figure 37a and b). In this case the RPOTS was 56.98 %. Also, in relation to the type of failure, there was difference in the wet conditioned tests. While in the natural aged samples the failure was entirely cohesive (Figure 37c), there were adhesive failures in the samples aged in the laboratory (Figure 37d). Thus, although the mean POTS in these cases were mathematically the same, it is not possible to affirm that there is relation.

Figure 37 – COR × ROR after aging



Source: author (2021)

### 3.5.5 Effects of bond strength on ravelling

It is known that in the early life of CS pavements, the ravelling distress is mostly related to the emulsion-aggregate bond (MORAES AND BAHIA, 2013). The late ravelling, on the other hand is mostly associated to embrittlement and strain-intolerance. Considering this information and regarding emulsions used in CS, it would be better recommended to evaluate aging by methods that give information about the evolution of the aging through the early life of the pavements, such as the UV method.

Also, regarding ravelling, Adams (2014) suggests a minimum value of BBS in certain conditions that are limits for good performance of emulsions used in CS pavements. Although these limits are based on the relation between bond strength and aggregate loss, it would be not adequate simply adopt this correlation for chip seals that uses different designs and aggregates with different characteristics. In fact, based on the bond obtained in the tests developed with the residues the materials would be adequate to ravelling performance in all levels of traffic, which contrasts with the information that the CS pavements in the state of Ceará are more susceptible to the distress of ravelling than bleeding (SILVA, 2018). However, the same methodology would be recommended for creating more reliable limits for CS designed in Brazil, especially in the state of Ceará.

### **3.6 Conclusions**

The main objective of this paper was to investigate how the bond between binder and aggregate evolves based on the aging of the binder. For this purpose, it was considered two methods to represent the aging that occurs in the binder throughout service-life: the UV aging and the PAV aging. Also, parameters associated to the bond strength were evaluated. In this perspective, we can outline some conclusions:

- i. The PAV aging was more effective in modifying the composition of the material and the parameters evaluated in this paper. This is related to the proposition of this test, which is to simulate the conditions of binders in the end of service life. Based on this, the UV aging is more recommended to evaluate phenomena that occurs in the early life of pavements, such as the relation between aggregate loss and bond strength.
- ii. Among all aging methods and binders, the POTS presented the tendency of increasing in relation to the unaged binder.
- iii. The BBS procedure could not be successfully applied to the aged samples of polymeric residues due to high viscosity at the temperature of test. This is also reflected in the low correlation obtained when analysing this material and in the tendency of behaviour differently from the unmodified binders.
- iv. In relation to the UV aging, little difference was observed between 120 and 240 hours of aging. Thus, there is indication that the increase of parameters that are related to this process does not follow a constant tendency.

- v. In dry conditioned tests, the  $|G^*|$  represented good correlation to POTS when analysing unmodified binders.
- vi. The oxidative mechanisms of aging increased the formation of functional groups such as carbonyl and sulfoxide that also correlated to POTS in dry conditioned tests.
- vii. In relation to the unmodified emulsion, there was no difference between the tests carried out with the residue cured on the substrate and isolated. However, when the aging methods were added to this analysis, there was divergence in the type of failure of wet conditioned samples: adhesive failure was obtained in the isolated aged residue.

### **Acknowledgement**

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## **4 PERFORMANCE OF CS: ANALYSIS OF AGGREGATE LOSS IN SURFACE TREATMENTS FOCUSING ON UNIFORMITY AND SHAPE PROPERTIES**

### **4.1 Abstract**

This paper aims to evaluate loss of aggregates in specimen of chip seal (CS) pavements. For this purpose, samples of CS constructed in laboratory were evaluated focusing on the defect of aggregate loss. Rapid-setting emulsion RR-2C and a granitic aggregate recommended for CS were used in the samples. The mixture was designed following national standards and set as reference mix. Next, seven different samples were designed maintaining the rates of emulsion and varying only the uniformity or the shape properties of aggregates. The eight mixes were tested in an adapted test of abrasion (WTAT). Results showed that aggregates with lower Performance-Based Uniformity Coefficient (PUC) and Flatness and Elongation ratio above 1:3 are recommended to use in CS coats.

### **4.2 General Considerations**

Efficiency in CS is related to the prevention of distresses that might lead pavements to failure. Among those, aggregate loss (raveling) and bleeding are frequently mentioned as the most common. Part of the occurrence of such distresses can be explained by parameters found in the materials.

For example, in relation to grain size of the aggregates, it is known that more uniform gradation leads to better performance. It is often mentioned that performance is also affected by shape properties of aggregates such as sphericity, angularity, and flatness. Frequently, these properties are analyzed using Digital Image Processing (DIP). There are plenty of applications to assist the analysis of aggregates using this resource. Part of them is particularly for aggregates used in construction of pavements and surface courses. In fact, the study of shape properties using DIP has been useful to achieve parameters of surface courses based on properties of aggregates.

Considering raveling, it is known that even when it is applied an emulsion with good adhesiveness to the aggregate, it is possible to occur loss of aggregates. This is related to the size and the arrangement of the particles of aggregate in the surface of pavement. Based on that, the analysis of shape properties of aggregates used in CS is an alternative to improve techniques of design and evaluation of coats based on this material and also to establish criteria for selection of aggregates for construction of new pavements.

Thus, the main objective of this paper is to evaluate the impact that changes in aggregate properties (shape and uniformity) have in both: the arrangement of the aggregates in the surface of CS coats and the performance concerning the problem of aggregate loss.

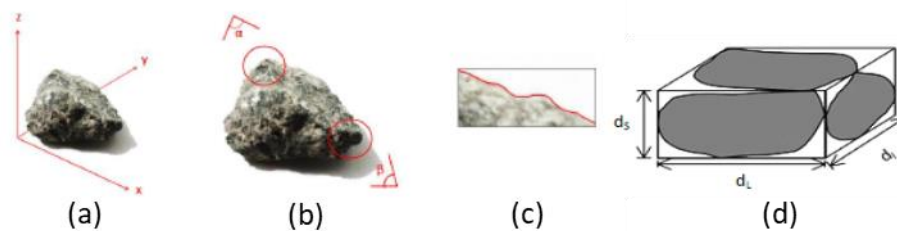
### 4.3 Literature Review

It is important to consider in the context of performance of CS factors such as: the type of aggregate, rate of binder and compaction. In relation to the role of aggregates, aspects such as mineralogy, shape properties and uniformity can explain the performance. Regarding the selection of this material for CS-type pavements, it is expected that aggregates present good texture, porosity, sphericity, and angularity (LEE and KIM, 2008). In addition, it is important to consider the size of particles, which indicates the thickness of the coat and the condition of its surface.

#### 4.3.1 Shape properties

The evaluation of shape of aggregates is commonly made by three properties: (i) sphericity, which is measured by the dimensions of each particle of aggregate; (ii) angularity, which refers to the edges created in the surface of the particles and (iii) microtexture, which refers to the irregularities of that surface when observed in microscopic scale (MASAD *et al.*, 1998). Figure 38a, Figure 38b and Figure 38c show the overview of the aspects of the aggregate measured in each property. Figure 38d shows a representation of a particle as a three-dimension block, used to calculate other properties such as flatness and elongation.

Figure 38 - Evaluation of shape properties in aggregates



Source: Masad *et al.* (1998)

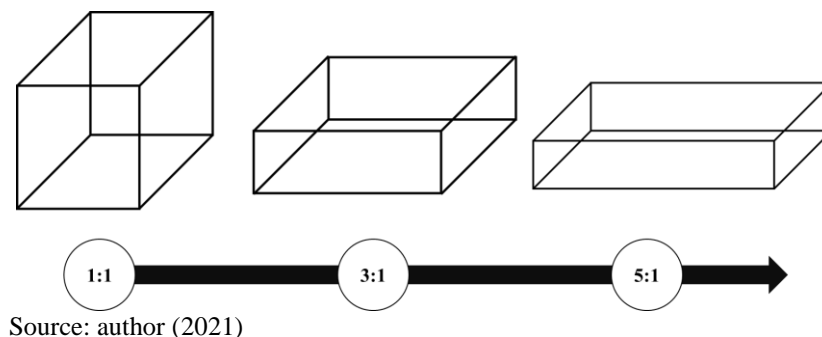
These properties commonly present relation to the performance of the services in which the referred aggregate is inserted. For example, it is expected good interrelation between shape properties and parameters obtained in surface courses, such as durability, workability, and performance. Based on that, this analysis is a good mean to infer the susceptibility of these materials to the occurrence of defects before mixture (ADAMS, 2014). For example, it is known

that sphericity relates to deformation of mixtures (ARASAN *et al.*, 2011) and that angularity and texture of gravel might explain part of the resistance of mixtures to rutting (PAZOS, SACRAMENTO and MOTTA, 2015).

It is commonly to study along with shape properties flatness and elongation of aggregates. These properties are obtained when analyzing particles in a simplified form, as a three-dimension block shown in Figure 38d. Those properties can be simply explained by the relations between the dimensions. The relation  $d_s/d_i$  between the shortest ( $d_s$ ) and the intermediate ( $d_i$ ) dimensions, for example, gives the flatness ratio. The elongation is given by the relation  $d_i/d_l$ , between the intermediate ( $d_i$ ) and the longest ( $d_l$ ) dimensions. Frequently it is used the relation  $d_l/d_s$  to demark the Flat and Elongated value (F and E) and the form of the particle (BESSA, CASTELO BRANCO, and SOARES, 2009). Based on the standard ASTM 4791 99 (ASTM, 1999), the aggregates can be defined as (i) flat, (ii) elongated or (iii) flat/elongated, depending on the properties of F and E.

In general, the construction of pavements requires particles of aggregate that has a form similar to a cube. Particles with high F and E value resemble a shape of plate and are more likely to crash. Figure 39 presents three examples of particles with different F and E values. The picture shows a line of growth of this ratio. The most critical F and E value is found in the particle of the right (5:1). The ratio 1:1, in the left, is also the lowest this parameter can achieve, representing a particle completely cubic. Considering there is no maximum value for this parameter it is frequently presented the inverted relation, in which 1 is the maximum F and E (referring to particle cubic 1:1) and 0 is the minimum ( $1:\infty$ ).

Figure 39 - F and E value in aggregates



The superpave method recommends that less than 10 % of the aggregate exceeds F and E ratio critical, set as 1:5. The ratios 1:2 and 1:3 were mentioned as better parameters to represent the flat/elongated characteristic of particles (PAZOS, 2015). Other research has presented improvement of performance when a lower ratio was considered.

Frequently, two particles of the same group of aggregate might show differences in shape properties. Due to this variability, tests to evaluate those properties must be performed in a representative sample to give a real indication of aggregate characteristics. Tests using DIP are prompt and accurate, usually representing the best alternative. One of the instruments used for this type of test is the Aggregate Imaging Measurement System (AIMS2), which gives analysis in two and three dimensions. Also, the software used for this test presents the results with statistical analysis (AL ROUSAN, 2004; IBIAPINA *et al.*, 2017). In this perspective, AIMS2 is also an appropriate instrument to categorize aggregates in the same region based on the most suitable service of engineering. In other countries, the effectiveness of this instrument has already been reported by paving agencies and local research (Gates *et al.*, 2011).

In the proceedings of this software, aggregates of the same size are tested together. After the test, it is possible to proceed the classification of each particle of a sample. This classification is made based on the origin of the aggregate. The categories are defined based on limits that each property must show to be adequate for service. Thus, it is recommended that each country or region determines a particular classification for its aggregates. As example, Ibiapina (2018) has presented a suggestion for national catalogue of classification of aggregates in early research.

#### **4.3.2 Uniformity**

The level of service of CS-type pavements is directly related to the selection of aggregates. Concerning this material, a parameter that influences performance as well as shape properties is the size of the particles. Based on that, the manuals of design and construction frequently present suggestions of granulometric ranges that are most appropriate to this service.

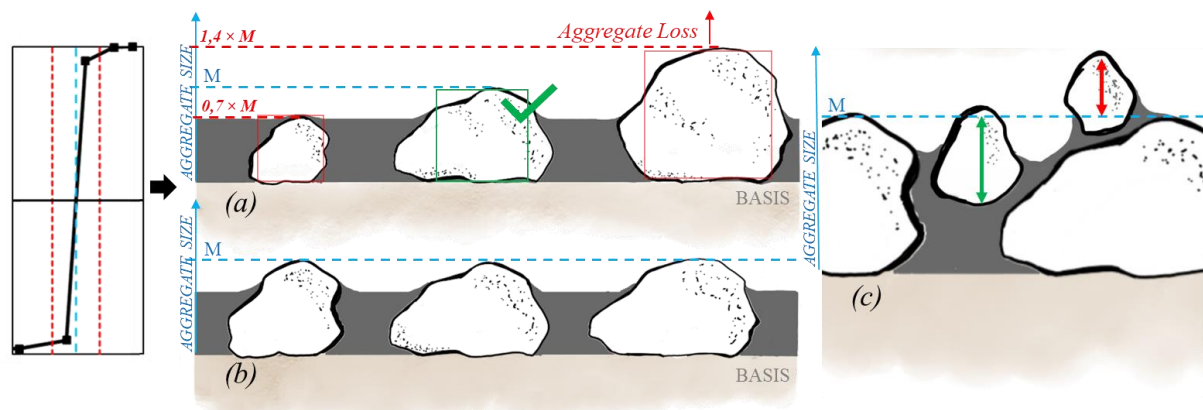
The rate of binder applied is also important for these specifications. It is known that for Single CS, a good performance is achieved in aggregates with at least 70 % of its surface covered by binder (MCLEOD, 1969; ADAMS, 2014). Based on this statement, it is possible to assume that the ideal binder rate for best performance is the one in which the maximum of particles is near this percentage. Consequently, particles that do not attend this criterion are more likely to lead the coat to failure. For example, when a particle is 100 % covered, it means it is below the surface of the coat, developing bleeding.

In contrast, particles that have less than half of its surface covered by binder, are more likely to be released from the coat, representing a critical condition for loss of aggregates. Considering this defect, it is possible to conclude that aggregates with lower variation of size

of particles are better recommended for CS considering they are more likely to achieve good performance.

Figure 40 shows an example of aggregate that was designed for Single CS. The range created by the red lines comprehends aggregates of ideal size, inserted in the interval of  $0,7 \times M$  and  $1,4 \times M$ , in which  $M$  is the median of the granulometric curve. Figure 40a shows a representation of three aggregates of critical size based on these criteria and Figure 40b represents three aggregates of the same width, adequate for this service. Figure 40c presents an event of overlay of aggregates. In this situation, the aggregates are more likely to be lost which is not ideal.

Figure 40 - Example of aggregate size using the McLeod (1969) criterion



Source: author (2021)

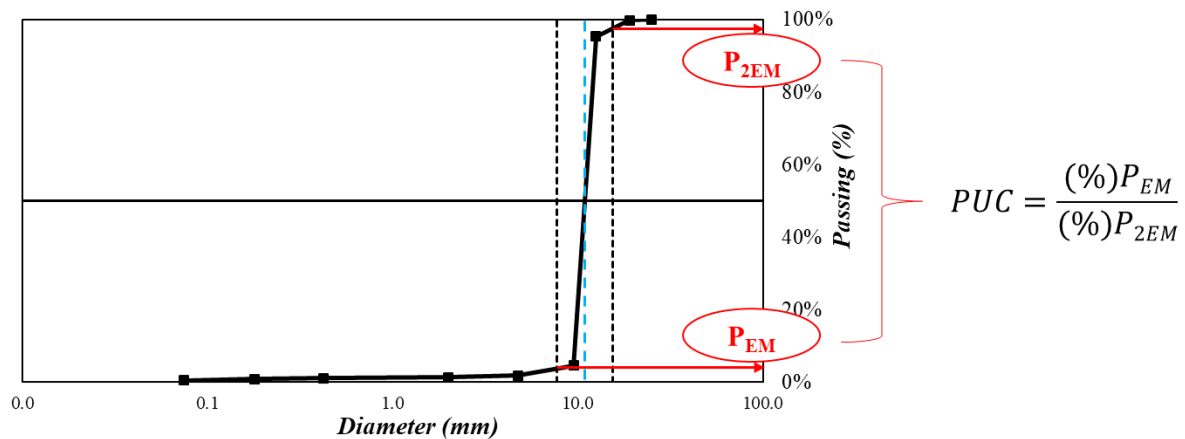
In this perspective, it is possible to understand that Single CS coats designed with uniform aggregates are more easily fit in the range of ideal size and consequently are more likely to lead pavements to good performance. Considering this relation, it is possible to identify the best aggregates for CS by using a coefficient of uniformity. The Performance-Based Uniformity Coefficient (PUC) is one coefficient created exclusively to make this type of selection (SILVA, BARROSO, and KIM, 2018; LEE and KIM, 2009). This coefficient is calculated analyzing, in the graph, the granulometric curve and the interval created by the diameters  $0,7 \times M$  and  $1,4 \times M$ .

Figure 41 shows how the PUC is calculated. The points in which the curve intersect the limits  $0,7 \times M$  and  $1,4 \times M$  refers, respectively, to the  $P_{EM}$  and  $P_{2EM}$  percentages, as shown in the image. In theory, (%) PEM relates to the chance of the pavement develop bleeding areas. Alternatively, “1 - (%)  $P_{2EM}$ ” is associated with defects such as aggregate loss. The calculation of PUC is made as presented in the image by the relation  $(\%) P_{EM} / (\%) P_{2EM}$ . The uniformity is



higher as the PUC is lower. Thus, limits of PUC are frequently presented for each type of aggregate as criteria for selection of this type of material.

Figure 41 - Calculation of PUC



Source: adapted from Lee and Kim (2009)

The PUC of each material can be easily calculated based only in the granulometry analysis. In addition, this coefficient shows good results when used to select aggregates for Single CS (SILVA, 2018). Based on that, one possibility to improve performance of CS pavements would be the definition of a limit of PUC for each type of aggregate as a criterion to select this material.

Considering national practice, the use of PUC tends to show good relation to the performance when the criterion is applied to Single CS. It is possible to indicate, for example, which specification of service recommends the best granulometric ranges for the aggregates (SILVA, 2018). Alternatively, there is no indicative that this coefficient is sufficient to select aggregates for CS of two or three layers.

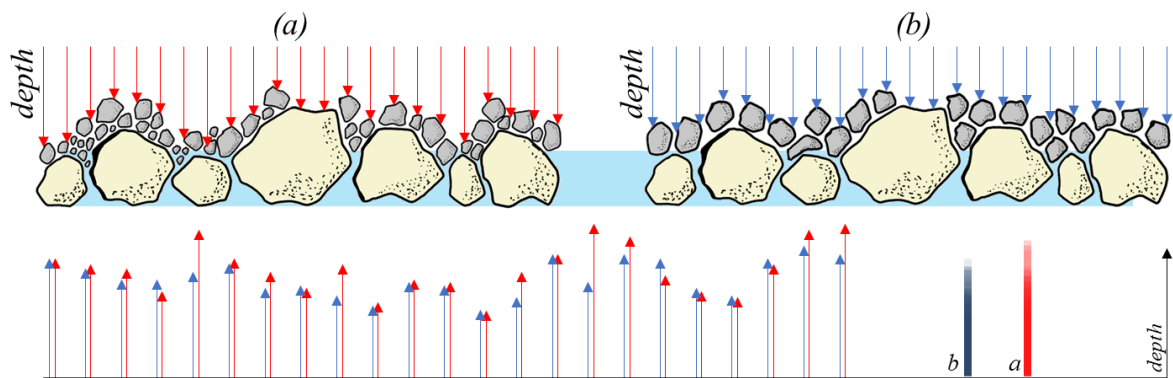
It is important to consider that, in double and triple CS, the upper layers tend to be designed to fill the empty spaces formed in the layers below. Since the PUC is calculated for each layer separately, it does not give enough pieces of information on how this interaction is developed. Considering that Double CS are more than half of the surface treatments constructed in some northeast states of Brazil such as Ceará, more information must be considered in the manuals of design of these regions concerning uniformity before selecting aggregates.

In general, to simplify the process of selection of materials, the manuals of design usually recommend that aggregates applied in Double CS have good uniformity in both layers. This would be also referred as a low value of PUC. However, there is no statement in the

specifications of a limit of PUC and how the performance should be affected, for example, if an aggregate of low PUC was placed over a layer of non-uniform aggregate.

Figure 42 shows an illustration of two Double CS in which: Figure 42a represents a non-uniform CS in the first layer with a non-uniform second layer; and Figure 42b represents the same first layer and a uniform distribution of aggregate on second layer. Analyzing the heights of surface, it is possible to see that, in this example, the surface of (a) presented more variation of peaks than (b). Making a parallel between this example and the criterion of uniformity used for Single CS, probably (b) would present better performance.

Figure 42 - Uniformity in the second layer of a Double CS



Source: author (2021)

Macrottexture has already been mentioned in research of CS as an indicator to evaluate performance of chip seals (ROQUE, 1991; AKTAS, KARASAHIN and TIGDEMIR, 2013). In fact, the mean texture depth was defined as a good parameter to indicate the evolution of performance during service life. Based on that, there is indication that a parameter obtained in the analysis of the macrottexture of the surface of the sample would be helpful to explain the interaction between two or more layers of the CS and consequently can provide information about the expected performance.

### 4.3.3 Aggregate Loss

Various tests can be used to quantify aggregate loss in a CS. Although the purpose of each one is similar, there are singularities in respect to the conditions replicated and consequently in the procedure. Various tests intend to analyze the coats in the early months when the pavement is first open for traffic. Agencies frequently report high rate of aggregate loss in this period due to emulsions not totally cured or bad compaction. Other tests include in

the procedure conditions that contribute to the increase of this defect in the pavement, for example, heating and freezing.

The tests mentioned in this section are, in general, procedures included in the design of CS treatments and can be performed in laboratory before the construction. In Brazil there is no national specification for a method of analysis of aggregate loss in laboratory. However, some authors have made efforts in the intention of validate an adaptation of the WTAT for CS (LOIOLA, 2009; PEREIRA, 2013; SILVA, 2018). This test has similarities to the Sweep Test (or Flip-over test, FOT), used by U.S agencies for the same purpose. The FOT is presented in the standard ASTM D7000.

The main test methods used for evaluating performance in terms of aggregate loss are summarized in Table 12. Based on the information given in the table, it is possible to compare the mechanism and conditioning procedure to the method of adapted WTAT which is often studied in Brazil.

Table 12 - Tests used to analyze aggregate loss in CS

<i>Test</i>	<i>Reference</i>	<i>Mechanism of test</i>	<i>Type of load</i>	<i>Conditioning</i>	<i>Total time</i>
<i>Adapted WTAT</i>	Loiola, 2009 Pereira, 2013 Silva, 2018	rotational and superficial	rubber	immersion in water	~ 20 minutes
<i>Flip-over test (FOT)</i>	ASTM D7000	rotational and superficial	brush	heating	~ 30 minutes
<i>Vialit adhesion test</i>	BS EN 12272-3 Lee, 2008	Axial (180°)	steel weight	-	-
<i>MMLS3</i>	Lee, 2007 Lee, 2008	Simulation	Pneumatic wheel	~25° C	> 2 hours
<i>HWT</i>	Boz, 2019	superficial	wheel	~19° C	-

Source: author (2021)

The techniques used in the construction of national pavements include still empiric criteria to determine the performance of treatments. Among the advantages of having a unique test to evaluate this defect is the possibility of comparing different types of treatments and improve the methods by, for example, inserting alternative materials and designs. For example, the test with adapted WTAT has been already applied in treatments of different types of design and is suitable for national practice. In addition, it is important to consider that the equipment for this test can be easily acquired and it is already used for most of the agencies, which makes the method more accessible.

## 4.4 Method

In the experimental plan developed in this chapter, RR-2C rapid-setting emulsions was defined as the asphalt binder. It was also obtained an aggregate which is natural from the same region and that also meets the recommendations of mineralogy suggested in prior research. Both materials were obtained in the state of Ceará – Brazil. After acquisition, the materials were tested in order to meet the requirements for CS practice. The materials were then submitted to the further tests defined in this paper.

### 4.4.1 Design procedure

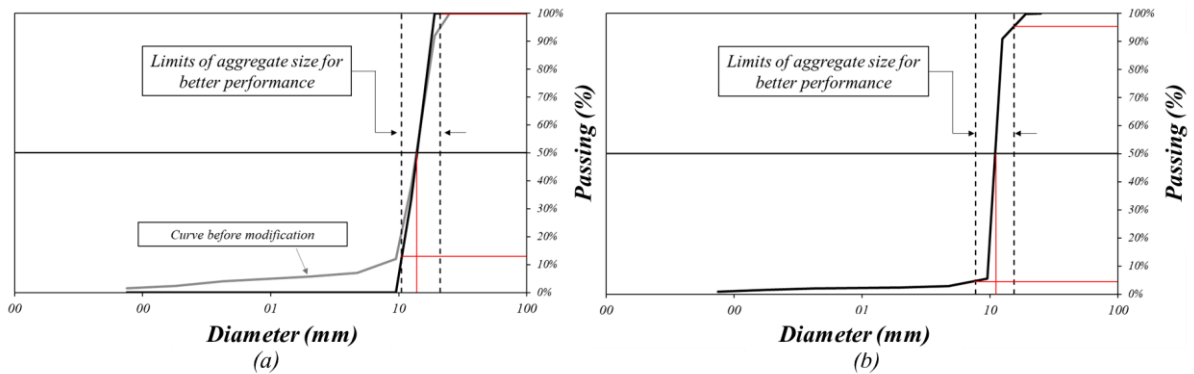
For the design procedure one mix was defined as reference. The other mixes were designed based on the mix of reference. As recommended by local statements of practice, the reference aggregate was organized in two different group of ranges. Firstly, it was produced an aggregate with a gradation of 3/4'' (referring to its nominal size). This material is designed to the first layer of Single, Double and Triple Seals. For the second layer it was produced an aggregate with a gradation of 3/8''. Those two gradations were also defined taking into consideration that services of CS in the state of Ceará are constructed mostly with two layers.

The aggregates were characterized in terms of granulometry, shape, density, absorption, and abrasion. In addition, the PUC of those aggregates was calculated based on granulometric curve. Those tests were used to evaluate the suitability of the aggregates for services of CS and, for that purpose, limits were previously defined for the coefficients obtained in the tests to accept these materials.

Figure 43 presents the corresponding curves of size distribution for the aggregates of reference. In each curve it was also presented the interval defined by the  $P_{EM}$  and  $P_{2EM}$  parameters, referring to aggregate best performance and used in the calculation of PUC. In Figure 43a, it is shown the curve of aggregate chosen for the first layer. In the first analysis, the PUC obtained for this material was 0,232. Based on previous research that also utilized this parameter in the evaluation of performance, it was defined in this research a PUC maximum of 0.200 as a criterion for accepting aggregates. Based on that, the aggregate of first layer had to be submitted to a process of fraction.

After fraction, the aggregate of first layer presented PUC of 0,120. The PUC of the second-layer aggregate was 0,053. Thus, this material was accepted without modification. Figure 43b shows the curve for the second layer.

Figure 43 - Granulometric curves of the reference mix



Source: author (2021)

The rate of aggregate in each layer of the mix designed was calculated and presented in unities of volume/area, as outlined by the construction practice. These rates were obtained using two parameters: the specific mass ( $\text{kg}/\text{m}^3$ ) of aggregates and the area density ( $\text{kg}/\text{m}^2$ ), calculated using a tray of determined dimensions and spreading the maximum of aggregate in its area (Figure 44). The rate of aggregate ( $R_{agg}$ ) was then determined in  $\text{L}/\text{m}^2$  using these parameters. The rate of emulsion ( $R_{em}$ ) was calculated by the relation  $R_{em} = 0,112 \times R_{agg}$ , often used by local agencies.

Figure 44 - Determination of rate of aggregate using tray



Source: author (2021)

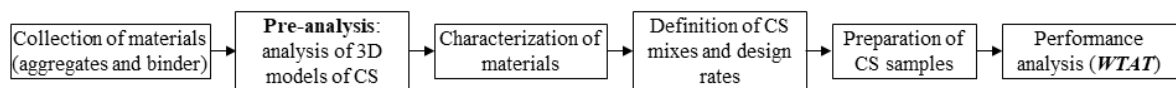
#### 4.4.2 Experimental plan

The experimental plan for this paper was subdivided in two parts. Firstly, a pre-analysis of surface was made with two different aggregates designed following the recommendations of two agencies of Brazil: The National Department of Transport Infrastructure (DNIT) and the local agency of the state of Ceará (DER-CE). Concerning the analysis of the surface of the coats, 3D models were digitally constructed. For this purpose, three applications were used: the

Agisoft Photos can and the Meshlab were used for aligning the pictures and the axis of the models; and the Mountains Map (Digital Surf) was used for analyzing the models.

The second part was subdivided in the steps shown in Figure 45. The first step is the collection and selection of materials (aggregates and emulsion). The second step was the characterization of the materials. After the materials were selected attending the first requirements, the characterization was proceeded based on the local recommendations for CS. Along with the conventional characterization of the aggregates it was performed the analysis of adhesion using BBS and the analysis of sphericity, angularity, flatness and elongation in AIMS2. Each fraction of the aggregates was classified based on the national method of classification (Ibiapina, 2018).

Figure 45 – Flow chart of the experimental plan (chapter 4)

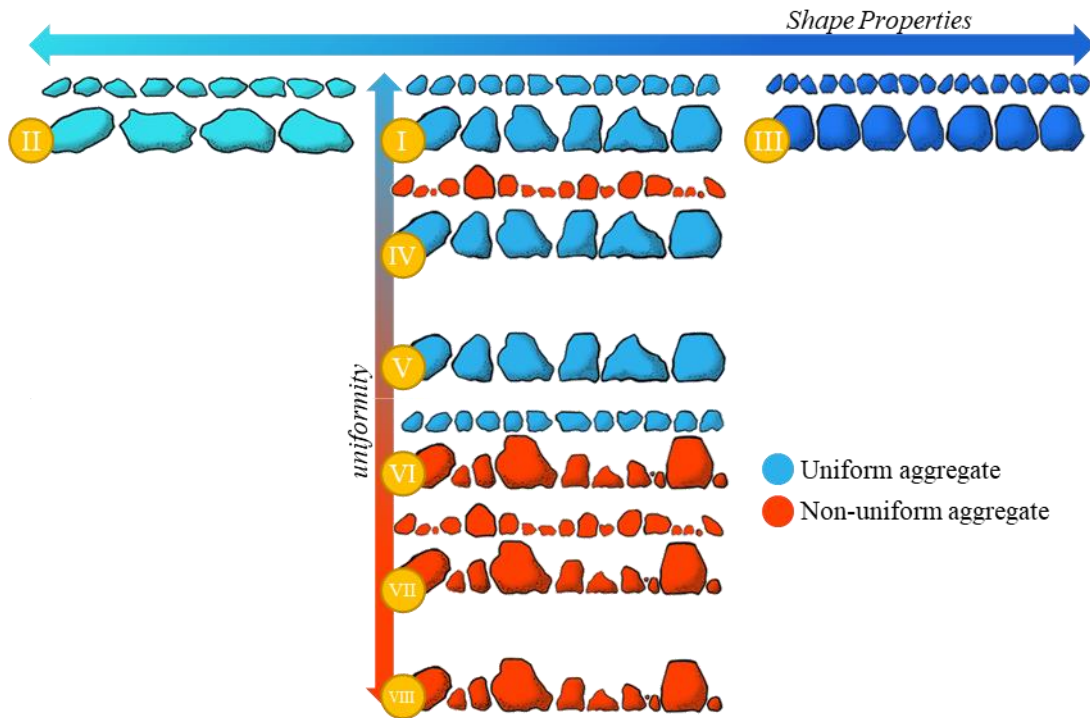


Source: author (2021)

The following step was the definition of samples and the design rates. In this part, the aggregate was fractioned and then the mixes were defined according to the percentage of each fraction in the first and the second layers. A reference mix was established according to both the criteria of PUC (SILVA, BARROSO, and KIM, 2018; LEE and KIM, 2009) and the gradation suggested by the local specifications (DER-CE, 2000). After that, this material was modified in terms of shape properties and uniformity to create seven new samples (Figure 46).

In relation to the samples created varying the shape properties, two samples were created maintaining the gradation and uniformity of the curves. Sieves of rectangular and circular openings were used for selecting more cubic and less cubic aggregates. These sieves are used to the evaluation of shape index and specified in the national standard DNER-ME 086/94 of this test and each sieve was selected according to the corresponding fraction of the aggregate. The passing material was identified as “less cubic” (II) and the material retained in the sieves was identified as “more cubic” (III).

Figure 46 - Overview of the studied mixes



Source: author (2021)

The other test samples were created changing the uniformity of the aggregate layers. Two new non-uniform gradations were created not attending the criteria of PUC: one for first layer and another for second layer. A specification that recommends non-uniform gradations was selected among the standards currently used in Brazil. The specification of the National Department of Transport Infrastructure (DNIT) was used for this purpose. Table 13 summarizes the composition of each aggregate.

Table 13 - gradation used in specimen

(mm)	<i>Uniform</i>		<i>Non-uniform</i>	
	<i>1st layer</i>	<i>2nd layer</i>	<i>1st layer</i>	<i>2nd layer</i>
19.00			5 %	
12.70	67 %		55 %	
9.50	33 %	10 %	35 %	10 %
4.80		90 %	5 %	65 %
2.00			0 %	20 %
200.00				5 %

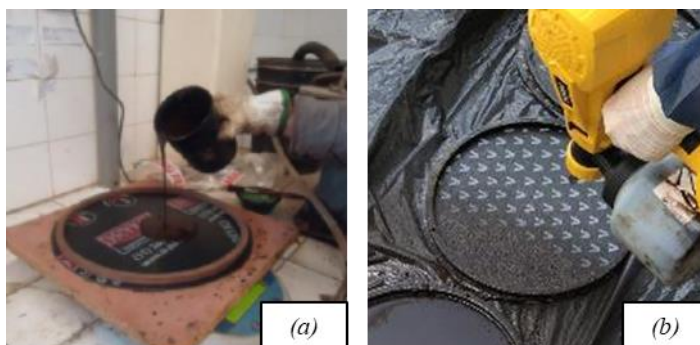
Source: author (2021)

Five test samples were created mixing the aggregates created: (IV) a double CS with uniform aggregate on first layer and non-uniform aggregate on second layer; (V) a single CS

with a layer of uniform aggregate; (VI) a double CS with non-uniform aggregate on first layer and uniform aggregate on second layer; (VII) a double CS with non-uniform aggregate on first layer and non-uniform aggregate on second layer; and (VIII) a single CS with a layer of non-uniform aggregate.

The test samples were designed according to the local specifications (DER-CE, 2000). The procedure used in the construction of the specimens in laboratory was the one created by Loiola (2009) and reviewed by Pereira (2013) and Silva (2018). Additionally, for spreading the emulsion, it was used a paint spray gun of rate 700 ml/min. Adams (2014) recommends for this purpose an equipment of approximate rate of 340 ml/min or higher, when applying unmodified emulsions. Figure 47 presents a comparison between the two mechanisms of spreading emulsion. Figure 47a is the spread of emulsion making use of beaker and paintbrush while Figure 47b refers to the use of paint spray gun. It is expected that the second would represent better the spreading of emulsion that occurs in the construction of CS coats.

Figure 47 - Spreading of emulsion – paintbrush (a) × spray gun (b)



Source: Pereira (2013) and Author (2021)

Lastly, for evaluating aggregate loss it was used the WTAT adapted for CS coats. The curing time was 24 hours, and the temperature of application of binder and cure of samples was 60° C. The conditioning method was the immersion in water for 30 minutes and the time of test was 3 minutes.

## 4.5 Results

### 4.5.1 Pre-analysis: analysis of surface of CS (3D)

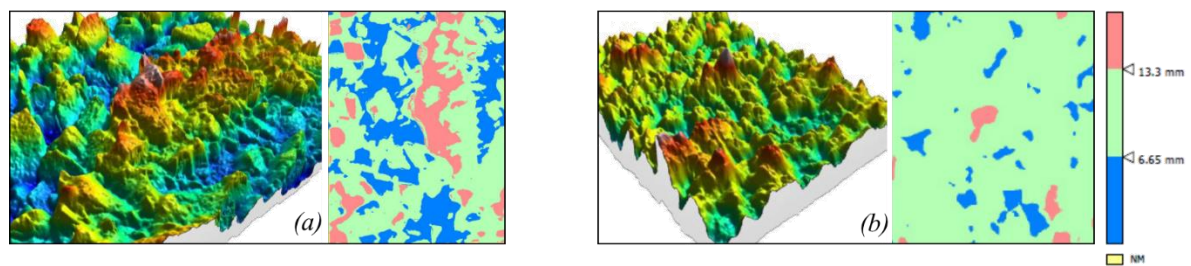
The analysis of surface of the CS was carried out using 3D models (MESQUITA Jr. *et al.*, 2019). For this purpose, two different aggregates framed respectively in the “uniform” and in the “non-uniform” gradation were used. It is important to highlight that in this pre-analysis only the gradation and the macrotecture of the aggregates were taken into consideration.



The models were created from a group of pictures taken in the specimen. The software used for creating the models (format “. xyz”) was the ReCap Photo. For analyzing the models, a different software was used: the MountainsMap Premium (Digital Surf). In this software various texture parameters can be obtained, including those referred in the ISO 25178-2/12. Among those, there are parameters that present high correlation to the operational performance of surfaces (FRANCO and SINATORA, 2015; BLATEYRON, 2013). For obtaining performance of the samples, it was used the Wet Track Abrasion Test (WTAT). Then, the texture parameters were used to explain the phenomenon of aggregate loss.

One improvement on the analysis of CS using 3D models is the possibility to identify valleys and peaks in the samples. For example, Figure 48 presents the analysis of two specimen of different CS samples: Figure 48a refers to a specimen of Single CS and Figure 48b, a Double CS. In the analysis, the topography of each model was sectioned in heights corresponding to the limits defined by the criteria of McLeod. With this tool, it is possible to remark areas with peaks, corresponding to aggregates larger than the ideal for CS, and areas corresponding to aggregates smaller than the limits. Comparing the composition of these two samples, the surface of the Double CS presented less occurrence of critical areas, which is expected considering the better performance of double CS.

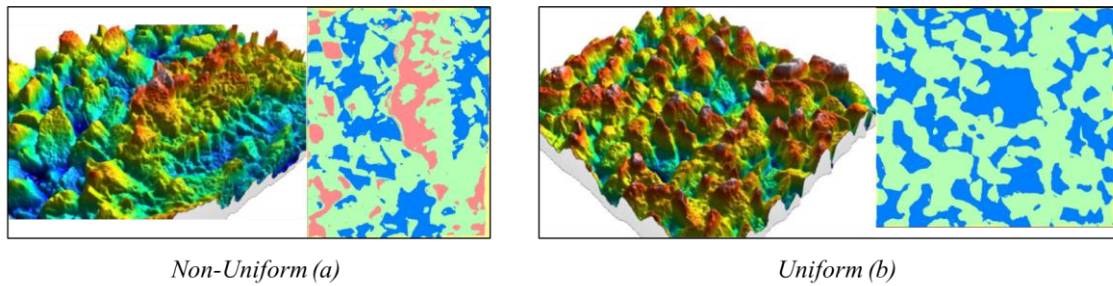
Figure 48 – Analysis of 3D models of Single CS (a) and Double CS (b)



Source: adapted from Mesquita Jr. *et al.* (2019)

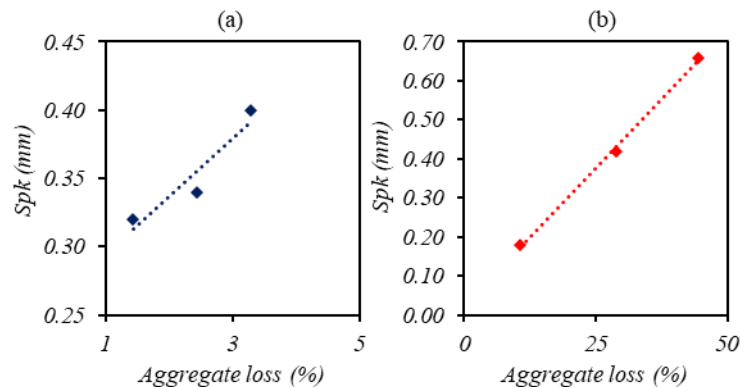
When it comes to the uniformity, it was also obtained different aspects in the analysis of the surface in the 3D models. Figure 49 presents the evaluation of peaks and valleys in obtained in Single CS constructed with different gradations. It is notorious that, in CS constructed with a single layer, the use of a non-uniform gradation reflects in a higher percentage of peaks and consequently in areas which are more susceptible to loss of aggregates.

Figure 49 – Analysis of surface of Single CS varying gradation



Source: adapted from Mesquita Jr. *et al.* (2019)

In this perspective, a combination of uniform and non-uniform layers was made to evaluate the surface when a second layer of aggregates is considered in the coat. In the analysis of the texture parameters of different samples with the same type of gradation in the first layer, it was obtained a good correlation between the parameter Spk, corresponding to the height of peaks above the core, and the loss of aggregate. Figure 50 presents the graphs corresponding to samples in which the aggregates used in the first layer were similar.

Figure 50 –Spk  $\times$  aggregate loss (%) using uniform (a) and non-uniform (b) first layer

Source: adapted from Mesquita Jr. *et al.* (2019)

#### 4.5.2 Characterization

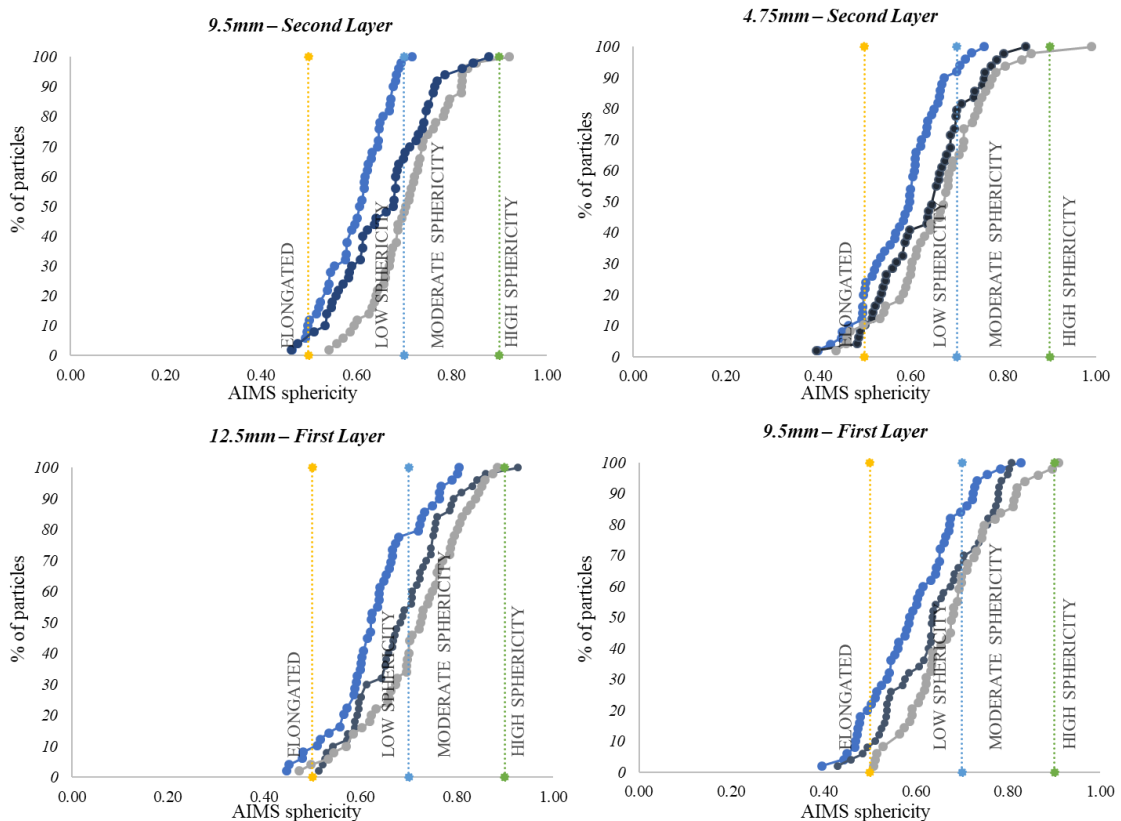
The aggregates were tested regarding to gradation, adhesiveness, and abrasion to guarantee the materials met the current requirements for use in CS treatments. The shape properties were analyzed using DIP, as mentioned before. Section 3.4.1 *Comparing residue on rock (ROR) and curing on rock (COR)* summarizes results of BBS test obtained for 24 hours of conditioning using the same unmodified emulsion applied in this chapter.

It was observed that the selecting process of more cubic and less cubic aggregates reflected on modifications on the properties of sphericity and flatness and elongation. The materials were classified following the national classification suggested by Ibiapina (2017).

Figure 51 summarizes the results of sphericity obtained in the analyze of aggregates. In all fractions a similar behavior was observed: after the process of selection of more cubic aggregates, the curves of sphericity tend to move in the direction of high sphericity. The material of the fraction 12.5 mm presented in the natural form a mean sphericity of 0.69, placing in in the class of low sphericity (between 0.5 and 0.7). The selection of more cubic materials increased the mean for 0.72, changing the classification for moderate sphericity. In relation to the fraction 9.5 mm of the first layer, there were no changes in the classification of sphericity.

In relation to the second layer, the fractions 9.50 mm and 4.75 mm were analyzed. The same tendency of movement of the curve of sphericity in the direction of high sphericity for the more cubic aggregate was observed. In these fractions, while the less cubic material accommodates mostly in the range of low sphericity, the more cubic presented more than half of the observations in the range of moderate sphericity.

Figure 51 – Sphericity results



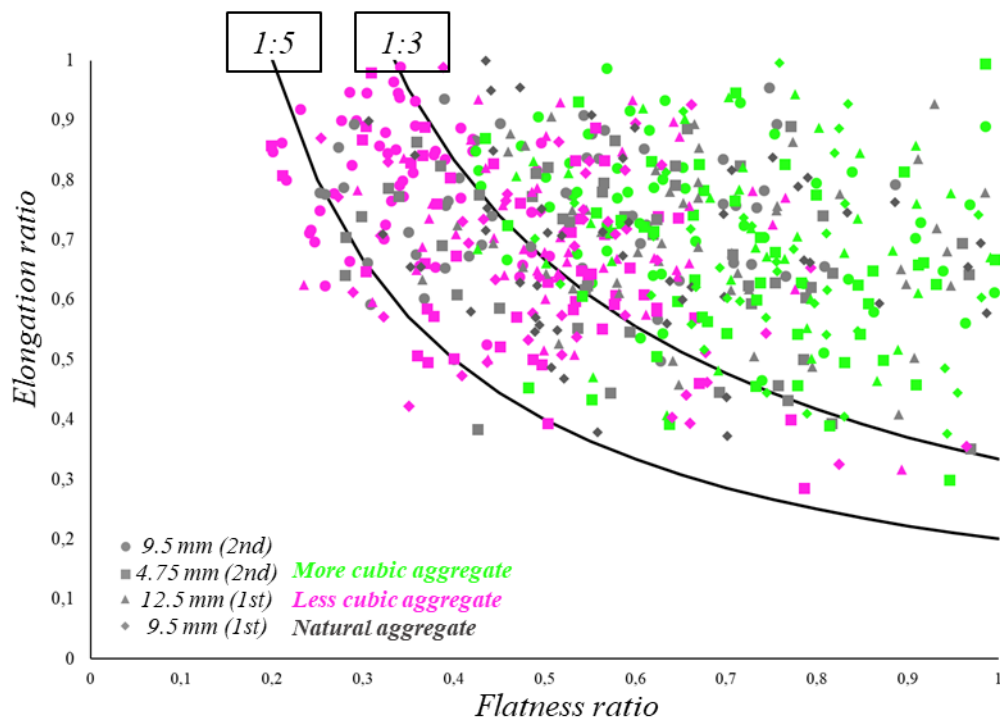
Source: author (2021)

The flatness and the elongation of the aggregates were also analyzed. Since it was used a sieve recommended to evaluate shape index, it was expected that the process of selection of more cubic aggregates would reflect in this analysis indicating materials closer to the F and E

relation 1:1. All the observations were presented in *Figure 13* in a graph. In this graph, the x-axis is the flatness ratio, and the y-axis is the elongation ratio. The curves corresponding to the F and E relations of 1:5 and 1:3 are also presented. In relation to national specifications of CS there is no criteria for limit of F and E for the aggregates, although there is recommendation for the use of more cubic materials.

In *Figure 52*, the aggregates identified as less cubic presented points below the curve of 1:5 and most of the observations arranged between the curve 1:5 and 1:3. In this perspective, although these materials were identified as “flake” particles, they would attend to the criteria of 1:5 recommended for asphalt mixtures. Alternatively, it was observed that the process of selection of cubic aggregates using the sieves recommended for analyze shape index was more effective in selecting materials with F and E relation above 1:3.

Figure 52 – F and E results



Source: author (2021)

#### 4.5.3 Design of CS samples

The rate of aggregate of each layer was calculated by the method defined in the experimental plan. Table 14 presents the rates calculated for each sample. When samples constructed by the combination of two types of aggregates (uniform or non-uniform) were

considered, the rates were maintained similar to those calculated in the samples in which the layers were designed with similar characteristics of uniformity.

Table 14 – Rates of aggregate in each sample

<i>Sample</i>	<i>1</i>	<i>2</i>	<i>3</i>	<i>4</i>	<i>5</i>	<i>6</i>	<i>7</i>	<i>8</i>
<i># layers</i>	2	2	2	2	1	2	2	1
<i>shape</i>	-	less cubic	more cubic	-	-	-	-	-
<i>uniformity</i>	U + U	U + U	U + U	U + N	U	N + U	N + N	N
<i>1st layer (kg/m<sup>2</sup>)</i>	12.80	11.37	15.44	12.80	12.80	15.06	15.06	15.06
<i>2nd layer (kg/m<sup>2</sup>)</i>	3.00	3.25	3.25	4.37		3.00	4.37	
<i>total (kg/m<sup>2</sup>)</i>	15.80	14.62	18.69	17.17	12.80	18.06	19.43	15.06

Source: author (2021)

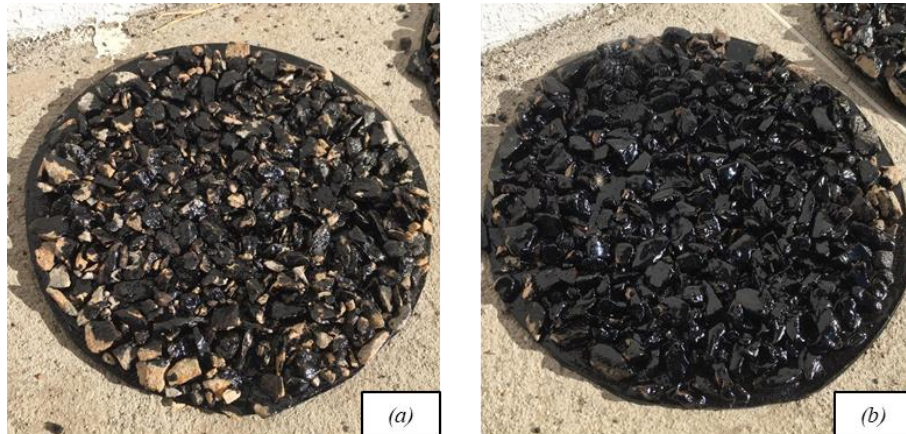
Samples 1, 2 and 3 refer to those in which there was change in the shape properties of the aggregates and the uniformity (PUC) was maintained the same. In relation to these samples, it was observed that the “more cubic” material (Sample 3) presented the highest rates of aggregates among those samples (18.69 kg/m<sup>2</sup>). Alternatively, the aggregate flatter and more elongated (Sample 2) presented lower rates of aggregates (14.62 kg/m<sup>2</sup>). In fact, this rate was the lowest among all double CS samples. Based on the common relation between rate of binder and rate of aggregate recommended in the specifications, it is possible to indicate that coats designed with more cubic aggregates tend to consume more emulsion and are, consequently, more expensive when it comes to cost of construction.

In relation to the samples designed with variation in the uniformity of the layers (Samples 1, 4, 5, 6, 7 and 8) it was observed that samples in which it is used non-uniform aggregates tend to present higher rates of this material. One explanation for this may be the fact that those gradations suggest the use of aggregates with higher diameter. Considering for example Samples 7 and 8, constructed exclusively with non-uniform aggregates, it was obtained a higher consumption of aggregate when comparing to samples 1 and 5, constructed with uniform aggregates. However, compared to the national specification (DNIT, 2011), all the experimental rates were inferior to the theoretical rates suggested in this specification, which is around 20 and 35 kg/m<sup>2</sup> in total.

The rates of emulsion were calculated for the samples 1 and 5, which are the samples that make use of uniform aggregates with no modifications on the shape properties. Those rates were then repeated for the other samples in order to eliminate the effects of the rate of binder in the final performance. Thus, the total rate of binder in the samples of double CS was 1.34 L/m<sup>2</sup> and for single CS this total rate was 1.18 L/m<sup>2</sup>. Then, it was calculated the time of flow of

the spray gun based both in the area of the specimen and the capacity of the equipment. Figure 53 shows a comparison between the surface of specimens constructed with the spread of emulsion using paintbrush (Figure 53a) and the spray gun (Figure 53b). It was observed that the use of the spray gun reflected in a better covering of the surface of the specimen.

Figure 53 – Comparison between paintbrush (a) and spray gun (b) in specimen surface



Source: author (2021)

#### 4.5.4 Evaluation of aggregate loss (WTAT)

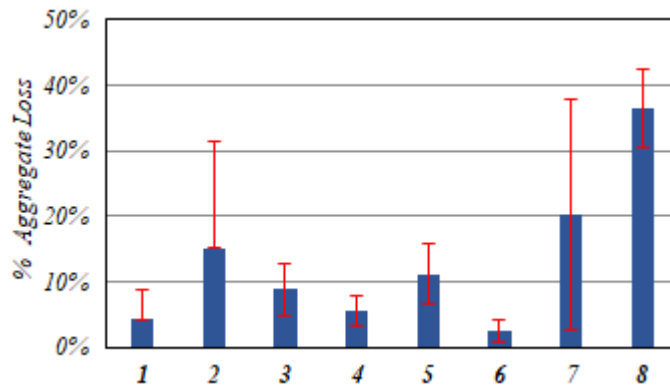
Table 15 presents the results of loss of aggregate obtained using the WTAT test. The mean abrasion and the standard deviation are presented in Figure 54.

Table 15 – WTAT results in samples

<i>Sample</i>	<i>1</i>	<i>2</i>	<i>3</i>	<i>4</i>	<i>5</i>	<i>6</i>	<i>7</i>	<i>8</i>
<i># layers</i>	2	2	2	2	1	2	2	1
<i>shape</i>	-	less cubic	more cubic	-	-	-	-	-
<i>uniformity</i>	U + U	U + U	U + U	U + N	U	N + U	N + N	N
$\mu$	4.20%	15.08%	8.87%	5.50%	11.14%	2.43%	20.27%	36.52%
<i>sd</i>	4.48%	16.29%	3.96%	2.33%	4.64%	1.74%	17.71%	5.98%
<i>CV</i>	1.07	1.08	0.45	0.42	0.42	0.72	0.87	0.16

Source: author (2021)

Figure 54 – Comparison of WTAT aggregate loss (%) in samples



Source: author (2021)

Among the samples tested with change in the shape properties (Samples 1, 2 and 3) it was observed that Sample 1 and 3 presented better performance than Sample 2. It is relevant to highlight that Sample 2 was composed of particles with F and E value between 1:3 and 1:5, which is acceptable for some specifications of asphalt mixture. However, this sample presented a high percentage of abrasion. It was also observed a high standard deviation, indicating that the organization of aggregates of this type in the surface of the coats may occur differently affecting the performance. Also, in relation to this sample it was observed the breaking of the aggregates in the phase of compaction, indicating low resistance of materials with these conditions.

In relation to the Samples 1, 4 and 5, designed with uniform aggregate in the first layer, all samples presented in average acceptable performance (below 20 % of aggregate loss). It was also observed that the use of a second layer tends to improve the performance of this material, independent of the PUC of this material. Statistically, it is not possible to affirm that there is a difference between the performance of Sample 1 and Sample 4.

When it comes to the samples designed with a non-uniform aggregate in the first layer (Samples 6, 7 and 8) a different behavior was observed. Sample 8, for example, presented the poorest performance among all tested samples. The mean aggregate loss in this sample was 36.52 %. In comparison to the other sample of Single CS, this was an abrasion around 3 times higher. Alternatively, it was also observed that there was improvement of performance with the addition of a second layer to this material. Sample 7, for example, presented a noticeable decrease in the percentage of aggregate loss. However, this percentage is still higher than the one obtained in the other test samples. On the other hand, the addition of a uniform aggregate to this material improved considerably the performance of the coat. In fact, the mean loss of aggregate in this sample (Sample 7) was the lowest among all samples (2.43 %). This indicates

that the use of a uniform aggregate in the second layer improved more the quality of the surface of the coat than a non-uniform aggregate.

#### **4.6 Conclusions**

The main objective of this paper was to evaluate the impact that changes in aggregate properties (shape and uniformity) have in both: the arrangement of aggregates in the surface of CS coats and the performance concerning the problem of aggregate loss. Based on this and on the discussion, we may highlight the following conclusions and recommendations:

- i. The use of uniform aggregates is preferred in single CS and in double CS to avoid the distress of aggregate loss. In the test of WTAT, the coats of single CS constructed with uniform aggregates presented a performance around three times better. When it comes to the double CS this improvement of performance is increased to around five times, indicating the preference for this type of aggregate.
- ii. When mixing aggregates with different uniformity, there is an indication that the use of an aggregate with lower PUC affects positively the performance of the coats. In this case, an analysis of surface is recommended to evaluate the interaction of the two layers and the organization of the particles.
- iii. The use of a paint spray gun is more effective when constructing the specimen due to a better capacity of covering the surfaces compared to the paintbrush. Based on the analysis of the authors, it is recommended that the construction of samples with this equipment occurs in groups depending on the capacity of volume of emulsion that the equipment can comport.
- iv. The F and E ratio of 1:5 is not recommended for CS designed by the methods used in this experiment. A ratio of 1:3 would select more adequate aggregates to this use.
- v. The use of parameters of texture collected with 3D models can help to comprehend the organization of the particles in the surface of CS coats. Among those, the Spk presented good correlation to the defect of aggregate loss, when applied to coats designed with the same aggregate in the first layer.

#### **Acknowledgement**



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## 5 CONCLUSIONS

This master thesis had as main objective to propose criteria for selection of aggregates and binder for CS Coats aiming to achieve best performance and reduce functional problems such as aggregate loss and bleeding. Based on this, an experimental plan of three parts was developed approaching individual aspects of the construction of CS coats separately. The first two papers focused on the performance of emulsions employed in CS coats during service life, analysing rheological parameters and binder-aggregate bond, respectively. The third paper presented an analysis of aggregate loss in CS samples in the perspective of aggregate parameters such as uniformity and shape properties.

Each chapter presents a conclusion section in which it is proposed and discussed the main conclusions obtained in the part of the experimental plan developed in the chapter. The following sections present the main conclusions obtained in each paper.

### 5.1 Main conclusions obtained in chapter 2

This paper had as main objective to investigate the effects that aging mechanisms present on rheological parameters of asphalt binders, in particular, those that are related to the performance of CS coats.

It was possible to conclude in this experiment that the UV radiation affects the properties of the emulsion residue, including those related to the performance of seal coats. Among those, a point of attention is the non-recoverable compliance ( $J_{nr}$ ), that is related to the distress of bleeding. This parameter decreased describing a non-linear curve when exposed to the UV-induced ageing. This is important to understand how this phenomenon evolves during service life when exposed to ageing mechanisms such as UV radiation.

### 5.2 Main conclusions obtained in chapter 3

The main objective of this paper was to investigate how the bond between binder and aggregate evolves based on the aging of the binder. In chapter 3, it was considered two methods to represent the aging that occurs in the binder throughout service-life: the UV aging and the PAV aging. Also, parameters associated to the bond strength were evaluated.

Some conclusions could be made in this experiment. In general, the PAV aging was more effective in modifying the composition of the material and the parameters related to adhesiveness and cohesiveness. Alternatively, the UV-induced ageing affected these

parameters in a lower intensity. In both situations, the Pull off Tensile Strength presented the tendency of increasing in relation to the unaged binder.

In the tests with the unmodified emulsion, there was no difference between the tests carried out with the residue cured on the substrate and isolated. The same was not observed in aged samples. This is also a point of attention, considering that in chip seals the break and cure of emulsion occur when the emulsion is in contact to the aggregate.

### **5.3 Main conclusions obtained in chapter 4**

The main objective of this paper was to evaluate the impact that changes in aggregate properties (shape and uniformity) have in both: the arrangement of aggregates in the surface of CS coats and the performance concerning the problem of aggregate loss. It was possible to conclude in this chapter that uniformity and shape properties must be considered in the design of coats to achieve best performance.

Uniform aggregates are preferred in CS coats to avoid the distress of aggregate loss. In the tests carried in this paper, coats constructed with uniform aggregates presented a performance around three (Single CS) and five (Double CS) times better. The Flatness and Elongation must also be considered. A F and E ratio of 1:5 is not recommended for CS designed by the methods used in this experiment. A ratio of 1:3 would select more adequate aggregates to this use.

### **5.4 General considerations**

Taking into consideration the main conclusions obtained in each paper, the following items present relevant points of attention among the conclusions obtained in this thesis:

- i. The UV-induced is a beneficial apparatus for the analysis of aging in CS coats. In some respects, this mechanism approximates more to the natural aging in this type of coats than other laboratory methods such as PAV, especially when it comes to the analysis of the first months of service, which were found to be critical to the performance of the coats.
- ii. During the period of test of 240 hours proposed in the experimental plan, the evolution of aging was found to be more intense in the first 120 hours. This was observed, for example, in the variation of the parameters  $J_{nr}$  and POTS, which are relevant for evaluating respectively bleeding and aggregate loss.

- iii. Among the samples of binder that were tested in the aging methods, the polymeric emulsion was the material that presented the most unexpected behaviour. This is probably due to the effects of the UV radiation and heating in the composition of the polymer content in the binder.

## 5.5 Recommendations for future work

Based on the conclusions discussed in this thesis, some suggestion might be proposed for future research. In this perspective, this section presents some recommendations for future work. It is also relevant to mention that, considering the specification for construction of CS coats in Brazil still outdated, this section is also organized aiming to help the efforts of construction of a nationwide manual of best practices for CS pavements.

Firstly, the following recommendations regarding the UV aging analysis in laboratory:

- i. Replicate the UV aging procedure with different conditions and include the effects of moisture cycles.
- ii. Extend the period of test for a period longer than 240 hours and investigate if there is variation in the main parameters.
- iii. Make an analysis of sensibility of the parameters of the weathering chamber such as UV incidence, irradiation frequency and temperature and give information about each one's relevancy in the variation of parameters.
- iv. Replicate the UV aging procedure to different types of emulsion, including those that are not proper for CS coats.
- v. Replicate the UV aging procedure to samples of CS built in laboratory and evaluate the evolution of parameters of performance such as percentage of aggregate loss and bleeding.
- vi. Reproduce the method developed in chapters 2 and 3 to a larger number of binders and create correlations of  $Jnr \times$  bleeding and  $POTS \times$  aggregate loss more adequate for national emulsions.

Also, these are technical recommendations for laboratory tests using CS samples:

- i. The use of spray gun is an adequate technique to simulate the spreading of emulsion. This technique showed more satisfactory recovery when compared to the use of paintbrush. For this purpose, a table spray gun of average

performance (700 ml/min) can be used. For the best use of the equipment, it is recommended to clean the inner tube to guarantee uniform spread of material.

- ii. The use of a national system to classify aggregates is useful to comprehend characteristics of aggregates used in CS coats.
- iii. The F and E ratio of 1:3 presented more efficient results than 1:5 for selecting aggregates for CS coats.
- iv. The use of more uniform aggregates is recommended for Single CS and for the first layer of Double CS for better performance.
- v. The parameter Spk is recommended for evaluating the surface propensity to develop the distress of aggregate loss in Double CS.

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