ADRIAN GRASSON FILHO

Evaluation of the specific surface method as a tool to determine the asphalt content of fine aggregate matrices (FAM)

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Advisor: Prof. Dr. Adalberto Leandro Faxina

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ADRIAN GRASSON FILHO

**Avaliação do método da superfície específica para determinação do teor de
asfalto de matrizes de agregado fino (MAF)**

Dissertação submetida ao Departamento de Engenharia de Transportes da Escola de Engenharia de São Carlos da Universidade de São Paulo (STT/EESC-USP), como parte dos requisitos para a obtenção do título de Mestre em Ciências.

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“Viver é um rasgar-se e remendar-se”

Guimarães Rosa

GRASSON FILHO, Adrian. **Avaliação do método da superfície específica para determinação do teor de asfalto de matrizes de agregado fino (MAF)**. 2017 – Escola de Engenharia de São Carlos, Universidade de São Paulo, São Carlos, 2017.

Os estudos da Matriz de Agregados Finos (MAF) são capazes de proporcionar informações importantes acerca do comportamento à fadiga das misturas asfálticas. Apesar do crescente número de pesquisas nesta área, não há consenso sobre o método mais adequado para estimar o teor de asfalto da MAF e que represente a fração MAF da mistura asfáltica. Dentre os métodos disponíveis, destaca-se o de Sousa et al. (2013), que calcula o teor de asfalto por meio da diferença de massas. Apesar de ser um método experimental relativamente prático, a extração de asfalto pelo processo de ignição pode ser uma dificuldade para alguns laboratórios. Como todo método experimental, o método de Sousa et al. (2013) também é suscetível à possível influência do operador. No intuito de contornar esta dificuldade, foi avaliada a aplicabilidade de um método analítico proposto por Ng et al. (2018), baseado no conceito de superfície específica do agregado mineral. Neste experimento, foram utilizados três agregados minerais (dois granitos e um basalto), quatro composições granulométricas e um asfalto (CAP 30/45 e PG 70-16). Em relação ao método de Sousa et al. (2013), os resultados deste estudo mostraram a importância de considerar a água de cristalização do agregado mineral, de modo a obter uma estimativa mais precisa do teor de asfalto da MAF. O método da superfície específica se mostrou capaz de fornecer uma estimativa confiável dos teores de asfalto das MAFs ao levar em consideração: (i) a estimativa da superfície específica do fíler por meio da difração a *laser* pelo equipamento *Malvern Mastersizer 2000* ($R^2 = 0,9948$) e pela curva de sedimentação segundo lei de Stokes ($R^2 = 0,8402$) e (ii) o módulo de riqueza da MAF obtido por uma relação com o módulo de riqueza da mistura asfáltica desenvolvida neste estudo.

ABSTRACT

GRASSON FILHO, Adrian. **Evaluation of the specific surface method as a tool to determine the asphalt content of fine aggregate matrices (FAM)**. 2017 – Escola de Engenharia de São Carlos, Universidade de São Paulo, São Carlos, 2017.

The studies of the Fine Aggregates Matrix (FAM) are able to provide important information about the fatigue behavior of the hot asphalt mixtures (HMA). Despite the increasing number of research works in this area, there is no consensus on the most appropriate method to estimate the asphalt binder content of the FAM and that represents the FAM fraction of the HMA mixture in a precise way. The method developed by Sousa et al. (2013) stands out among the methods available in the literature. It is an experimental method which calculates the asphalt binder content by mass difference. In spite of being a practical experimental method, the binder extraction by means of the ignition process might be a barrier for some laboratories. As any experimental method, the Sousa's method is also susceptible to the influence of the operator. In order to overcome these issues, the applicability of an analytical method, as proposed by Ng et al. (2018) and based on the specific surface concept of the mineral aggregate, was evaluated. In this experiment, three mineral aggregates (two granites and one basalt rock), four aggregate gradations and one asphalt binder (PG 70-16) were used. Concerning the Sousa's method, the results of this study showed the importance of taking into account the crystallization water of the mineral aggregate in order to obtain a more precise estimate of the FAM binder content. The specific surface method proved to be capable of producing a reliable estimate of the FAM binder contents, by taking the following aspects into account: (i) the estimate of the specific surface area of the filler by means of the laser diffraction method by using the Malvern Mastersizer 2000 ($R^2 = 0.9948$) and the sedimentation curve as per the Stokes law ($R^2 = 0.8402$), and (ii) the FAM richness modulus obtained by means of a relationship with the richness modulus of the HMA mixture developed in this study.

Keywords: Fine Aggregate Matrix. Asphalt Binder Content. Specific Surface.

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1 INTRODUCTION

1.1 Research context and problem statement

The evolution of the damage or degradation of pavements is a subject of great importance in the research works that intend to propose performance models for flexible pavements with wearing courses built with hot mix asphalt (HMA) mixtures. It is known that the main distresses of flexible pavements are rutting and fatigue cracking of the asphalt layer, both caused by the magnitude and frequency of loads, as well as by the climatic conditions. The evolution of such deterioration mechanisms is directly associated with the mechanical responses of the materials and, for this reason, it is fundamental to characterize the mechanical behavior of asphalt mixtures appropriately.

Fatigue cracks are treated as a phenomenon that develops from the micro scale to the macro scale. The process of repeated dynamic load applications from heavy vehicles to the pavement structure promotes the development of microcracks in the HMA structure, which grow to macrocracks due to the propagation of cracks and the coalescence process. The evolution of the cracks into the HMA structure occurs as result of the adhesive failure (at the interface aggregate-mortar) and/or cohesive failure (within the mortar). In this scenario, the fine portion of the asphalt mixture can be approached as a representative scale for the development of this phenomenon in the full asphalt mixture. By adopting this hypothesis, some researchers (Kim, 2003; Kim, Little, Song II, 2003; Kim, Little, Lytton, 2003) have investigated the fatigue characteristics of the fine aggregate matrix (FAM). The idea behind such studies is to understand the fatigue mechanisms at a scale that is not affected by the coarse aggregate particles, as well as observe the effect of different materials on the fatigue performance.

The fine aggregate matrix (FAM) is defined as the portion of the full asphalt mixture (HMA) composed of fine aggregates, filler, asphalt binder and air voids. It represents an intermediate scale between mastic (asphalt + filler) and concrete asphalt, and according to Masad et. Al. (2006) it presents an internal structure more homogeneous than the HMA mixture. Another advantage of the FAM is the reduced size of the samples - cylindrical specimens measuring 12 mm in diameter and 45 to 50 mm in height - which represents a considerable reduction in material consumption and laboratorial work as compared to the amount of material and time required to prepare HMA specimens.

Despite the growing number of studies in this area, there is no consensus among

researchers on an adequate design method that can be applied to most materials and could represent the FAM fraction within the HMA mixture. The air voids content that best represents the fine aggregate matrix is not well defined and, as a consequence, the methods developed to determine the FAM binder content tend to be experimental and correlated to the asphalt content obtained in the design of the full asphalt mixture.

Kim (2003) adopted a fixed value of 8% for the FAM binder content, which corresponds to an asphalt film thickness of 10 micra. After that, Castelo Branco (2008) assumed that the FAM binder content is equal to the binder content of the fine aggregate portion of the full asphalt mixture. Karki (2010) adopted the same assumption presented by Kim (2003) and proposed calculations based on a film thickness of 12 micra. Other researchers have suggested experimental methods: Coutinho et al. (2011) determined the FAM binder content using a solvent to extract the asphalt binder of the fine aggregate portion of the HMA, and Sousa et al. (2013) used the method of the ignition oven to extract the asphalt binder.

1.2 Problem approach

The procedure developed by Sousa et al. (2013), although effective and easy to reproduce, presents two aspects that could make it difficult to apply: (i) it requires that the laboratory be equipped with an ignition oven to calcinate the asphalt binder, what can not be the reality of many laboratories, and (ii) it is a long procedure when compared to other traditional procedures for asphalt mixtures: the experience of reproducing the method in the laboratory shows that the whole process, including material sieving, oven conditioning, manual fractionation using sieves #4, #8 and #16 and, finally, calcination of the asphalt binder in the ignition oven, takes something around 3 days.

In order to facilitate the estimation of the initial value of binder content for the production of FAM specimens, a procedure based on the concept of the specific surface of mineral aggregates is evaluated. It is believed that a procedure based on this concept is simpler and easier to apply, besides being free of some inherent limitations of the experimental methods available in the literature for determining the FAM binder content (Ng, 2017). Such limitations are related not only to the need for the laboratory to have specific equipment and to the time to perform the whole procedure, but also, and mainly, to the existence of a series of stages on which there is a great influence of the operator. Differently from the experimental procedures available in the literature, the specific surface method is simple, fast and less prone to operator influence, due to a procedure consisting of a reduced

number of steps. It is important to pay attention in the fact that the estimation of the mineral aggregate shape and geometry are still inaccurate, these properties are essential for the determination of the specific surface area.

1.3 Objectives of this research

The main objective of this research is to evaluate the specific surface method as an alternative tool to obtain an initial estimate of the binder content to be used in the production of fine aggregates matrices (FAM). After a careful literature review on the topics addressed in this study, it was decided to go deeper into two additional topics: (i) the evaluation of the influence of the crystallization water on the determination of the binder content when the method proposed by Sousa et al. (2013) is applied, once that the accuracy of the binder content determined by using the ignition oven may be compromised by not taking the crystallization water into account, and (ii) the comparison of two methods employed to determine the specific surface area of the mineral filler, as a way to evaluate the influence of the surface area of the mineral filler on the estimate of the binder content.

The proposed study seeks to associate the current knowledge in the area of determination of the specific surface of mineral aggregates to the current knowledge on the estimation of the binder content of fine aggregate matrices. The specific surface concept was first applied by Arrambide and Durrieu (1959) and has wide applicability for dosage purposes, not only of asphaltic concretes (Brown et al., 2009; Asphalt Institute, 2010), but also of Portland cement concrete (Alexander and Mindess, 2010).

The main studies on FAM and a discussion on the applicability of the specific surface method to mineral aggregates, in particular fine particles, will be briefly presented in order to contextualize this research. Although the investigation does not aim to study the FAM mechanical properties, the determination of the binder content for sample preparation is of great importance, since, as it is known, the binder content directly affects the asphalt mixture performance.

1.4 Dissertation structure

This dissertation is divided into five chapters. Chapter 1 presents the initial considerations of this master's dissertation, the research context and problem statement, the problem approach, the objectives of the research, the experimental plan outline, and the dissertation structure.

Chapter 2 presents a literature review comprised of relevant studies related to the fine aggregate matrix and the current knowledge about the determination of the FAM binder content. It also brings a discussion about the estimation of the specific surface area of mineral aggregates.

Chapter 3 contains the material and methods utilized to estimate binder content present on the fine aggregate matrix of the full HMA mixture. At first, it brings a detailed description of the aggregates and asphalt binder utilized to produce the specimens of HMA and FAM mixtures. Afterwards, the procedures to determine the binder content by both methods: Sousa et al. (2013) and Ng et al. (2018) were detailed. Chapter 3 also brings two suggestions: (i) to include a calibration factor associated to crystallization water into the method proposed by Sousa et al. (2013), and (ii) to estimate the filler surface area instead of adopting a predetermined value.

Chapter 4 presents the results obtained from the experiments performed in this study. It brings a discussion on the parameters that most influence the production of a FAM that best represents the respective original HMA mixture. The chapter also shows a discussion about the influence of the crystallization water on the estimation of binder content by Sousa et al. (2013), and the influence of specific surface area of the filler on the estimation of binder content by Ng et al. (2018).

Chapter 5 presents the conclusions of this study and the recommendation for future work. At the end of the document, a list of references mentioned along this manuscript is presented.

2 LITERATURE REVIEW

The main objects of this literature review are (i) the fine aggregate mixtures (FAM): its history, definition, behavior in induced moisture damage and fatigue and the recent studies developed in this area; (ii) the determination of de FAM binder content, the history of FAM design methods; and (iii) the estimation of the specific surface area of mineral aggregates.

2.1 Fine aggregate matrix (FAM)

Studies with FAMs started at Texas A&M University, with the work conducted by Kim (2003). The research was motivated by the fact that the FAM has a relatively more homogeneous structure than the full hot asphalt mixture and has great influence on the formation and propagation of the cracking phenomenon (Masad et al., 2006). Kim (2003) characterized the fatigue damage of FAMs in dynamic shear rheometer (DSR), using the elastic-viscoelastic correspondence principle proposed by Schapery (1984). Zollinger (2005) continued this research focusing on the characterization of induced moisture damage in FAM samples. Castelo Branco (2008) used a model for the evaluation of the cracking progression proposed by Masad et al. (2006) for different loading and amplitude modes for stress and controlled strain. Afterwards, Aragão (2011) developed a model based on micromechanics and Finite Element Method (FEM) to predict the dynamic modulus of HMA mixtures using properties of their respective components: coarse aggregates and FAMs.

There are evidences that the fine portion of the full asphalt mixtures has a strong influence on the development of flexible pavement degradation mechanisms, which reinforces the suitability of its use as an intermediate step in predicting the HMA mixture performance. The studies of Zollinger (2005), Masad et al. (2006) and Underwood and Kim (2013) have shown that FAM test results correlate well with laboratory and field measurements obtained from HMA mixtures. On the other hand, no studies are found in the literature relating FAM properties to rutting resistance, since this phenomenon is governed predominantly by granular interlocking, whose responsibility is attributed to the coarse aggregate fraction.

There is a lack of consensus between researchers in the literature concerning the choice of the sieve that limits the nominal maximum aggregate size (NMAS) of the fine aggregate portion to be used in the studies with FAM for fatigue damage and induced moisture damage. Some studies (Kim, 2003; Kim, Little, Song II, 2003; Kim, Little, Lytton, 2003; Zollinger, 2005; Arambula, Masad, Martin, 2007; Masad et al., 2006; Caro et al., 2008a; Caro et al., 2008b; Castelo Branco, 2008) set sieve #16 (1.18mm) as the upper limit

for design FAM samples. Aragão et al. (2011) and Dai and You (2007) used different sieves (0.6 and 2.36mm, respectively) to separate the coarse part from the fine part of the asphalt mixture. In Brazil, the research carried out by Coutinho et al. (2010), Coutinho et al. (2011) and Coutinho (2012) uses sieve #10 (2.00mm) as the sieving limit of the fine fraction, because sieve #16 (1.18mm) is not part of the the Brazilian sieve series and is close to the American sieve. Freire, Castelo Branco and Vasconcelos (2014) tested samples made with particles passing sieves #10 and #16 for fatigue damage, and concluded that there are evidences that the fatigue characteristics are not affected by this difference on the aggregate size, meaning that both maximum aggregate size can be used to represent the fine portion.

2.2 FAM as representative of the HMA

An asphalt mixture is basically represented by a specific aggregate gradation and an amount of asphalt binder (Asphalt Institute, 2010). This way, in order to establish a relation between the fine aggregate matrix and the full asphalt mixture that it represents, two considerations must be taken into account: (i) the gradation of the FAM must be proportional to the gradation of the fine portion of the full HMA mixture and (ii) the binder content of the FAM must have a relation to the binder content present in the fine portion of the HMA.

2.2.1 Determination of FAM gradation

As far as aggregate gradation is concerned, in the first studies with FAM, researches from Texas A&M University chosen sieve #16 (1.19mm) to divide the mixture into the fine and coarse portion. The proportionally of the fine aggregates that composes the FAM was kept the same as in the full HMA aggregate gradation, but normalized. It means that 100% of the aggregate in the FAM passes sieve #16, and the percentage of aggregates passing sieves #ii below sieve #16 is determined by Eq. (2.1). Figure 2.1 shows a comparison of FAM aggregate gradations and the corresponding full mixture gradation. This concept of proportionality is well accepted and all the next studies with FAM used this relation to produce the FAM mixtures. The proportionality was adopted even when the choice of the sieve that separates both portions varied.

$$\frac{\text{Mass of aggregate passing sieve \#ii in full mixture}}{\text{Mass of aggregate passing sieve \#16 in full mixture}} \times 100\% \quad (2.1)$$

Figure 2.1 – Aggregate gradation of the HMA and the FAM mixtures

2.2.2 Determination of FAM binder content

Another key topic in studies on the fine fraction of the hot asphalt mixture is the determination of the asphalt binder content that is presented only in the fine aggregate matrix. During the review of FAM design methods, no standard procedure was found for determination of the asphalt binder content in order to generate FAMs with characteristics similar to the fine portion of the HMA mixture they represent.

In the first studies with FAMs, Kim (2003) and Zollinger (2005) assumed a film thickness of 10 micra, which represented 8% asphalt binder content, in the preparation of FAMs mixtures, regardless of aggregate gradation and asphalt binder content of the correspondent HMA mixture. Some studies developed later determined the asphalt binder content based on the estimated thickness of the asphalt film (Vasconcelos et al., 2007; Castelo Branco, 2008; Caro et al., 2008a; Caro et al., 2008b; Vasconcelos, Bhasin, Little, 2010; Vasconcelos et al., 2011). However, the binder contents obtained were very high and made it impossible to compact and extract the samples.

Five design methods are available in the literature: Castelo Branco (2008), Karki (2010), Coutinho et al. (2011) - later corrected by Freire (2015), Sousa et al. (2013) and Ng (2017). In the method proposed by Castelo Branco (2008), the FAM binder content is obtained assuming that the FAM binder content is proportional to the mass of material passing sieve #16. The method proposed by Karki (2010) estimates the binder content based on the film thickness shared by coarse and fine aggregates. The methods proposed by Coutinho et al. (2011) and Sousa et al. (2013) are experimental and basically distinguished by

the method used to extract the asphalt binder of the fine portion of the HMA mixture. Coutinho et al. (2011) employ solvent extraction and Sousa et al. (2013) employ extraction using the ignition oven. The method used by Ng et al. (2018) applies the concept of the specific surface proposed by Arrambide and Duriez (1959) to calculate the binder content.

The method proposed by Sousa et al. (2013) is the latest experimental method proposed in the series of studies related to the determination of the asphalt binder content of the FAM fraction on the HMA mixtures. It was developed at Texas A&M University using four HMA mixtures produced with the most used mineral aggregates in roads on the American state of Texas. Although the method has a consistent rational basis, it is fundamentally composed of an experimental sequence, and it is important to verify its applicability in terms of the variability of results when different materials, operators and equipment are used.

Ng et al. (2018) reproduced the procedures proposed by Castelo Branco (2008), Coutinho et al. (2011) - later corrected by Freire (2015) and Sousa et al. (2013) using one source of basalt rock and four binders, one neat and three modified (the modifier used were: (i) crumb rubber (ii) SBS copolymer and (iii) PPA). Ng et al. (2018) showed that each method resulted in a different binder content: for the procedure proposed by Castelo Branco (2008) the binder content were so high that made it difficult to extract the FAM specimens, by the method proposed by Coutinho et al. (2011) – later corrected by Freire (2015), no proportionality with the binder content in the HMA mixtures was observed, it was also difficult to separate the mixture particles when sieving by hand. For the modified binder with crumb rubber, it was found influence of the particulate material in the calculations of the mass difference. In order to overcome those issues, Ng et al (2018) proposed a method based on the concept of the surface area (S_s) and the richness modulus “K” developed by Arrambide and Duriez (1959).

The method proposed by Ng et al. (2018) adapted the method developed by Arrambide and Duriez (1959) to the FAM gradation and sieve series of the study. The results obtained for the basalt rock using the proposed method were equivalent to those obtained by applying the procedure of Sousa et al. (2013), what was interpreted as an evidence of the effectiveness of the proposed method.

2.3 Estimation of the specific surface area of mineral aggregates

The mineral aggregate presents a wide variability of mineral composition, shape,

roughness and texture. The specific surface represents an indirect measure of these characteristic of the mineral particles. The surface area of a set of mineral aggregates is defined predominantly by the fraction of fine aggregates, since for a given mass of material the surface area is inversely proportional to the particle size (Alexander and Mindess, 2010).

The surface area of mineral aggregates is usually calculated using specific surface factors, which represent an average surface area of cubes or spheres with diameters equivalent to the average of the nominal opening of two adjacent sieves (Wang and Frost, 2002). This procedure was first proposed by Arrambide and Duriez (1959). The application of this procedure provides the smallest surface area of all possible forms for a particle and, for this reason, the method is not able to properly account for the effects of shape, roughness and texture of mineral aggregates (Wang and Frost, 2002). If, in practice, all particles were cubic or spherical, the calculation would be relatively simple, and only the average particle size should be known. However, it is almost impossible to find particles with a perfectly cubic or spherical shape in an aggregate sample, and this is particularly true for crushed mineral aggregates (Alexander and Mindess, 2010).

The particles of crushed mineral aggregate present irregular shape and grooves, and because of this the surface area of a real particle may be very different from that estimated by assuming a cubic or spherical shape. The less regular the particle shape the larger the specific surface (Alexander and Mindess, 2010). According to Wang and Frost (2002), the more irregular the particle shape the greater the roughness and the sphericity. Sphericity is a measure of shape and roughness and is obtained as the ratio of the surface area of a particle to the surface area of a perfect sphere of the same volume (Wang and Frost, 2002). For an example, a sphere has sphericity equal to one and all other forms have sphericity greater than one.

The estimation of the surface area of the aggregate mixture in a hot asphalt mixture is one of the fundamental factors to quantify the asphalt binder content necessary to adequately cover all the mineral particles (Panda, Das, Sahoo, 2016). In this way, Anochie-Boateng, Komba and Tutumluer (2011) and Anochie-Boateng and Maina (2013) point out that the precise determination of the surface area of the HMA mixtures aggregate particle size distribution (PSD) is of fundamental importance in estimating the strength of the mixture. In HMA mixtures, the surface area of the mineral aggregates is directly related to the thickness of the asphalt film and, consequently, to the fatigue and rutting performance of the asphalt concrete (Wang and Frost, 2002). This is true not only for asphalt concretes but also for

Portland cement concrete (Cepuritis et al., 2017), in which information about the particle size and particle area are fundamental for the correct proportioning of the materials.

The morphological characteristics of the mineral aggregates, such as shape factors, angularity and surface texture, directly affect the performance of flexible pavements (Wang et al., 2015a; Wang et al., 2016), by means of the stability of the hot asphalt mixture (Herrin, Goetz, Campen, 1954; Masad et al., 2001), fatigue resistance (Monismith, 1970; Kim, Y., Kim, N., Khosla, 1992), and rutting resistance (Barksdale, 1972; Moghaddam, Karim, Abdelaziz, 2011) and friction (Maerz, 2004; Wang et al., 2014; Wang et al., 2015b).

The coarse and fine aggregates have distinct roles in the internal structure of an asphalt mixture. The coarse aggregates are responsible for the granular interlocking and the fine aggregates are responsible for the integrity of the mixture (Xiaoguang et al., 2017). The granular structure provided by the coarse aggregates provides stability and support capacity for asphalt mixtures (Li, Kett, 1967; Rao, Tutumluer, Kim, 2002; Pan, Tutumluer, Carpenter, 2006), while the fine portion has a direct effect on the shear resistance (Pan, Tutumluer, Carpenter, 2006; Roque, Shin-Che, Byron, 1997). The aggregate particle size distribution and the morphological characteristics of the fine aggregates also affect the surface microtexture, and interfere directly with the skidding resistance (Masad et al., 1999; Chen, Wang, 2011; Wang et al., 2013), while the percentage and the morphological characteristics of the coarse aggregates determine the surface macrotexture and drainage capacity of the wearing course (Xiaoguang et al., 2017).

According to Panda, Das and Sahoo (2016) the calculation method to determinate the specific surface area of the mineral aggregates based on the factors related to the sieve maximum nominal openings is purely manual, subject to operator errors, besides presenting low statistical significance. For these reasons, more sophisticated methods, such as images in two or three dimensions must be employed. Wang and Frost (2002) published one of the earliest papers using the X-ray tomography method to quantify the surface area of a calcareous rock sample with particles between #3/8 "and #4 (9.51 mm and 4.76 mm). They observed that the specific surface and sphericity have a large variation between individual particles within the same sieve interval and that the specific surface of the mineral aggregates is much larger (84%) than that of spheres of equivalent size. However, it is important to note that this conclusion was obtained by the measurement of the specific surface area of only coarse aggregate particles (Wang and Frost, 2002).

In an attempt to provide a more practical and simple method, Panda, Das and Sahoo

(2016) proposed a mathematical model to estimate the surface area of a mixture of aggregates based on simple physical properties of the mineral aggregate, such as particle size distribution and percentage of flat and elongated particles. Panda, Das and Sahoo (2016) obtained the surface area of 47 samples and compared the results with other calculation methods, such as the one recommended by the Asphalt Institute, using a model based on three-dimensional images and modified area factors. According to those researchers, the results obtained by this method of calculation showed good correlations with other more sophisticated methods.

The complexity associated with determining the surface area increases with the reduction of particle size, and it is extremely difficult to accurately measure the surface area of the filler. Cepuritis et al. (2017) presented a comparison between different methods to obtain the aggregate particle size distribution and the specific area of crushed fine mineral aggregates (particles smaller than 250 micra). Cepuritis et al. (2017) pointed out that, among the methods evaluated in their study, only the X-ray sedimentation and the laser diffraction could measure particles of this magnitude. Cepuritis et al. (2017) recommend the X-ray sedimentation, because it presents adequate resolution and requires more precise parameters. The authors combined tomography and spherical harmonic analysis to estimate the real error introduced when the estimation of the area of the particles is made based only on particle size distribution. The conclusions of the study appear to be very consistent, since the researchers employed fines aggregates from 10 rocks, covering a large range of mineralogic components.

Cepuritis et al. (2017) concluded that the error in calculating the surface area by assuming the sphericity of the particles is of the order of 20 to 30% over the total range of investigated particle sizes. This conclusion is important, as the authors said, since it simplifies the determination of the specific area of the particles. The authors recommend that the results obtained based on sieving, assuming spherical shape particles, should be adjusted upwards by 20%, for particles smaller than 20 micra, and 30%, for particles larger than 20 micra, in order to determine the specific area of crushed fine mineral accurately.

Cepuritis et al. (2017) also pointed out that for crushed fine minerals passing sieves with opening of 125 or 63 micra about 50% of the specific area is concentrated in the particles whose equivalent spherical dimension is of the order of 5 micra. In an earlier paper, Cepuritis et al. (2014) stated that around 90% of the specific area of the samples is concentrated in the range of particles whose size is smaller than 20 micra.

Cepuritis et al. (2014) state that since the smaller particles are responsible for a large part of the specific area of the fine aggregates, only the methods that account for the aggregate

gradation of particles smaller than 5 micra are adequate to determine the surface area of fine minerals over the entire range of particle sizes. They also pointed out that it will be the resolution of the method used to determine the particle sizes smaller than 5 micra that will determine the accuracy of the results when using the information of particle size distribution to calculate the specific area, assuming a regular shape for the mineral particles.

As can be inferred from the discussion presented earlier and also pointed out by Alexander and Mindess (2010), there are practical difficulties in measuring or estimating the real surface area of a sample of mineral aggregates formed by particles with different shapes. The measurement of the surface area of the aggregate mixture or even of a single particle is difficult because of the irregular shape and particle texture (Panda, Das, Sahoo, 2016). A measure of the complexity associated with this determination is given by Cepuritis et al. (2014), where they affirm that different methods of measurement of filler particles (eg.: Blaine, BET, laser diffraction, sedimentation) can easily provide results for specific areas that differ from one another by factors of the order of up to 10 times, where the laser diffraction method has the highest resolution.

2.3.1 Estimation of the specific surface area by means of the aggregate particle size distribution (PSD)

Arrambide and Duriez (1959) proposed a design method for asphalt mixtures which is based on the assumption that all particles should be covered by an asphalt film where the thickness of the film grows (non-linearly) with the particle size. It means that the binder content is a function of two factors: the richness modulus “K” defined as a value proportional to the film thickness and traffic and (ii) the specific surface of the mineral aggregates (S_s)

Arrambide and Duriez (1959) assumed that the specific surface of an aggregate mixture with different sizes is a combination of the specific surfaces calculated from each sieve interval. For the coarse aggregates, this information is obtained from the aggregate sieving, and for the filler fraction this information can be inferred from the hydrometer analysis and physical principles of sedimentation. However, they point out that the value of $135 \text{ m}^2/\text{kg}$ is a mean value that could be used for the specific surface area of filler, and it is actually largely adopted (Bernucci et al. 2008).

The specific surface coefficient for each sieve interval is determined by taking into account the particle area and volume and the bulk specific density, which is represented by Eq. (2.2)

$$S = \frac{A}{V \times \rho} \quad (2.2)$$

where:

S: specific surface coefficient (m²/kg);

A: particle area (m²);

V: particle volume (m³);

ρ: bulk specific density (kg/m³)

Arrambide and Duriez (1959) assumed the shape of the aggregate particles as a perfect cube. Following such assumption, area and volume can be calculated according to Eq. (2.3) and (2.4)

$$A = 6 \times a^2 \quad (2.3)$$

$$V = a^3 \quad (2.4)$$

where “a” is the face of the cube (m). The diagonal of the cube, Eq. (2.5), is defined by the average of the nominal maximum aggregate diameter for each sieve interval.

$$d = a \times \sqrt{3} \quad (2.5)$$

This way, the total specific surface of an aggregate mixture is the sum of the coefficients multiplied for the percent of particle between two sieves (P_i), for each sieve interval, as per Eq. (2.6).

$$S_s = \frac{\sum (S_i \times P_i)}{100} \quad (2.6)$$

2.3.1.1 Estimate of the specific surface by laser diffraction (LD)

The laser diffraction method (LDM) was developed in the 1970's and it has become a popular technique in many fields for measuring the particle size distribution (PSD). This method is based on the concept that a diffraction pattern of a laser beam passing through a particle can measure the geometrical dimensions of that particle (Ma et al., 2000). A brief discussion about the determination of the specific surface from the particle size distribution presented in the literature review shows that LDM has the highest resolution to provide a measurement of the surface area of the mineral filler particles (Cepuritis et al., 2014).

Among the advantages of the laser diffraction, the following ones deserve to be mentioned: short analysis time, high repeatability, low required sample volume, a wide measurement range and a wide range of sorted fractions. The different physical principles behind the laser diffraction and the sedimentation method lead to different results of the grain

size analysis. Because of this, the results determined by laser diffraction cannot be directly compared with the conventional method (pipet, hydrometer etc.) (Šinkovičová et al., 2017).

The User Manual of the Malvern Mastersizer 2000 presents the method applied by the software in order to obtain the grain size distribution. It indicates that the equipment uses the optical bench to capture the actual scattering pattern from a field of particles to calculate the size of particles by using the Mie theory. The Mie theory was developed to predict the way the light is scattered by spherical particles and deals with the way light passes through, or is adsorbed by, the particle. This theory assumes the user knows some specific information about the particle, such as its refractive index and its absorption. Once this information is obtained by the Mastersizer 2000, it can work backwards through the theory and provide the grain size distribution.

The User Manual of the Malvern Mastersizer 2000 also presents the procedures to measure the dimensions of the sample by following the steps below:

- (i) The sample must be prepared and dispersed to the correct concentration and then delivered to the optical bench.
- (ii) The optical bench performs the measurement of capturing the scattering pattern of the sample.
- (iii) The detector array within the optical bench is made up of many individual detectors.
- (iv) Each detector collects the light scattering from a particular range of angles.

A typical light scattering pattern is shown in Figure 2.2.

Figure 2.2 – Data Graph – Light Scattering

Source: Malvern Mastersizer 2000 User Guide.

Each bar in the histogram represents the light scattering from one of the detector elements (known as a channel). The detector array takes a “snapshot” of the scattering pattern. Obviously, this snapshot only captures the scattering pattern from the particles that are passing through the analyzer beam at that particular time. Taking only one snapshot may not give a representative reading of the scattering pattern. To overcome this, the Mastersizer takes many snapshots (known as snaps) and averages the result. Typically, over 2000 snaps are made for each measurement, with each snap taking 1ms. Once the measurement is complete, the raw data is analyzed by the Malvern software.

2.3.1.2 Estimate of the specific surface area by means of the hydrometer analysis

Stefano, Ferro and Mirabile (2010) compared grain-size analyses using laser diffraction method and the sieve-hydrometer method (SHM) for 228 samples with different textures classification from the Sicilian basin. They point out that sedimentation methods assume a single particle density, which is a major source of error, whereas LDM measurements are independent of particle density. They also pointed out that both methods are affected by the deviations from the sphericity of particles, with an irregular shaped particle having longer settling time than their equivalent spheres, which result in an overestimation of values. However, the study showed that there was no significant difference in the particle size distribution using both methods. The Analysis of all samples showed that the sand content determined by SHM was similar to that obtained by LDM as can be seen in Figure 2.3.

Figure 2.3 – Relationship between sand fraction obtained by LDM and SHM

Source: Stefano, Ferro and Mirabile (2010)

Still according to Stefano, Ferro and Mirabile (2010), the sedimentation methods are time consuming, whereas LDM provides more information and is more efficient. Although no ideal method can be defined, once that the final choice of a grain-size technique depends on many factors as type of sediments, speed of measurement, specific aims of the study, etc. Arrambide and Duriez (1959) pointed out that the filler specific surface area can be inferred from sedimentation curve obtained from the hydrometer analysis.

According to ASTM D7928-17, the hydrometer analysis method (HM) defines a particle diameter as equivalent to that of a sphere setting in the same liquid with the same speed as the unknown sized particles, called as “Stokes diameter”. HM uses a hydrometer having a graduated stem and weight bulb to measure the specific density of the suspension. The specific density depends on the weight of the particles in the suspension at the time of measurement.

Hydrometer analysis is based on Stokes’ law that establishes the velocity at which particles settle in suspension assuming that: (i) particles are rigid, spherical and smooth; (ii) particles have similar densities; (iii) particle-to-particle interference and boundary effects from the walls of the sedimentation column are negligible; and (iv) particle sizes are small enough to ensure that the induced fluid flow is within the laminar flow regime (Stefano, Ferro and Mirabile, 2010).

2.3.1.3 Richness Modulus

The richness modulus is a coefficient of great importance for the determination of the binder content, because it is the binder content that controls the flexibility of the asphalt mixture, its ability to adapt and self-repair, as well as its rigidity and deformability. A low richness modulus defines a brittle and usually not impermeable asphalt mixture. On the other hand, a high richness modulus defines an adaptable, self-repairable mixture, although slippery, deformable and not stable when subjected to traffic (Arrambide and Duriez, 1959).

The choice of the richness modulus involves a consideration about the nature of the subgrade, the level of traffic and also the impermeability wanted to the wearing surface course. A high value is chosen for the mineral aggregates with high absorption and roughness and also for the new pavement structures placed above a stabilized subgrade. On the other hand, a low value is chosen for mineral aggregates with smooth surfaces and when it is placed above a previous structure already rigid and stable (Arrambide and Duriez, 1959). Table 2.1 presents the recommended richness modulus values according to the wearing course type.

Table 2.1 – Richness modulus according to wearing coarse type

Surface coarse	K
Hot Mix Asphalt Concrete (highway)	3,5 – 4,0
Dense Mixture	3-75 - 4,5
Sand Asphalt	4,0 – 4,5
Sheet Asphalt	5,0

Source: Arrambide and Duriez (1959)

2.4 Remarks on the FAM design methods and on the determination of the specific surface area of the mineral aggregates

The determination of the binder content of an HMA mixture is based on the assumption that there is an optimum amount of binder necessary to cover all the mineral aggregates particles with a specific film thickness, in such a way that the resulting mixture be capable of resisting the traffic loading and environmental effects. The smaller the particles the bigger the total specific surface area for the same mass of material and, consequently, the higher the amount of binder needed to cover the surface of that material.

The method proposed by Sousa et al. (2013) estimates the binder content of the fine portion of the full HMA mixtures (material passing sieve #10) by extraction using the ignition oven, while the method proposed by Ng et al. (2018) obtains the binder content by means of characteristics of the mineral aggregate such as specific surface area and specific bulk density. The Sousa's method is experimental and has a logical basis, but it is fundamentally composed of an experimental sequence. Because of this, it is important to check its applicability in terms of data variability when different materials, operators and pieces of equipment are used. The Ng's method is more theoretical and fundamentally composed of a sequence of calculations where some assumptions have to be made, such as the shape, roughness and texture of the crushed mineral particles. Because of this, it is important to check its capacity to provide results similar to the ones obtained by the experimental method (Sousa's method).

In both methods, there are some points that require a careful look. Concerning the method proposed by Sousa et al. (2013), it is supposed that the consideration of the crystallization water of the mineral aggregates can affect the calculation of the mass difference of the mixture after the extraction of the binder in the ignition oven. Regarding the method proposed by Ng et al. (2018), two items deserve more discussion: the method to determine the specific surface area of the filler and the choice of the richness modulus of the mixture by means of an objective criterion.

3 MATERIALS AND METHODS

This chapter presents the materials and methods used during the development of this research. It also brings the adapted method of sedimentation for mineral aggregates in order to provide the particle size distribution of filler, and a suggested correction to the method of Sousa et al (2013).

3.1 Materials

3.1.1 Mineral Aggregates

Test materials used in this study were chosen to represent aggregates that are commonly used on flexible pavement in the Southeast region of Brazil, i.e., basalt and granite.

One source of basalt and two sources of granite were used. The basalt was obtained from Bandeirantes Quarry, located in São Carlos, State of São Paulo, and the granites were obtained from construction works carried out by *CCR NovaDutra* road concession. Granites from two different quarries were collected: (i) Pombal, located in Barra Mansa, State of Rio de Janeiro and (ii) Jambeiro, located in Jambeiro, State of São Paulo.

Mineral aggregates from three different sources will be used in an attempt to evaluate the influence of mineral aggregate characteristics, such as the specific gravity and absorption, on the determination of the FAM binder contents.

The mineral aggregates were characterized following the standard procedures from DNIT (Departamento Nacional de Infraestrutura de Transportes – National Department of Transportation Infrastructure) and ASTM (American Society of Testing and Material). These characteristics of the mineral aggregates are presented in Table 3.1.

Table 3.1 – Mineral aggregates characteristics

Aggregate Quarry	Pombal	Jambeiro	Bandeirantes
Aggregate Type	Granite	Granite	Basalt
Specific gravity of coarse aggregates (g/cm ³)	2.805	2.655	2.904
Specific gravity of fine aggregates (g/cm ³)	2.864	2.661	2.999
Specific gravity of filler (g/cm ³)	2.811	2.637	2.769
Absorption (%)	0.3	0.3	0.6

In order to establish a relation between the three mineral aggregates, the HMA mixtures were prepared with a dense gradation (range C) meeting requirements of the Brazilian standard DNIT 031/2006-ES. On the other hand, in order to establish a relation

between the same mineral aggregate type with different specific gravity, the HMA mixtures produced with the granites from Pombal and Jambeiro were also prepared in three different sieve gradations: two gradations followed the Superpave (Superior Preforming Pavements) requirements for 12.5mm and 9.5mm nominal maximum size (NMA) and the other was specified by *CCR NovaDutra* following the limits of the Caltrans (California State Transportation Agency) known as *Gap-Graded*. Table 3.2 presents the percentage of material passing the sieve series of the four aggregate gradations.

Table 3.2 – Aggregate gradations

Sieves #	mm	DNIT – range C			Superpave-9.5mm		Superpave – 12.5mm		<i>Gap Graded</i>	
		Pombal	Jambeiro	Bandeirantes	Pombal	Jambeiro	Pombal	Jambeiro	Pombal	Jambeiro
3/4"	19	100	100	100	100	100	100	100	100	100
1/2"	12.5	92.8	85.1	88.9	99.9	100	91.4	94	96.4	100
3/8"	9.5	84.3	78.3	81.3	90.9	99.2	81	90.7	77.3	99
Nº04	4.75	50	53.1	51.5	42.4	49.9	39.4	44.5	29.9	37.6
Nº10	2.00	39.7	37.9	38.8	32.6	33.4	30.6	29.7	21.7	22.9
Nº40	0.42	28	22.5	25.2	23.6	19.7	22	17.7	14.5	13.5
Nº80	0.18	18.4	14.6	16.5	15.9	12.9	14.7	11.6	8.4	9.4
Nº200	0.075	8.4	8.6	8.5	7.7	7.8	7.1	7.1	4.4	5.8

The curves for Jambeiro and Pombal are not exactly the same but very close, and because of it they will be compared by assuming that the curves for both mineral aggregates are equivalent. This assumption is plausible because the differences between the curves are within the allowable tolerances of the DNIT 031/2006 – ES. Figure 3.1 shows the four gradation curves.

Figure 3.1 – Aggregate gradation curves

3.1.2 Asphalt Binder

The asphalt cement (AC) used in this research is graded according to the Brazilian specification (penetration system) as a 30/45, and graded as a PG 70-16 in the PG system. The material was supplied by *CCR NovaDutra* road concession. The characterization of the asphalt binder were performed at the *Laboratório de Estradas* (STT-EESC/USP) following the ASTM protocols shown in

Table 3.3, where the characteristics of the asphalt binder obtained by means of these tests can also be seen.

Table 3.3 – Characteristics of the asphalt binder

Parameter	Unit	Standard	Result
density	g/dm ³	ASTM D 70-18	1.013
Penetration [100g/25°C/5s]	0.1mm	ASTM D 5-19	33
Viscosity 135°C	mPa.s (cP)	ASTM D 4402-15	424.5
Viscosity 150°C	mPa.s (cP)	ASTM D 4402-15	208.0
Viscosity 177°C	mPa.s (cP)	ASTM D 4402-15	75.1
Mixing Temperatures	°C	-	156 - 162
Compaction Temperatures	°C	-	145 - 150
Continuous grade HT (virgin)	°C	ASTM D 7175-15	70,8
Continuous grade HT (RTFO)	°C	ASTM D 7175-15	70,2
Continuous grade LT (S[60])	°C	ASTM D 6816-11	-20,5
Continuous grade LT (m[60])	°C	ASTM D 6816-11	-21,3
PG	-	ASTM D 6373-16	70-16
J _{nr} @64°C, 3.2 kPa	kPa ⁻¹	ASTM D 7405-15	4.0 (H)
J _{nr} @70°C, 3.2 kPa	kPa ⁻¹	ASTM D 7405-15	6.4 (S)

3.2 Methods

3.2.1 Determination of FAM binder content as per Sousa et al. (2013)

The method proposed by Sousa et al. (2013) for FAM design is experimental. This method is based on the preparation of samples of HMA mixtures following the Superpave design method (AASHTO T209). The method requires the separation of the fine portion using sieve #16 (<1.18mm), and calculates the asphalt content present in the fine portion. The method proposed by Sousa et al. (2013) must be performed following the steps described below and indicated in Figure 3.2:

Figure 3.2 – Steps of the method proposed by Sousa et al. (2013)

(a) Preparation of the HMA mixtures

(b) Manual mixing of the HMA mixture

(c) Conditioning period

(d) Fractioning the material

(e) Muffle furnace at 427°C

Source: The author

a) Three loose samples of HMA mixture are prepared (according to AASHTO T209) - the minimum amount depends on the nominal maximum aggregate size (NMAS) of the mineral aggregate: 4,000 g for NMAS of 37.5 mm, 2,500 g for NMAS of 19.0 mm or 25 mm, and 1,500 g for NMAS lower than 12.5 mm;

b) The three samples are subjected to a two-hour conditioning period in oven at $135\pm 5^{\circ}\text{C}$, according to AASHTO T209;

c) The mixture conglomerates are separated by hand and allowed to cool to room

temperature for 30 minutes;

d) Mechanical sieving is used to sieve the samples into different sizes (4.76 mm (#4), 2.38 mm (#8) and 1.19 mm (#16)). Stainless steel balls of 9.5 mm in diameter are recommended to facilitate the separation of the particles.

e) After fractioning, the material is separated into the following groups: (i) material retained in sieve 4.76 mm (#4); (ii) material passing sieve #4 and retained in sieve 2.38 mm (#8); (iii) material passing sieve #8 and retained in sieve 1.19 mm (#16); and (iv) material passing sieve 1.19 mm (#16); after that, each group of material is dried at 110°C;

f) Each group of material is weighted in order to register the mass of the pan (W_p) and the mass of the pair pan+sample (W_{Mi});

g) The containers are taken to the ignition oven at 427°C to calcinate the asphalt; after extraction, let the pair pan+sample cool to room temperature and measure the mass of aggregate for each group (W_{Ai}).

Eq. (3.1) is used to calculate the binder content of each group. The FAM binder content is the one calculated for group 4 (material passing sieve #16).

$$P_{bi} = \frac{W_{Mi} - W_{Ai}}{W_{Mi} - W_p} \times 100\%, \text{ where } i=1 \text{ to } 4 \quad (3.1)$$

In order to adapt the sieve intervals to the Brazilian standard sieves series, the sieves 4.76 mm (#4), 2.38 mm (#8) and 1.19 mm (#16) were replaced, respectively, by sieves 9.51 mm (#3/8"), 4.76 mm (#4) and 2.00 mm (#10). As Freire, Castelo Branco and Vasconcelos (2014) compared samples with particles passing sieve 1.19 mm (#16) and 2.00 mm (#10), and concluded that the fatigue characteristics should not be affected by aggregates sizes.

One aspect that was not taken into account during the development of Sousa's method is the crystallization water of the mineral aggregates. Owing to the impact of the crystallization water on the determination of the FAM binder content, it is assumed that this information must be taken into account in order to provide a more precise calculation. In practice, it is a very difficult task to obtain the weight of the water present in the mineral particles. The procedure described on ASTM D6307-16 was reproduced in order to determine this value. This procedure had to be adapted in order to match the procedure of Sousa et al. (2013) in terms of the (i) gradation (aggregates passing sieve 2.00 mm #10) and (ii) temperature (Sousa et al. (2013) set the value of 427°C to burn the asphalt binder in the ignition oven).

The procedure to determine the crystallization water content of the mineral aggregates followed the steps below:

- a) Prepare three calibration samples of blended aggregates in accordance to the FAM gradation. The sample should be approximately 200 g;
- b) Oven-dry the aggregates samples to a constant mass at 150°C;
- c) Determine and record the mass of the sample (M_1);
- d) Heat the calibration samples in the ignition furnace at 427°C for the same period of time used to calcinate the asphalt;
- e) Measure and record the mass of the sample after ignition (M_2);
- f) Calculate the calibration factor (C_F) as Eq. (3.2) below:

$$C_F = \frac{M_1 - M_2}{M_1} \times 100\% \quad (3.2)$$

By assuming that the actual asphalt binder content needs to consider the crystallization water of the mineral aggregates, a correction must be made. The final FAM binder content is then calculated by subtracting the percentage of crystallization water in the mineral aggregates of the value obtained by using the method by Sousa et al. (2013). The corrected asphalt binder content is calculated by using Eq. (3.3):

$$P_{bi,corrected} = P_{bi} - C_F \quad (3.3)$$

3.2.2 Determination of FAM binder content as per Ng et al. (2018)

This design method to prepare FAM samples was first used by Ng et al. (2018). It follows the procedure developed by Arrambide and Duriez (1959) to estimate the HMA binder content. This method is based on the richness modulus “K” and the surface area of the mineral aggregates (Ss). Ng et al. (2018) adapted the original method designed for HMA mixtures to account for the FAM gradation and the sieve intervals.

The specific surface coefficient for each sieve interval is calculated by Eq. (2.2), (2.3), (2.4) and (2.5). This way, as the Eq. (2.6), Eq. (3.4) can be built to describe the specific surface of a granite aggregate with $\rho_{real} = 2.655 \text{ g/cm}^3$ (For the mineral aggregates supplied by Jambeiro Quarry on the dense gradation).

$$S_{SFAM} = \frac{3.23(P_{2.00} - P_{0.42}) + 13.04(P_{0.42} - P_{0.18}) + 30.69(P_{0.18} - P_{0.075}) + 135(P_{0.075})}{100} \quad (3.4)$$

where:

- S_{SFAM} : the specific surface of the aggregates of the FAM (m^2/kg);
- $P_{2.00} - P_{0.42}$ the percent of particles between 2.00 mm and 0.42 mm;
- $P_{0.42} - P_{0.18}$ the percent of particles between 0.42 mm and 0.18 mm;

$P_{0.18} - P_{0.075}$ the percent of particles between 0.18 mm and 0.075 mm, and
 $P_{0.075}$ the percent of particles smaller than 0.075 mm.

After the determination of the surface area (S_{SFAM}) as calculated by Eq. (3.4) and after adopting a richness modulus “K”, the FAM asphalt content can be calculated by Eq. (3.5), where T_{FAM} is the FAM binder content for the total weight of the mixture.

$$T_{FAM} = \frac{100 \times K \times \sqrt[5]{S_{SFAM}}}{100 + (K \times \sqrt[5]{S_{SFAM}})} \quad (3.5)$$

Another important property that has to be included in the calculations is the aggregate absorption. The fine aggregate absorption is obtained by following the procedure ASTM C128-15. The final FAM binder content is obtained by Equation (3.6), where $\%A_{abs}$ is the fine aggregate absorption (%).

$$T'_{FAM} = T_{FAM} \times (1 + \%A_{abs}) \quad (3.6)$$

However, one issue of concern came up during the reproduction of the method: the value of 135 m²/kg adopted by Ng et al. (2018) for the specific surface area of the aggregate particles passing sieve 0.075mm (#200) is the same value suggested by Arrambide and Duriez (1959), but such value was found undersized. In order to find a better solution, a procedure to determine the filler specific surface area by the sedimentation curve was added to the procedure.

3.2.3 Sedimentation by hydrometer analysis (ASTM D7928-17)

The hydrometer analysis is based on the physical principles of sedimentation described by the Stokes' law. Which relates the force required to move a sphere through a given viscous fluid at a low uniform velocity to the velocity and radius of the sphere. The general approach is to mix the sediment into a suspension and then allow sedimentation while measuring the density of the suspended sediment at a specific depth. The procedure to obtain the sedimentation curve by ASTM D7928 is described as per the steps below and represented in Figure 3.3:

- a) Prepare a sample of 80g of material passing sieve 0.075 mm (#200) and place it into the specimen-mixing container;
 - b) Add 50 mL of sodium hexametaphosphate and 250 mL of test water to the specimen-container;
 - c) Mix the slurry for 20 minutes using the mixing device;
-

-
- d) Transfer the slurry to the sedimentation cylinder and add additional test water as necessary to achieve the volume of 1000 mL;
 - e) Mix the slurry using the agitator device at a rate about one cycle per second over a distance of several centimeters to dislodge any material stuck to the bottom of the cylinder;
 - f) Cover the cylinder to prevent evaporation and allow the cylinder to sit overnight. This conditional period allows the temperature to equilibrate and the specimen to deflocculate;
 - g) Uncover the cylinder and mix the suspension using the agitator again at the rate about one cycle per second for one minute;
 - h) After removing the agitator, allow the “free” liquid on it to drain back into the sedimentation cylinder;
 - i) Hydrometer readings shall be taken at elapsed times of approximately: 0.25, 0.50, 1, 2, 4, 8, 15, 30, 60, 120, 240, and 480 minutes. Additional readings can be taken to better define the particle size distribution (gradation) relationship;
 - j) Record the temperature of each hydrometer reading;

After collecting the readings, the sedimentation curve can be created by calculating the maximum particle diameter in suspensions for each hydrometer reading by Eq. (3.7):

$$D = \left(\frac{18 \times \mu \times z}{(\rho_s - \rho_w) \times g \times t} \right)^{1/2} \quad (3.7)$$

where:

- D: particle diameter (mm);
- μ : viscosity of water (g/[cm·s]);
- z: distance the particle falls (cm);
- ρ_s : density of solids (g/cm³);
- ρ_w : water density (g/cm³);
- g: force of gravity (cm/s²);
- t: time for fall (s).

The percent passing the maximum particle diameter (D) is obtained by Eq. (3.8):

$$P(<D) = \frac{\rho_s \times L_c}{(\rho_s - \rho_w) \times M_s} \times 100 \quad (3.8)$$

where:

L_c : is the hydrometer reading in suspension at time (t) and corrected with temperature (T);

M_s : dry solid mass of hydrometer specimen (g).

With the values of maximum particle diameter and percent passing on hands, it is possible to create a sedimentation curve.

Figure 3.3 – Hydrometer analysis sedimentation procedure

(a) 80g of material passing sieve 0.075 mm (#200)

(b) Mixing device

(c) Sedimentation cylinder with suspended material

Source: The author

3.2.4 Laser diffraction method

The following paragraphs describe the procedure to perform measurement of the particles size distribution of a sample of mineral filler by means of laser diffraction with the equipment Mastersizer 2000. The careful preparation of the samples before being taken to the equipment is crucial to obtain reliable test results. If the sample particles are sticking together, dissolving or floating on the surface, or if the sample is not representative, the result will be compromised. About one half of the problems observed when running the measurements is caused by bad sample preparation. (Mastersizer 2000, User Manual).

The Malvern equipment has some tools such as the Standard Operation Process

(SOP), which is a user defined procedure that is easily programmed and allows that samples of a same material to be tested at standard conditions. The SOP is convenient for the operator once the measurement strategy is already settled. It automatically runs through the defined measurement procedure, prompting the operator to perform tasks such as adding the sample. The SOP takes full control of the dispersion unit, making all adjustments and settings (such as pump speeds and ultrasonic power).

In order to perform a measurement, the User Manual indicates the following steps:

- a) Switch on the dispersion unit(s);
- b) Switch on the optical bench;
- c) Switch on the computer and start the software by double-clicking on the Mastersizer 2000 icon;
- d) Start an SOP by selecting Measure-Start SOP - a dialog box will appear showing the measurement display, as indicated in Figure 3.4;

Figure 3.4 – Measurement display dialogue

- e) The Status area at the bottom left of the dialogue reports what is going on and what to do next. The information in the dialogue changes depending on the stage of the measurement. The SOP measurement runs as automatically as possible;
- f) Start adding the sample to the dispersion unit tank;
- g) Once the amount of sample has been added, press the Start button to start the actual measurement;
- h) The SOP measurement will automatically clean and fill the dispersion unit, measure the background, align the optics, calculate the size distribution, and save

the record to a measurement file. All the user has to do is wait for a prompt from the SOP to perform the actions detailed above.

3.2.5 Compaction and coring method

In order to obtain the FAM samples, specimens with 100 mm in diameter are compacted in the Superpave Gyratory Compactor (SGC), with pressure of 600 ± 18 kPa and gyratory angle of $1.25 \pm 0.02^\circ$. The FAM samples are extracted using a diamond drill coupled to a drilling machine, as depicted by Figure 3.5.

Figure 3.5 – Compaction of the SGC specimen and FAM samples extraction

(a) Superpave Gyratory Compactor (SGC)

(b) Drilling machine with diamond drill

(c) SGC specimen after FAM samples were extracted

(d) FAM sample

Source: The author

The stop compaction criterion to the 100 mm specimens is the height of the specimens obtained by assuming the air voids content of 4%. It is in accordance to some researchers that assume that all air voids of the HMA mixture is within the FAM phase (Castelo Branco, 2008; Freire, 2015; Im et al., 2015; Freire et al., 2014, 2015, 2017). It is possible to calculate

the height by knowing the specific gravity of the mixture from ASTM 2041, and the mass of material. Eq (3.9), (3.10), (3.11) and (3.12) show the calculation steps.

$$V_{air} = \frac{G_{mm} - G_{mb}}{G_{mm}} \quad (3.9)$$

$$G_{mb} = G_{mm}(1 - V_{air}) \quad (3.10)$$

$$G_{mb} = \frac{M_{mixture}}{V_{mixture}} = \frac{M_{mixture}}{\pi \left(\frac{d}{2}\right)^2 h} \quad (3.11)$$

$$h = \frac{M_{mixture}}{25\pi} \frac{1}{G_{mm}(1 - V_{air})} \quad (3.12)$$

where:

- V_{air} : percent of air voids in compacted mixtures (%);
- G_{mm} : theoretical maximum specific gravity - ASTM 2041 (g/cm³);
- G_{mb} : bulk specific gravity – ASTM 2726 (g/cm³);
- $M_{mixture}$: mass of the dry mixture in air (g);
- $V_{mixture}$: volume of the compacted mixture (cm³).

3.3 Experimental program and method of analysis

In order to achieve the objectives originally proposed for the research, it is necessary to split the experimental plan into two parts. The first part comprehends the evaluation of the influence of the mineral aggregate type on the estimate of the FAM binder content. Mineral aggregates from three different quarries are used: one basalt and two granites. The two granites are characterized by different specific gravities. The second part cover the evaluation of the effect of aggregate gradation on the estimate of the FAM binder content. Four gradations are tested using the two granite sources.

An asphalt binder graded in the penetration system as 30/45, and graded as a PG 70-16 in the PG system is used to produce all asphalt mixtures. These mixtures were designed according to the Marshall Design Method. The FAM samples will be compacted in the Superpave gyratory compactor, and the FAM specimens will be cored using a diamond drill coupled to a drilling machine.

Dense-graded HMA mixtures will be produced with the basalt and the granites (gradation C of the *Departamento Nacional de Infraestrutura de Transportes, DNIT*). HMA mixtures with three other gradations will also be produced with the granites from the two

different quarries: two Superpave (Superior Preforming Pavements) HMA mixtures with 12.5mm and 9.5mm nominal maximum sizes (NMS), and another composition used by the Road Concession *CCR NovaDutra* which follows the limits of the California State Transportation Agency (Caltrans) and known as *Gap-Graded*. Figure 3.6 depicts the set of HMA mixtures to be used in this study.

Figure 3.6 - HMA mixtures used in this study

After the production of the HMA mixtures, the FAM binder content of each mixture will be determined by means of two methods: Sousa et al. (2013) and Ng et al. (2018). These methods which will be adapted according to the following: (i) the Sousa's method will be performed with the inclusion of the crystallization water in the calculations, in order to determine the error caused when not taking this property into account. The Ng's method will be performed with the measurement of the specific surface area of the mineral filler by means of two tests: sedimentations by Hydrometer Analysis and sedimentation by laser diffraction. Figure 3.7 outlines the experimental plan conceived to achieve such objectives. This combination of factors and levels resulted in an experiment with 27 combinations.

Figure 3.7 – Experimental plan outline

4 RESULTS AND FINDINGS

The objective of this chapter is to show and discuss the results obtained from the tests performed to compare the two FAM design methods: the experimental method of Sousa et al. (2013) with the consideration of the crystallization water, and the one proposed by Ng et al. (2018) with the adjustment in the filler surface area.

4.1 HMA Mixtures

The HMA mixtures used in this research were designed by means of the Marshall method. In the HMA mixture design, specimens are produced with different binder contents to find out the content capable of producing mixtures with 4% air voids. In order to determine the air voids, the mixture maximum specific gravity (G_{mm}) is obtained by the Rice test. More details about this method can be found in Bernucci et al. (2008) and ABNT NBR 15785/2010. The design parameters for the HMA mixtures are presented in Table 4.1 for the mineral aggregates supplied by Bandeirantes Quarry (SP), in Table 4.2 for the mineral aggregates supplied by Pombal Quarry (RJ), and in Table 4.3 for the mineral aggregate supplied by Jambeiro Quarry (SP).

Table 4.1 – HMA mixture parameters for the aggregates supplied by Bandeirantes Quarry (SP)

Gradation	Range C – (DNIT)
Asphalt Binder	CAP 30/45
Asphalt Binder Content (%)	4.5
Asphalt absorption by aggregate (%)	0.6
Air Voids (%)	4.0
Bulk Density (g/cm ³)	2.549
Maximum Specific Gravity - G_{mm} (g/cm ³)	2.655

Table 4.2 – HMA mixture parameters for the aggregates supplied by Pombal Quarry (RJ)

Gradation	Range C - (DNIT)	Superpave - NMAS 12.5mm	Superpave - NMAS 9.5mm	Gap-graded
Asphalt Binder	CAP 30/45	CAP 30/45	CAP 30/45	CAP 30/45
Asphalt Binder Content (%)	5.0	4.9	4.8	4.3
Asphalt absorption by aggregate (%)	0.2	0.3	0.3	0.2
Air Voids (%)	4.2	4.0	3.9	4.9
Bulk Density (g/cm ³)	2.470	2.483	2.483	2.463
Maximum Specific Gravity - G_{mm} (g/cm ³)	2.577	2.587	2.583	2.589

Table 4.3 - HMA mixture parameters for aggregates supplied by Jambeiro Quarry (SP)

Gradation	Range C - (DNIT)	Superpave – NMAAS 12.5mm	Superpave – NMAAS 9.5mm	Gap-graded
Asphalt Binder	CAP 30/45	CAP 30/45	CAP 30/45	CAP 30/45
Asphalt Binder Content (%)	4.5	4.6	4.8	4.7
Asphalt absorption by aggregate (%)	0.2	0.3	0.3	0.4
Air Voids Content (%)	3.9	4.3	4.0	5.1
Bulk Density (g/cm ³)	2.378	2.357	2.355	2.328
Maximum Specific Gravity - Gmm (g/cm ³)	2.474	2.461	2.453	2.453

4.2 FAM mixtures

4.2.1 FAM gradation

The fine aggregate matrices were composed with the aggregate gradation obtained by Eq. (2.1) using the asphalt binder content obtained by the method of Sousa et al. (2013). Figure 4.1 presents the FAM gradation for the mineral aggregate supplied by Jambeiro Quarry (SP).

Figure 4.1 - FAM gradations

The results show that the gradations almost superposed after normalization by Eq. (2.1), despite the differences between the gradations of the original HMA mixtures. It is possible to infer that the mineral aggregate gradation does not present a significant influence on the FAM design. If it is true, one can infer that it will be the difference in the FAM binder

contents that will determine the behavior of the mixtures. Although in the mixture scale variables such as aggregate gradation and binder content are decisive to differentiate one mixture from the other, in the FAM scale, the variable that will best represent its correspondent HMA mixtures is the binder content, and the aggregate gradation will not have a significant effect.

4.2.2 Weighted average specific gravity

The specific gravities of the fine aggregates (F.A.) (materials passing sieves #10, #40, #80) were obtained as per ASTM C 128, and the filler (material passing sieve #200) specific gravities were obtained by applying ASTM C 188. In order to obtain a realistic value for the mixture of aggregates that comprises the FAM gradation, a weighted average between fine aggregates and filler was calculated by means of Eq. (4.1):

$$\rho = \frac{1}{\frac{\%MAF_{fine}}{\rho_{fine}} + \frac{\%MAF_{filler}}{\rho_{filler}}} \times 100 \quad (4.1)$$

The results of the weight average specific gravity for the mineral aggregates are shown in Table 4.4 for the aggregates supplied by Bandeirantes Quarry, in Table 4.5 for the aggregates supplied by Pombal Quarry, and in

Table 4.6 for the aggregates supplied by Jambeiro Quarry.

Table 4.4 – Weighted average specific gravity for the aggregates supplied by Bandeirantes Quarry (SP)

	Range C - (DNIT)	
	F.A.	Filler
Specific gravity (g/cm ³)	3.013	2.769
% of MAF	78.1	21.9
Weighted average (g/cm ³)	2.956	

Table 4.5 – Weighted average specific gravity for the aggregates supplied by Pombal Quarry (RJ)

	Range C - (DNIT)		Superpave - NMAS 12.5mm		Superpave - NMAS 9.5mm		Gap-graded	
	F.A.	Filler	F.A.	Filler	F.A.	Filler	F.A.	Filler
Specific gravity (g/cm ³)	2.864	2.811	2.863	2.811	2.863	2.811	2.860	2.811
% of FAM	78.8	21.2	76.8	23.2	76.4	23.6	79.9	20.1
Weighted average (g/cm ³)	2.852		2.851		2.851		2.850	

Table 4.6 – Weighted average specific gravity for the aggregates supplied by Jambeiro Quarry (SP)

	Range C - (DNIT)		Superpave - NMAS 12.5mm		Superpave - NMAS 9.5mm		Gap-graded	
	F.A.	Filler	F.A.	Filler	F.A.	Filler	F.A.	Filler
Specific gravity (g/cm ³)	2.661	2.637	2.628	2.637	2.650	2.637	2.664	2.637
% of FAM	77.3	22.7	76.1	23.9	76.6	23.4	74.7	25.3
Weighted average (g/cm ³)	2.656		2.630		2.647		2.657	

4.3 Crystallization Water

In order to measure the amount of crystallization water present in the fine aggregates, the procedure described by ASTM D6307-16 was reproduced for the three mineral aggregates used in this study. The procedure was carried out in a muffle furnace (EDG, model 3P-S) with adaptations on temperature and material gradation.

The following calibration factors were found: 0.51% for the basalt rock from Bandeirantes Quarry (SP), 0.27% for the granite from Jambeiro Quarry (SP), and 0.11% for the granite from Pombal Quarry (RJ). These results show the great importance of taking the crystallization water into account in order to determinate the asphalt binder content by calcination in an ignition oven or in a muffle furnace. A 0.5% difference in the asphalt content could produce a FAM mixture that does not precisely represent the original HMA mixture.

4.4 Sedimentation by hydrometer analysis

The sedimentation curves of the fillers were built by means of hydrometer analysis in order to obtain the particle size distribution (PSD). The values of maximum particle diameter in suspension and percent passing along time is presented in Table 4.7. These values were used to build the sedimentation curves depicted in Figure 4.2.

Based on those curves, it was possible to estimate the filler specific surface area by using the same procedure developed by Arrambide and Duriez (1959). However, as hydrometer analysis is based on Stokes' law, where particles are considered perfect spheres, Eq. (2.3), (2.4) and (2.5) had to be adapted, and the area and volume were calculated according to Eq.(4.1) and (4.2), where “r” is the radius of the sphere (m). The diameter “d” of the sphere is defined by Eq. (4.3) and can be calculated as the average of the nominal maximum aggregate diameter for each interval between the maximum particles diameters obtained by Eq. (3.7). This way, the total specific surface of the filler is the sum of the coefficients multiplied by the percent of particle between two diameters, for each interval, as per Eq. (2.6).

Table 4.7 – Hydrometer Analysis of the fillers

Time (min)	Pombal (RJ)		Jambeiro (SP)		Bandeirantes (SP)	
	Diameter (mm)	% passing	Diameter (mm)	% passing	Diameter (mm)	% passing
0.25	0.076	86.9	0.082	84.5	0.077	88.9
0.5	0.057	75.7	0.061	74.4	0.056	81.0
1	0.043	58.2	0.046	59.7	0.042	67.0
2	0.032	43.1	0.034	43.4	0.032	54.1
4	0.024	31.5	0.025	31.8	0.023	40.2
8	0.017	20.8	0.018	21.7	0.017	30.2
15	0.013	15.9	0.013	17.6	0.012	24.3
30	0.009	10.1	0.009	12.5	0.009	17.3
60	0.006	8.1	0.007	9.5	0.006	12.3
120	0.005	5.8	0.005	7.4	0.005	9.3
240	0.003	3.4	0.003	7.2	0.003	7.1

$$A = 4 \times \pi \times r^2 \quad (4.1)$$

$$V = \frac{4 \times \pi \times r^3}{3} \quad (4.2)$$

$$d = \frac{D_i - D_{ii}}{2} \quad (4.3)$$

Figure 4.2 – Sedimentation curves of the fillers

The filler specific surface areas obtained for the three aggregates were: 206 m²/kg for the aggregate supplied by Bandeirantes Quarry (SP), 177 m²/kg for the aggregate supplied by Jambeiro Quarry (SP), and 135 m²/kg for the aggregate supplied by Pombal Quarry (RJ). It is possible to infer that the value of 135 m²/kg suggested by Arrambide and Duriez (1959) is an estimation which is not representative of all cases. The main implication of adopting such a fixed value for any material is that the estimated binder content may produce asphalt mixtures with less binder content than the necessary to represent the correspondent HMA mixture.

Ng et al. (2018) adopted the suggested value of 135 m²/kg in the calculations of the specific surface area instead of carrying out the hydrometer analysis. In spite of that, Ng et al. (2018) obtained binder contents by the method proposed by Sousa et al. (2013) that are very close to the ones obtained by the method of the specific surface area. However, the results shown here make it clear that the estimation of binder content by the method proposed by Ng et al. (2018) for other mineral aggregates should be adapted to take into account the values of specific surface area of fillers obtained by other methods, such as sedimentation by hydrometer analysis.

4.5 Particle size distribution by Malvern Mastersizer 2000

The equipment Malvern Mastersizer 2000 was used to obtain the filler specific surface area by laser diffraction for the three sources of mineral aggregate. This method consists in measuring the intensity of laser light scattered on the aggregate particles. The intensity of scattered light depends on the particles size in the measurement system. The smaller the particle, the greater the angle at which the light is scattered/refracted.

The results of the specific surface area of the fillers are presented in Table 4.8. The values obtained by laser diffraction are 3 to 4 times higher than the ones obtained by sedimentation by hydrometer analysis. The highest specific surface area was obtained for the basalt and the values for the granites are close to each other for both methods. However, it is interesting to see that the specific surface area for the sedimentation by hydrometer analysis for the mineral aggregate from Jambeiro Quarry is 31% higher than the one for the mineral aggregate from the Pombal Quarry, while the difference in laser diffraction for these two materials is 3.5%.

Table 4.8 – Filler specific surface area for the mineral aggregates

	Mastersizer 2000 (m ² /kg)	Hydrometer Analyses (m ² /kg)	Ratio (Master/ Hydrometer)
Bandeirantes Quarry	776	206	3.8
Jambeiro Quarry	470	177	2.7
Pombal Quarry	454	135	3.4

As presented in the literature review, there are practical difficulties in measuring the real surface area due to the irregular shape and particle texture. The sphericity of the particles is a relevant factor in the differences between the specific surface area for both methods. The sedimentation method assumes a single particle density, whereas the laser diffraction is independent of density. This implies that an irregular shaped particle has longer settling times than their equivalent spheres, which result in different specific surface areas.

Figure 4.3a presents a comparison of the results for the specific surface area of the three mineral aggregates for both methods and the fitted linear model. The adjusted line does not intersect the origin as in reality it is not possible to have a non-zero value obtained with the Mastersizer 2000 with a zero content of a given fraction of the material. In order to correct it, the model was rebuilt by enforcing it to cross the origin. After doing it, a new fitted line was obtained (Figure 4.3b).

Figure 4.3 – Comparison of filler specific surface area for two different methods: Mastersizer 2000 and Hydrometer Analysis

Figures 4.4 to 4.6 presents the gradation curves obtained from the sedimentation by the hydrometer analysis and from the particle size distribution by laser diffraction in the Mastersizer 2000. As can be seen, the curves overlapped. The estimates of the specific surface area for both methods are the same, and it is defined by the average of the nominal aggregate diameter for each sieve interval. It is possible to infer that the estimate of specific surface area is more accurate when performed by laser diffraction as it produces more diameter intervals.

Figure 4.4 – Gradation curves obtained by sedimentation (HA) and laser diffraction (M2000) for the mineral aggregates provided by Pombal Quarry.

Figure 4.5 - Gradation curves obtained by sedimentation (HA) and laser diffraction (M2000) for the mineral aggregates provided by Jambeiro Quarry.

Figure 4.6 - Gradation curves obtained by sedimentation (HA) and laser diffraction (M2000) for the mineral aggregates provided by Bandeirantes Quarry.

4.6 Richness modulus “K”

As discussed in the literature review, the choice of a richness modulus is driven basically by the purpose of the wearing coarse surface, i.e., 3.75 is the average value for highways and 4.0 for dense mixtures in airports. In other words, there is no objective criterion to choose the richness modulus (Arrambide and Duriez, 1959). On the other hand, by isolating the richness modulus “K” from Eq. (3.5) and (3.6), it is possible to calculate the exact value that results in a binder content equal to the one obtained by the procedure of Sousa et al. (2013), as presented in Eq. (4.4), where ($P_{bi,CW}$) is the binder content with the consideration of the crystallization water, and (S_{SFAM}) is the specific surface area of the FAM.

$$K_{FAM} = \frac{1}{\sqrt[5]{S_{SFAM}}} \times \left(\frac{100 \times P_{bi,CW}}{100 \times (1 + \%A_{abs}) - P_{bi,CW}} \right) \quad (4.4)$$

In order to evaluate the influence of the specific surface area of particles passing sieve #200 (0.074 mm) obtained by the hydrometer analysis and by the Mastersizer 2000, and the influence of the shape of the particles, the richness modulus “K” was calculated using the S_{SFAM} values presented in Table 4.8. The results of the richness modulus for the nine FAMs with the consideration of the crystallization water are presented in Table 4.9. Table 4.10

presents the results of “K” for the HMA mixtures.

Table 4.9 – Richness modulus of the FAMs calculated using the binder contents obtained by Sousa et al. (2013) with the consideration of the crystallization water

		Binder content by Sousa et al. (2013) - CW	A	B	C	D
Jambeiro (SP)	Range C - (DNIT)	7.7%	3.8	3.9	3.2	3.2
	NMAS 9.5 mm	9.0%	4.5	4.6	3.8	3.8
	NMAS 12.5 mm	8.4%	4.1	4.2	3.5	3.5
	Gap Graded	7.9%	3.8	3.9	3.2	3.3
Pombal (RJ)	Range C - (DNIT)	9.3%	4.9	5.0	4.0	4.0
	NMAS 9.5 mm	9.7%	5.0	5.2	4.1	4.1
	NMAS 12.5 mm	9.6%	5.0	5.1	4.1	4.1
	Gap-graded	9.4%	5.0	5.1	4.1	4.1
Bandeirantes (SP)	Range C - (DNIT)	9.0%	4.4	4.5	3.5	3.5

Notes: A: assumes particle shape as perfect cubes; specific surface area of particles passing sieve #200 obtained by hydrometer analysis.

B: assumes particle shape as perfect spheres; specific surface area of particles passing sieve #200 obtained by hydrometer analysis.

C: assumes particle shape as perfect cubes; specific surface area of particles passing sieve #200 obtained by Mastersizer 2000.

D: assumes particle shape as perfect spheres; specific surface area of particles passing sieve #200 obtained by Mastersizer 2000.

From Table 4.9 and Table 4.10, it is possible to see that there is a pattern in the results obtained for the mineral aggregates supplied by Pombal Quarry for the four gradations. However, the same pattern cannot be seen for the mineral aggregates supplied by Jambeiro Quarry. So, the point is how to choose the adequate richness modulus that is capable of producing FAMs that represent the original HMA mixtures. Results in Table 4.9 and Table 4.10 also show that the “K” values are not significantly influenced by the assumed shape of the particles (cube or sphere). Table 4.11 presents the differences calculated by the average values of the columns.

Table 4.10 - Richness modulus of the HMA mixtures calculated with basis on the binder contents

		Binder content HMA mix design	E	F	G	H
Jambeiro (SP)	Range C - (DNIT)	4.5%	2.6	2.7	2.2	2.2
	NMAS 9.5 mm	4.8%	2.8	2.9	2.4	2.4
	NMAS 12.5 mm	4.6%	2.8	2.8	2.3	2.4
	Gap-graded	4.7%	2.9	3.0	2.5	2.5
Pombal (RJ)	Range C - (DNIT)	5.0%	3.0	3.1	2.5	2.5
	NMAS 9.5 mm	4.8%	2.9	3.0	2.4	2.4
	NMAS 12.5 mm	4.9%	3.1	3.1	2.5	2.5
	Gap-graded	4.3%	2.9	3.0	2.4	2.4
Bandeirantes (SP)	Range C - (DNIT)	4.5%	2.5	2.6	2.0	2.0

Note: E: assumes particle shape as perfect cubes; specific surface area of particles passing sieve #200 obtained by hydrometer analysis.

F: assumes particle shape as perfect sphere; specific surface area of particles passing sieve #200 obtained by hydrometer analysis.

G: assumes particle shape as perfect cubes; specific surface area of particles passing sieve #200 obtained on Mastersizer 2000.

H: assumes particle shape as perfect spheres; specific surface area of particles passing sieve #200 obtained on Mastersizer 2000.

Table 4.11 - Differences of the average values of richness modulus by assuming the shape of the particles as perfect cube or spheres

FAM	A-B	C-D
Differences	1.8%	0.6%
HMA	E-F	G-H
Differences	2.2%	0.9%

In an attempt to predict the value of the richness modulus to be used to estimate the FAM binder content, a correction coefficient (α) relative to the density of the aggregates was applied. Table 4.12 presents the steps of the calculations to determine a coefficient that relates the FAM richness modulus (K_{FAM}) to the richness modulus (K_{HMA}) of the HMA mixture, when assuming the particle shape as perfect cubes and for the specific surface area of particles passing sieve #200 obtained by hydrometer analysis. Table 4.13 shows the same calculations for the specific surface area obtained by the Mastersizer 2000.

Table 4.12 – Relation between richness modulus for particle shape as perfect cubes and specific surface area obtained by hydrometer analysis

		K_{FAM}	K_{HMA}	ρ_{agg} (g/cm ³)	$\alpha = \frac{2.650}{\rho_{agg}}$	$C = \frac{K_{FAM}}{K_{HMA}} \times \alpha$
Jambeiro (SP)	Range C - (DNIT)	3.8	2.6	2.656	1.00	1.5
	NMAS 9.5 mm	4.5	2.8	2.647	1.00	1.6
	NMAS 12.5 mm	4.1	2.8	2.63	1.01	1.5
	Gap-graded	3.8	2.9	2.657	1.00	1.3
Pombal (RJ)	Range C - (DNIT)	4.9	3.0	2.852	0.93	1.5
	NMAS 9.5 mm	5.0	2.9	2.851	0.93	1.6
	NMAS 12.5 mm	5.0	3.1	2.851	0.93	1.5
	Gap-graded	5.0	2.9	2.85	0.93	1.6
Bandeirantes (SP)	Range C - (DNIT)	4.4	2.5	2.956	0.90	1.6
Average						1.51

Table 4.13 - Relation between richness modulus for particle shape as perfect cubes and specific surface area obtained on Mastersizer 2000

		K_{FAM}	K_{HMA}	ρ_{agg} (g/cm ³)	$\alpha = \frac{2.650}{\rho_{agg}}$	$C = \frac{K_{FAM}}{K_{HMA}} \times \alpha$
Jambeiro (SP)	Range C - (DNIT)	3.2	2.2	2.656	1.00	1.4
	NMAS 9.5 mm	3.8	2.4	2.647	1.00	1.6
	NMAS 12.5 mm	3.5	2.3	2.63	1.01	1.5
	Gap-graded	3.2	2.5	2.657	1.00	1.3
Pombal (RJ)	Range C - (DNIT)	4.0	2.5	2.852	0.93	1.5
	NMAS 9.5 mm	4.1	2.4	2.851	0.93	1.6
	NMAS 12.5 mm	4.1	2.5	2.851	0.93	1.5
	Gap-graded	4.1	2.4	2.85	0.93	1.6
Bandeirantes (SP)	Range C - (DNIT)	3.5	2.0	2.956	0.90	1.6
Average						1.51

The results presented allow one to infer that 1.51 is a factor that could be used to multiply the richness modulus of the HMA mixture in order to predict the richness modulus to be used to estimate the FAM binder content by the method proposed by Ng et al. (2018), as shown in Eq. (4.5).

$$K_{FAM} = 1.51 \times K_{HMA} \times \frac{\rho_{agg}}{2.650} \quad (4.5)$$

4.7 Study of FAM design methods

The FAM design procedures were replicated for the nine FAMs assessed in this study: one produced with basalt rock from Bandeirantes Quarry, four produced with granites from Jambeiro Quarry, and four produced with granites from Pombal Quarry. The same design protocol was reproduced for the three mineral aggregates and for the different gradations.

4.7.1 Method proposed by Sousa et al. (2013)

In order to reproduce the method developed by Sousa et al. (2013), a muffle furnace (model EDG, 3P-S) was used in substitution for the ignition oven, and the calcination of the asphalt binder was carried out at 427°C as recommended by Sousa et al. (2013). The calculation of the FAM binder content takes into account only the material whose particles pass sieve #10 (particles smaller than 2.00mm) and disregard the fine particles glued to the coarse aggregate particles.

Table 4.14 presents the binder contents of the HMA mixtures for the three mineral aggregates, the binder contents obtained by the Sousa's method for the aggregates supplied by Jambeiro e Pombal Quarry in the four gradations, and the binder contents obtained by Sousa's method with the consideration of the crystallization water. The error caused by ignoring the calibration factor derived from the crystallization water is also shown in Table 4.14.

Table 4.14 – Binder content by Sousa et al. (2013) and with the consideration of the crystallization water (CW)

		HMA mix design	Sousa et al.(2013)	Sousa et al. (2013) - CW	Error (%)
Jambeiro (SP)	Range C - (DNIT)	4.5%	7.9%	7.7%	3%
	NMAS 9.5 mm	4.8%	9.3%	9.0%	3%
	NMAS 12.5 mm	4.6%	8.7%	8.4%	3%
	Gap-graded	4.7%	8.2%	7.9%	3%
Pombal (RJ)	Range C - (DNIT)	5.0%	9.4%	9.3%	1%
	NMAS 9.5 mm	4.8%	9.8%	9.7%	1%
	NMAS 12.5 mm	4.9%	9.7%	9.6%	1%
	Gap-graded	4.3%	9.5%	9.4%	1%
Bandeirantes (SP)	Range C - (DNIT)	4.5%	9.5%	9.0%	5%

A comparison of such results is depicted in Figure 4.7, which shows that the position of a certain HMA mixture in a rank order of the binder content is not the same of the correspondent FAM. This was observed for both mineral aggregates supplied by the Jambeiro Quarry and the mineral aggregates supplied by the Pombal Quarry. Such results contradict the ones that could be expected if one assumes that the FAM represents the fine matrix of the full mixture and because of that the binder content of the fine matrix of the HMA mixture should be directly proportional to the binder content of the full HMA mixture.

Figure 4.7 – Binder content for the full HMA mixture and obtained by Sousa et al. (2013) with the consideration of the crystallization water (CW)

4.7.2 Method proposed by Ng et al. (2018)

The determination of the FAM binder content by the method proposed by Ng et al. (2018) is based on the following assumptions: (i) the shape of the aggregates particles is a perfect cube; (ii) the diagonal of the cube for each sieve interval is the average of the maximum nominal sieve opening of each sieve interval; and (iii) the specific gravity of fine aggregates is the weighted average specific gravity of fine aggregates and fillers. These assumptions affect three critical parameters: (i) the FAM aggregate mixture specific gravity; (ii) the surface area of the portion of particles sizes smaller than 0.075mm; and (iii) the richness modulus “K” of the mixtures.

Regarding the specific gravity of the aggregate mixtures, the weight average was adopted following the assumption made by Ng et al. (2018). For the richness modulus “K”, it was adopted the value calculated by Eq. (4.5), which relates the richness modulus of the HMA mixture to the richness modulus of the FAM. There remains some room for discussion

about the choice of the method to determine the specific surface area of particles passing sieve #200 (0.075 mm).

Table 4.15 presents the binder contents obtained by applying the Ng's method using the specific surface area determined by hydrometer analysis. Table 4.16 presents the binder contents by means of the same method and using the specific surface areas obtained by the Mastersizer 2000.

Table 4.15 – Binder contents as per Ng et al. (2018) using the specific surface areas obtained by hydrometer analysis

		K_{HMA}	ρ_{agg} (g/cm ³)	K_{FAM} (calculated)	Sousa et al. (2013) - CW	Ng et al. (2018) – H.A.
Jambeiro (SP)	Range C - (DNIT)	2.6	2.656	3.93	7.7%	7.9%
	NMAS 9.5 mm	2.8	2.647	4.27	9.0%	8.6%
	NMAS 12.5 mm	2.8	2.630	4.13	8.4%	8.3%
	Gap-graded	2.9	2.657	4.44	7.9%	9.0%
Pombal (RJ)	Range C - (DNIT)	3.0	2.852	4.86	9.3%	9.2%
	NMAS 9.5 mm	2.9	2.851	4.75	9.7%	9.4%
	NMAS 12.5 mm	3.1	2.851	4.93	9.6%	9.7%
	Gap-graded	2.9	2.850	4.68	9.4%	9.0%
Bandeirantes (SP)	Range C - (DNIT)	2.5	2.956	4.27	9.0%	8.7%

Table 4.16 - Binder contents as per Ng et al. (2018) by using the specific surface areas obtained by the Mastersizer 2000

		K_{HMA}	ρ_{agg} (g/cm ³)	K_{FAM} (calculated)	Sousa et al. (2013) - CW	Ng et al. (2018) – M2000
Jambeiro (SP)	Range C - (DNIT)	2.2	2.656	3.31	7.7%	7.9%
	NMAS 9.5 mm	2.4	2.647	3.60	9.0%	8.6%
	NMAS 12.5 mm	2.3	2.630	3.49	8.4%	8.4%
	Gap-graded	2.5	2.657	3.74	7.9%	9.0%
Pombal (RJ)	Range C - (DNIT)	2.5	2.852	4.00	9.3%	9.3%
	NMAS 9.5 mm	2.4	2.851	3.90	9.7%	9.5%
	NMAS 12.5 mm	2.5	2.851	4.05	9.6%	9.8%
	Gap-graded	2.4	2.850	3.87	9.4%	9.0%
Bandeirantes (SP)	Range C - (DNIT)	2.0	2.956	3.37	9.0%	8.7%

The results show that in most of the cases the binder contents obtained for both methods are close to each other and the binder content calculated for the gap-graded gradation for the mineral aggregate supplied by Jambeiro Quarry resulted in the largest difference. Figure 4.8 shows the relationship between the binder contents obtained by the Sousa's

method, with the consideration of the crystallization water, and by the Ng's method using the specific surface area of particles passing sieve #200 obtained by hydrometer analysis and Mastersizer 2000, and in this figure it is also possible to visualize how distant the binder contents obtained by both methods are from each other. After excluding this data, the correlation jumped to an $R^2 = 0.8402$ for the hydrometer analysis and an $R^2 = 0.9948$ for the Mastersizer 2000, as indicated in Figure 4.9. As can be seen, there are objective and scientific reasons for classifying such a point as an outlier, which support its suppression.

Figure 4.8 Relationship between the binder contents obtained by the Sousa's method with the consideration of the crystallization water, and by the Ng's method using the specific surface area of particles passing sieve #200 obtained by hydrometer analysis (HA) and Mastersizer 2000 (M2000) – considering all FAMs

Figure 4.9 - Relationship between the binder contents obtained by the Sousa's method with the consideration of the crystallization water, and by the Ng's method using the specific surface area of particles passing sieve #200 obtained by hydrometer analysis (HA) and Mastersizer 2000 (M2000) – excluding the binder contents of the gap-graded gradation

By comparing the FAM binder contents provided by the Sousa's method with the ones calculated by the Ng's method, it is possible to conclude that the method to determine the FAM binder content by means of the specific surface area is capable to yield the FAM binder content provided that the richness modulus "K" is calculated by Eq. (4.5). The best equivalence between the results yielded by both methods were obtained when the determination of the specific surface area of the filler is carried out with the Mastersizer 2000. On the other hand, for practical purposes, the method of laser diffraction for determining the filler specific surface area seems to be expensive and could be a limitation for some laboratories, which highlights the importance of the results obtained here with the determination of the filler specific area using hydrometer analysis.

4.7.3 Final remarks on the evaluation of the methods used to estimate the FAM binder content

After the reproduction of both methods (Sousa's and Ng's), the results led to the proposal of refinements that could help improve the efficiency of the procedures and the achievement of more reliable results.

As far as the procedure developed by Sousa et al. (2013) is concerned, the recommendation is to include the crystallization water of the mineral aggregates in the calculations, when calcining the asphalt mixture in the ignition oven. This step will determine the amount of water that evaporates from the mineral aggregate structure. This value can influence the mass difference and so the estimate of the binder content. The general recommendation is perform the test as per ASTM D6307-16 and use Eq. (3.3) to calculate the binder content by means of the Sousa's method.

Regarding the procedure developed by Arrambide and Duriez (1959) and adapted for FAMs by Ng et al. (2018), the results led to the proposal of two refinements: (i) to estimate the specific surface area of the filler by means of a more precise method (such as laser diffraction) and (ii) to estimate the richness modulus of the FAM obtained from the full HMA mixture. After applying these refinements, the method of the specific surface area proved to be effective with a good correlation coefficient ($R^2 = 0.9948$) in a way that is simpler, faster, less operator- and equipment-dependent than other methods available in the literature.

In summary, the following steps are proposed to estimate the FAM binder content by means of the specific surface area of the mineral aggregates:

- 1) Design of the HMA mixture as per Marshall or Superpave® methods.

Information such as gradation, bulk specific gravity and absorption of the mineral

aggregates will be determined;

- 2) The FAM gradation can be obtained from the gradation of the HMA mixture by using Eq. (2.1);

$$\frac{\text{Mass of aggregate passing sieve \#ii in full mixture}}{\text{Mass of aggregate passing sieve \#16 in full mixture}} \times 100\% \quad (2.1)$$

- 3) Perform one of these tests to estimate the specific surface area of the mineral filler: sedimentation by hydrometer analysis or particle size distribution by laser diffraction;
- 4) With the data from the HMA mixture (gradation and bulk specific gravity of the mineral aggregates) on hands, it is possible to calculate the specific surface area of the HMA mixture by using Eq (2.2) to (2.6);

$$S = \frac{A}{V \times \rho} \quad (2.2)$$

$$A = 6 \times a \quad (2.3)$$

$$V = a^3 \quad (2.4)$$

$$d = a \times \sqrt{3} \quad (2.5)$$

$$S_s = \frac{\sum (S_i \times P_i)}{100} \quad (2.6)$$

- 6) With the data from the FAM (gradation, bulk specific gravity of the mineral aggregates and specific surface area of the filler) on hands, it is possible to calculate the specific surface area of the FAM fraction by equations Eq. (2.2) to (2.6);
- 7) By knowing the binder content of the HMA mixture from the design in step 1 and the specific surface area obtained in step 4, it is possible to calculate the richness modulus “K” of the HMA mixture by Eq. (4.4);

$$K_{HMA} = \frac{1}{\sqrt[5]{S_{SHMA}}} \times \left(\frac{100 \times P_{bi,HMA}}{100 \times (1 + \%A_{abs}) - P_{bi,HMA}} \right) \quad (4.4)$$

- 8) By using the “K_{HMA}”, it is possible to estimate the FAM richness modulus “K” of the by Eq. (4.5);

$$K_{FAM} = 1.51 \times K_{HMA} \times \frac{P_{agg}}{2.650} \quad (4.5)$$

- 9) By knowing the S_{SFAM} from step 5 and “ K_{FAM} ” from step 7, it is possible to estimate the FAM binder content by Eq. (3.5) and Eq. (3.6).

$$T_{FAM} = \frac{100 \times K \times \sqrt[5]{S_{SFAM}}}{100 + (K \times \sqrt[5]{S_{SFAM}})} \quad (3.5)$$

$$T'_{FAM} = T_{FAM} \times (1 + \%A_{abs}) \quad (3.6)$$

4.8 Compaction of the FAM specimens

The specimens were compacted using only the binder contents obtained by the Sousa’s method. Specimens with 100 mm in diameter were compacted in the SGC with the specimen height as the stop criterion. However, it was not possible to achieve the calculated height for some specimens. In some cases, the specimen height became constant before the desired height was achieved. It means that it would not be possible to reduce the specimen height even with more gyrations. Sousa et al. (2013) also compacted the specimens up to no changes in specimen height with additional gyrations.

For the specimens that achieved the calculated height, a difference between the calculated values and the values measured by the equipment was observed. This difference is intrinsic to the SGC and its resolution, but it showed to have no significant impact on the specimen air voids as all differences were lower than 1 mm.

The parameters used for the compaction of the specimens are presented in Table 4.17 for the mineral aggregates provided by the Pombal Quarry and in Table 4.18 for the mineral

Table 4.17 – Compaction parameters and air voids of SGC specimens prepared with mineral aggregates supplied by the Pombal Quarry

Gradation Curve	Range C - (DNIT)	NMAS 12.5mm	NMAS 9.5mm	Gap Graded
Gmm (g/cm ³)	2.420	2.408	2.399	2.416
mass of mixture (g)	1198.5	1199.5	1197.6	1198.4
calculated height (mm)	65.7	66.1	66.2	65.8
final height (mm)	67.1	67.2	67.0	66.7
gyrations	235	202	217	185
automatic stop	No	No	Yes	Yes
FAM air voids (%)	5.1	4.9	4.0	4.6
HMA air voids (%)	4.2	4.0	3.9	4.9

aggregates provided by the Jambeiro Quarry. The Gmm values were obtained by applying the procedure ASTM 2041. The variation in the mass of mixture (1,200 g) is due to the manual

process of mixing the material, in which the material can adhere to the mixer. However, this difference has no significant impact on the process.

Table 4.18 – Compaction parameters and air voids of SGC specimens prepared with mineral aggregates supplied by the Jambeiro

Gradation Curve	Range C – (DNIT)	NMAS 12.5mm	NMAS 9.5mm	Gap Graded
Gmm (g/cm ³)	2.340	2.316	2.297	2.336
Mass of mixture (g)	1193.2	1196.3	1195.9	1195.2
calculated height (mm)	67.6	68.5	69.0	67.9
final height (mm)	68.8	69.0	69.2	69.0
gyrations	324	194	117	246
automatic stop	No	Yes	Yes	No
FAM air voids (%)	4.4	4.2	4.1	4.5
HMA air voids (%)	3.9	4.3	4.0	5.1

The air voids for the specimens of 100 mm in diameter were calculated using Eq. (3.9), where the bulk specific gravity was obtained by ASTM 2726. For the mixtures prepared with the aggregates supplied by Pombal Quarry, the air voids of 4% was reached only for the gradation with NMAS of 9.5 mm. For the other specimens, the air voids resulted higher than the target value. However, for the mixtures prepared with the aggregates supplied by Jambeiro Quarry, the four specimens reached air voids close to 4%. It is hypothesized that the height of the specimens could be achieved, allowing the air voids to be reached, even with less gyrations, if the compaction pressure were increased.

Despite the differences between the air voids found for the SGC specimens and the target value of 4%, in most of the cases, the air voids for the SGC specimens matched the design air voids for the HMA mixtures. Except for the specimens prepared with the Range C – (DNIT) and the NMAS of 12.5 mm for the aggregates supplied by Pombal Quarry, the other specimens seem to have provided FAMs that represent the full HMA mixtures.

4.9 Air voids content of the FAM samples

After the compaction of the specimens in the SGC, the FAM samples were cored out. The FAM cylinders were sawed to 45 mm in height aiming to fit the DSR dimensions. It was possible to extract around 14 FAM cylinders from a single SGC specimen with a diamond drill coupled to a drilling machine of 0.5 inches in diameter.

The air voids of the FAMs cylinders are depicted in Figure 4.10 and Figure 4.11 for the mixtures produced with the aggregates supplied by Pombal and Jambeiro Quarries,

respectively. The FAM air voids correspond in average to 75% of the air voids of the SGC specimens for both materials. If the hypothesis that the air voids in the FAM scale are equal to those observed for HMA mixtures, as hypothesized by Castelo Branco (2008), Freire (2015), Im et al. (2015) and Freire et al. (2014, 2015, 2017), the SGC specimens should be compacted assuming higher air voids.

By looking into the differences in air voids for the FAM specimens extracted from the same SGC specimens, it is possible to see that none of them differs more than 35% from each other. Furthermore, by looking into the samples for both materials for the same gradation, one can see that they all have a difference in air voids lower than 35%. According to Castelo Branco (2008), this variation of 35% is acceptable and the samples can be compared. Underwood and Kim (2013) evaluated the effect of volumetric factors on FAM mechanical behavior and stated that in order to compare results the replicates must be within $\pm 0.5\%$ of the reported mean. Border effect was not observed in the samples cored from the SGC specimens.

Figure 4.10 - Air voids distribution of FAM samples extracted from the SGC specimens for the four gradations – Pombal Quarry

Figure 4.11 – Air voids distribution of FAM samples extracted from the SGC specimens for the four gradations – Jambeiro Quarry

5 CONCLUSIONS AND RECOMMENDATIONS

This research was focused on the evaluation of the specific surface method as an alternative tool to estimate the binder content of fine aggregate matrices (FAMs). The major findings of this study are presented below:

- the method based on the asphalt ignition (Sousa's method) showed that the asphalt mixtures and their correspondent FAMs do not occupy the same position when ranked in terms of their binder contents; this was expected in a certain way as variables relative to the mineral aggregate, such as type, gradation, specific gravity and absorption, have decisive influence on the determination of the FAM binder contents;

- a careful look at the calculations of the method based on the mineral aggregate specific surface area (Ng et al. 2018) leads to four main variables that are capable of influencing the results, i.e., (i) gradation, (ii) coefficient for filler surface area, (iii) richness modulus and (iv) mineral aggregate specific gravity. The results obtained in this study led to the following approaches to these variables: (i) by normalizing the percentages of material passing the cutting sieve, the gradation curves are nearly the same, which is a way of controlling the effect of aggregate gradation on FAM properties; (ii) the filler specific surface area should be estimated by means of the laser diffraction (Mastersizer 2000) or by means of the hydrometer analysis and the sedimentation curve based on the Stokes' law; (iii) a model was presented to estimate the richness modulus which is based on the HMA richness modulus and in a correction factor, and (iv) the specific gravity was adopted as the weight average;

- a relationship between the richness modulus of the HMA mixture and the FAM binder content was established, which turns the method based on the specific surface area of fine aggregates into a viable and promising tool to estimate the FAM binder content;

- the method based on the mineral aggregate specific surface area (Ng et al. 2018) showed to be capable of estimating the binder contents to be used in the composition of FAMs; such binder contents are comparable to the ones obtained by the Sousa's method provided that a coefficient of correction relative to the aggregate crystallization water is applied;

- the estimate of the filler specific surface area by laser diffraction and hydrometer analysis resulted in overlapped gradation curves, which allows one to infer that both methods are equivalent tools for this aim; the surface area obtained by the Mastersizer 2000 is higher than the one obtained by hydrometer analysis because the Mastersizer has a larger number of diameter intervals, resulting in a more accurate value;

- the compaction of the specimens on the SGC proved to be effective in ensuring a homogenous distribution of the air voids content between the FAM samples, as no evidences of edge effects were noticed; and

- the air voids of the SGC specimens showed correlation with the air voids of the HMA mixtures, and for all FAM samples the coefficient of variation is less than 10%.

The method based on the mineral aggregate specific surface area (Ng et al. 2018) proved to be effective in estimating the asphalt content of fine aggregate matrices in a way that is simpler, faster, less operator- and equipment-dependent than other methods available in the literature; it is believed that by making the determination of FAM asphalt content a simpler, easier, faster and cheaper process, a greater number of researchers will be interested in developing studies in this area.

Recommendations for future work include:

- (i) the evaluation of the influence of other sources of mineral aggregate and other types of filler (such as lime or Portland cement) on the estimation of the FAM binder content;
- (ii) the reproduction of this test set with modified binders in order to check the validity of the conclusions obtained in this study to modified binders.

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APPENDIX A – Mastersizer 2000 Result Analysis Report

MASTERSIZER

Result Analysis Report

Sample Name:
Basalto - Bandeirantes - Average
Sample Source & type:
Supplier = Adrian Grasson USP
Sample bulk lot ref:

SOP Name:
Measured by:
Laura
Result Source:
Averaged

Measured:
terça-feira, 22 de janeiro de 2019 10:54:47
Analysed:
terça-feira, 22 de janeiro de 2019 10:54:48

Particle Name:
Fraunhofer
Particle RI:
0.000
Dispersant Name:
Water

Accessory Name:
Hydro 2000MU (A)
Absorption:
0
Dispersant RI:
1.330

Analysis model:
General purpose
Size range:
0.020 to 2000.0... μm
Weighted Residual:
0.887 %

Sensitivity:
Normal
Obscuration:
13.32 %
Result Emulation:
Off

Concentration:
0.0157 %Vol

Span :
2.630

Uniformity:
0.823

Result units:
Volume

Specific Surface Area:
0.776 m^2/g

Surface Weighted Mean D[3,2]:
7.731 μm

Vol. Weighted Mean D[4,3]:
37.050 μm

d(0.1): 3.051 μm

d(0.5): 30.041 μm

d(0.9): 82.069 μm

Size (μm)	Volume in %	Size (μm)	Volume in %	Size (μm)	Volume in %	Size (μm)	Volume in %	Size (μm)	Volume in %	Size (μm)	Volume in %
0.010	0.00	0.105	0.00	1.096	0.78	11.482	2.64	120.226	1.13	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.84	13.183	2.89	138.038	0.19	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.90	15.136	3.15	158.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	0.94	17.378	3.43	181.970	0.00	1905.401	0.00
0.017	0.00	0.182	0.00	1.905	0.98	19.953	3.75	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	1.01	22.909	4.10	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	1.05	26.303	4.50	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	1.10	30.200	4.93	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.01	3.311	1.16	34.674	5.36	363.078	0.00	3801.804	0.00
0.035	0.00	0.363	0.06	3.802	1.23	39.811	5.72	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.16	4.365	1.33	45.709	5.94	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.24	5.012	1.45	52.481	5.96	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.33	5.754	1.59	60.256	5.69	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.42	6.607	1.76	69.183	5.13	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.52	7.596	1.95	79.433	4.29	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.61	8.710	2.17	91.201	3.27	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	0.70	10.000	2.40	104.713	2.21	1096.478	0.00		
0.105	0.00	1.096		11.482		120.226		1258.925	0.00		

Operator notes:

MASTERSIZER

Result Analysis Report

Sample Name:
Granito - Pombal - Average

Sample Source & type:
Supplier = Adrian Grasson USP

Sample bulk lot ref:

SOP Name:

Measured by:
Laura

Result Source:
Averaged

Measured:
terça-feira, 22 de janeiro de 2019 11:47:43

Analysed:
terça-feira, 22 de janeiro de 2019 11:47:44

Particle Name: Fraunhofer	Accessory Name: Hydro 2000MU (A)	Analysis model: General purpose	Sensitivity: Normal
Particle RI: 0.000	Absorption: 0	Size range: 0.020 to 2000.0... um	Obscuration: 15.20 %
Dispersant Name: Water	Dispersant RI: 1.330	Weighted Residual: 1.000 %	Result Emulation: Off

Concentration: 0.0309 %Vol	Span : 2.165	Uniformity: 0.67	Result units: Volume
Specific Surface Area: 0.454 m ² /g	Surface Weighted Mean D[3,2]: 13.207 um	Vol. Weighted Mean D[4,3]: 45.835 um	

d(0.1): 6.774 um d(0.5): 40.149 um d(0.9): 93.702 um

Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %
0.010	0.00	0.105	0.00	1.096	0.37	11.482	2.22	120.226	2.23	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.40	13.183	2.50	138.038	0.74	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.44	15.136	2.81	156.489	0.00	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	0.47	17.378	3.15	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	0.51	19.953	3.53	208.930	0.00	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.56	22.909	3.98	239.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.61	26.303	4.49	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.66	30.200	5.07	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.73	34.674	5.68	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.80	39.811	6.27	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.89	45.709	6.75	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.06	5.012	1.00	52.481	7.02	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.12	5.754	1.13	60.206	6.98	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.18	6.607	1.29	69.183	5.77	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.23	7.586	1.48	79.433	4.69	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.28	8.710	1.70	91.201	3.35	954.993	0.00	10000.000	0.00
0.091	0.00	0.955	0.33	10.000	1.95	104.713		1096.478	0.00		
0.105	0.00	1.096		11.482		120.226		1258.925			

Operator notes:

MASTERSIZER

Result Analysis Report

Sample Name:
Granito - Jambeiro - Average
Sample Source & type:
Supplier = Adrian Grasson USP
Sample bulk lot ref:

SOP Name:
Measured by:
Laura
Result Source:
Averaged

Measured:
terça-feira, 22 de janeiro de 2019 13:38:41
Analysed:
terça-feira, 22 de janeiro de 2019 13:38:42

Particle Name:
Fraunhofer
Particle RI:
0.000
Dispersant Name:
Water

Accessory Name:
Hydro 2000MU (A)
Absorption:
0
Dispersant RI:
1.330

Analysis model:
General purpose
Size range:
0.020 to 2000.0... um
Weighted Residual:
1.140 %

Sensitivity:
Normal
Obscuration:
19.10 %
Result Emulation:
Off

Concentration:
0.0384 %Vol

Span :
2.131

Uniformity:
0.655

Result units:
Volume

Specific Surface Area:
0.47 m²/g

Surface Weighted Mean D[3,2]:
12.753 um

Vol. Weighted Mean D[4,3]:
48.618 um

d(0.1): 6.371 um

d(0.5): 43.276 um

d(0.9): 98.594 um

Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %	Size (µm)	Volume in %
0.010	0.00	0.105	0.00	1.006	0.43	11.482	1.89	120.226	2.62	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.47	13.183	2.13	138.038	1.25	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.51	15.136	2.41	158.489	0.15	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	0.54	17.378	2.73	181.970	0.00	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	0.57	19.959	3.12	208.930	0.00	2187.782	0.00
0.020	0.00	0.209	0.00	2.188	0.61	22.909	3.60	236.883	0.00	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.65	26.303	4.16	275.423	0.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.69	30.200	4.82	316.228	0.00	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.74	34.674	5.53	363.078	0.00	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.80	39.811	6.22	416.869	0.00	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.87	45.709	6.82	478.630	0.00	5011.872	0.00
0.046	0.00	0.479	0.07	5.012	0.96	52.481	7.20	549.541	0.00	5754.399	0.00
0.052	0.00	0.550	0.13	5.754	1.05	60.296	7.26	630.957	0.00	6606.934	0.00
0.060	0.00	0.631	0.21	6.607	1.17	69.183	6.93	724.436	0.00	7585.776	0.00
0.069	0.00	0.724	0.27	7.596	1.31	79.439	6.20	831.764	0.00	8709.636	0.00
0.079	0.00	0.832	0.33	8.710	1.48	91.201	5.15	954.969	0.00	10000.000	0.00
0.091	0.00	0.955	0.38	10.000	1.67	104.713	3.88	1096.478	0.00		
0.105	0.00	1.096	0.38	11.482		120.226		1258.925	0.00		

Operator notes:

APPENDIX B – Example of the production of the Eq. (3.4)

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T																
1	Cálculo dos coeficientes para a fórmula de Superfície Específica																																			
2																																				
3	Considerações para o cálculo																																			
4	Centro da Faixa "C" de DNIT																																			
5	(mm)	% passante		Intervalo 19,1		12,5		Intervalo 9,5		4,75		Intervalo 2		Intervalo 0,75		Intervalo 0,234		Intervalo 0,075																		
6	19	100		Diam. Máx. Nom.(cm)		1,58		Diam. Máx. Nom.(cm)		0,912		Diam. Máx. Nom.(cm)		0,411		Diam. Máx. Nom.(cm)		0,03375																		
7	12,5	85,1		Lado (cm)		0,912		Lado (cm)		0,411		Lado (cm)		0,00375		Lado (cm)		0,00375																		
8	9,5	78,3		Área (cm²)		4,99		Área (cm²)		1,02		Área (cm²)		0,000325		Área (cm²)		0,0000281																		
9	4,75	53,1		Volume (cm³)		0,759		Volume (cm³)		0,070		Volume (cm³)		0,0000040		Volume (cm³)		0,000000101																		
10	2	37,9		Massa (g)		2,016		Massa (g)		0,185		Massa (g)		0,00000106		Massa (g)		0,0000000270																		
11	0,42	22,5		Y (m³/kg)		0,248		Z (m³/kg)		0,549		U (m³/kg)		104,340																						
12	0,18	14,6		Intervalo 2		0,42		Intervalo 0,18		0,075		Intervalo 0,075		Intervalo 0,075		Intervalo 0,075		Intervalo 0,075																		
13	0,075	8,6		Diam. Máx. Nom.(cm)		0,121		Diam. Máx. Nom.(cm)		0,03		Diam. Máx. Nom.(cm)		0,01275		Diam. Máx. Nom.(cm)		0,00375																		
14	Densidade real do agregado miúdo		Lado (cm)		0,0699		Lado (cm)		0,0173		Lado (cm)		Lado (cm)		0,0074		Lado (cm)		0,0022																	
15	2,656	g/cm³		Área (cm²)		0,0293		Área (cm²)		0,00180		Área (cm²)		0,000325		Área (cm²)		0,0000281																		
16				Volume (cm³)		0,000341		Volume (cm³)		0,0000052		Volume (cm³)		0,00000040		Volume (cm³)		0,000000101																		
17	Considerando as partículas como CUBO		Massa (g)		0,000906		Massa (g)		0,0000138		Massa (g)		Massa (g)		0,00000106		Massa (g)		0,0000000270																	
18	$a = d / \sqrt{12}$		T (m³/kg)		13,043		T (m³/kg)		13,043		T (m³/kg)		T (m³/kg)		30,688		T (m³/kg)		104,340																	
19	$A = 6 \times a^2$																																			
20	$V = a^3$																																			
21																																				
22																																				
23																																				
24	Cálculo da Superfície Específica (m²/kg)																																			
25																																				
26	CURVA PARA MAE (Centro da Faixa "C" de DNIT)																																			
27	(mm)	% passante		Intervalo 1		0,248		Intervalo 0,356		Intervalo 0,549		Intervalo 0,75		Intervalo 1,159		Intervalo 1,159		Intervalo 1,159																		
28	2	100,0		Diam. Máx. Nom.(cm)		13,043		Diam. Máx. Nom.(cm)		30,688		Diam. Máx. Nom.(cm)		30,688		Diam. Máx. Nom.(cm)		30,688																		
29	0,42	59,4		Lado (cm)		13,043		Lado (cm)		30,688		Lado (cm)		30,688		Lado (cm)		30,688																		
30	0,18	38,5		Área (cm²)		13,043		Área (cm²)		30,688		Área (cm²)		30,688		Área (cm²)		30,688																		
31	0,075	22,7		Volume (cm³)		13,043		Volume (cm³)		30,688		Volume (cm³)		30,688		Volume (cm³)		30,688																		
32				Massa (g)		13,043		Massa (g)		30,688		Massa (g)		30,688		Massa (g)		30,688																		
33				T (m³/kg)		13,043		T (m³/kg)		30,688		T (m³/kg)		30,688		T (m³/kg)		30,688																		
34				S (m³/kg)		13,043		S (m³/kg)		30,688		S (m³/kg)		30,688		S (m³/kg)		30,688																		