

# A new laboratory methodology for optimization of mixture design of asphalt concrete containing reclaimed asphalt pavement material

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**Abstract** The reduction of virgin bitumen added to asphalt mixtures containing Reclaimed Asphalt Pavement (RAP) is based on the typical assumption that all the aged binder function in the same way as the virgin binder. However, recent studies conducted by the authors for a specific case show that a blend or mobilization of RAP binder are negligible. The aged bitumen becomes softer acting as glue facilitating cluster formation between small-size RAP particles. The reduction of small-size particles causes changes in the target grading curve and in the voids-fill, affecting the compactability of RAP mixtures. Therefore the target grading curve of RAP mixtures needs to be readjusted, using different proportions of virgin aggregates and taking into account the cluster phenomenon. The objective of this paper is to develop a new mix design approach for RAP mixtures, taking into account

the cluster phenomenon and the contribution of the aged bitumen in the compactability. The virgin aggregates, filler and RAP are investigated and individually included in the calculation. 3D images of the virgin aggregates allowed the determination of new surface area factors; the concept of critical filler concentration led to the definition of the minimum bitumen quantity required to maintain the mastic in a diluted state and fill the voids. A RAP clustering model was introduced to predict the agglomeration of small-size RAP particles. The readjustment of the target grading curve was analytically calculated, allowing the correct estimation of the amount of virgin bitumen to be added to asphalt mixtures. Finally, a first verification of the entire process was carried out performing laboratory tests. These promising results enable the challenge of a new mix design optimization for HMA with high RAP content to be addressed.

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## 1 Background

Despite the massive use of RAP in practice, the chemo-physical phenomena in a new mixture with high RAP content have not yet been sufficiently explored. The characterization and understanding of these phenomena is fundamental for improving the

recycling technology because they determine the mixture characteristics. Since they may significantly affect the mechanical behaviour and performance of RAP mixtures they have to be considered to achieve a reliable mix design calculation.

### 1.1 The key to the mix design: bitumen film thickness and mineral aggregate specific surface area (SSA)

The estimation of the optimal bitumen quantity in Hot Mix Asphalt (HMA) has always been a challenge since it is based on the thickness of the bitumen film on the mineral aggregate surface as well as on other volumetric characteristics (VMA) [1, 2]. The complexity of the determination of these properties is related to the fact that they have to be estimated and are not measured [3]. For instance, all the equations for the bitumen film calculation [4–7], MDT [8] assume the use of specific surface area (SSA) that in turn depends on the surface area factors. The origin and subsequent development of the calculation of the surface area factors currently used are vague in the literature. Hveem [9] first proposed surface area factors that were subsequently adopted by the Asphalt Institute. This first method assumes that the particles are spheres with smooth surfaces [10]. Duriez and Arrambide [11] proposed the following equation to define the SSA ( $\text{m}^2/\text{kg}$ ):

$$\text{SSA} = 0.25g + 2.30c + 12s + 135f \quad (1)$$

where  $g$  is the percentage of gravel (grains above 5-mm sieve) [%],  $c$  is the percentage of coarse sand (grains between 0.315 and 5-mm sieves) [%],  $s$  is the percentage of fine sand (grains between 0.080 and 0.315-mm sieves) [%] and  $f$  is the percentage of filler (grains below 80 microns) [%].

Nevertheless, these coefficients were defined based on the approximation of considering the aggregates as cubes and without any distinction regarding the mineral aggregates and filler mineralogy and properties. Moreover, the fractions were grouped and considered with the same surface area factors (for example  $g$  groups all the grains bigger than 5 mm) and the same density is always assumed. Craus and Ishai [12] later introduced other surface area factors but still considering the aggregates as spheres or cubes. Chapuis and Légaré [13] extended the grading investigation to fine particles smaller than 80 microns for

distinguishing the contributions of different types of fillers in terms of shapes and sizes. However, this filler distinction was not integrated in mix design calculations. Indeed, there is a missing link between the measurements of the surface of the filler and its use in the mix design calculation. This step will be thoroughly discussed later in the paper.

The need for further studies regarding the SSA of aggregates and fillers and the contribution of these last to bitumen content calculations should be combined with efforts to define the optimal bitumen film thickness required to achieve high performance levels of asphalt mixtures. In fact, also in this case the literature is controversial involving approximations, such as assuming all aggregates (small and large particles) being uniformly and equally coated. Hence, it is possible to find several optimal values of thicknesses between 6 and 10.5 microns [5, 14, 15]. The definition of the optimal thickness should be carefully considered as poor coverage can cause lack of bonding between aggregate particles in the mixture allowing water to penetrate more easily and cause moisture damage [15]. On the other hand too thick film may result in rutting distress.

### 1.2 Mix design of asphalt mixtures containing RAP

When using RAP, additional aspects to the general mix design of Hot Mixture Asphalt (HMA) should be taken into account. The Asphalt Institute [16] proposed a formula for the quantity of virgin binder to be added for recycled mixtures, expressed as a percentage by weight of the total mixture:

$$p_{vb} = \frac{(100^2 - p_{na} \cdot p_{rb})p_{mb}}{100(100 - p_{rb})} - \frac{(100 - p_{na})p_{rb}}{100 - p_{rb}} \quad (2)$$

where  $p_{vb}$  is the percentage by weight of total mixture of virgin binder to be added [%],  $p_{mb}$  is the percentage by weight of total mixture of binder required for type of mixture [%],  $p_{na}$  is the percentage of new aggregates in mixture [%] and  $p_{rb}$  is the percentage by weight of binder contributed by RAP material [%].

The formula suggested by the Asphalt Institute does not consider other parameters related to the chemical and physical phenomena occurring during the fabrication of a new mixture with RAP. In fact, several additional elements should be considered such as the



aggregate and binder blend [17–25], the effect of the rejuvenator [26] and the variability of RAP materials.

One of the main issues is certainly the interaction between virgin and aged binder and how the latter functions in a new mixture in terms of workability, compactability and in service performance. Recent studies conducted for specific RAP mixtures by the authors led to the hypothesis that the blending between old and virgin binders is marginal. No actual migration of aged binder was observed whereas when the latter is heated it becomes softer (reactivated binder) acting as glue and causing RAP-cluster formation. Since the old binder acts as glue one can think that this is the normal binder activity. The problem is that the old binder does not migrate or blend homogeneously with the virgin bitumen, but it becomes softer acting as glue excluding the presence, in the contact points among RAP individual particles or clusters, of the virgin bitumen that has characteristics completely different from the aged binder. The results could be the non-homogenous characteristics of the binder gluing the aggregates and the heterogeneity in the asphalt mixture composition. The presence of clusters was detected using the Environmental Scanning Electron Microscope [27] and the mixing temperature was confirmed to be a crucial factor for the clusters formation. The results obtained showed how at high temperatures (180 °C) the RAP particles do not form evident clusters and each RAP particle tends to be covered by a first layer of old RAP binder and a second layer of virgin bitumen. The RAP clusters have been classified, in particular two types of agglomerations can be identified: “old clusters” that refer to RAP particles agglomeration already present before the mixing phase of the new mixture; “new clusters” that refer to the clusters of particles that are created during the new mixing phase. Clusters, old or new, may have important consequences on the RAP mixture behaviour that should be taken into account for the mix design procedure. They could prevent a uniform distribution of the virgin binder and uniform coating of the aggregates, increasing the heterogeneity of the mixture. In addition, the new RAP clusters lead to a reduction of small-size particles in the mixture, causing changes in the design grading curve. This may affect the voids and quantity of virgin bitumen required for adequate coating of the grains [28]. Moreover, RAP has an impact on the compaction and

workability of asphalt mixtures [29] and therefore the volumetric properties of RAP mixtures have to be considered for a correct mix design.

## 2 Objectives and research stages

The objective of the present work is to propose a new methodology for estimating the dosage of virgin bitumen required in RAP mixtures for Asphalt Concrete (AC). In the first step, new surface area factors were calculated based on 3D images of virgin aggregates captured with a laser scanner. In the second step filler's properties and contribution to SSA calculation were investigated. Filler was considered separately because its behaviour and role in the mixtures are different than the ones of the coarse aggregates. Indeed, whereas the aggregates represent the “lytic skeleton” to be covered, the filler/bitumen mastic is in a diluted phase and has to fill the interstitial voids [30]. In the third step a cluster prediction model was introduced in the calculation that defines the final bitumen content. Indeed, the surface of RAP to be covered by bitumen was calculated separately from the virgin aggregates in order to account for the reduction of small-size particles caused by clustering. These three steps allow the separate investigation of the contribution of virgin aggregates, filler and RAP as well as the determination of the quantity of the virgin bitumen required by each single component.

Once the overall equation for the mix design calculation was defined (fourth step), a laboratory verification was conducted. Marshall tests, gyratory compactor and indirect tensile strength test (ITST) were performed in order to verify that the optimal binder content calculated with the proposed methodology represents a valid estimation of the measured one.

Finally, in the fifth step, the compaction and workability for high RAP content were analysed, since it was essential to confirm whether the estimated bitumen content provided by the methodology met the volumetric requirements (compactability and air voids). A specific analysis was conducted to understand the impact of the aged reactivated binder and clusters in the compaction of RAP mixtures.

In the framework of this study no rejuvenator has been used.

## 2.1 Materials

Mineral aggregates and limestone filler ( $d < 0.063$  mm) from a Swiss quarry in Choëx/Massongex, RAP 0/16 from Granges-de-Vesin (Switzerland) and unmodified virgin bitumen 70/100 were used for this study. The main characteristics of the materials are summarised in Table 1.

The virgin aggregates were washed and dried and all the fine particles ( $d < 0.063$ ) were replaced with the limestone filler.

## 3 Methodology

### 3.1 New surface area factors

The first stage of the study concerns the calculation of new surface area factors for the virgin aggregates

without having to consider their shapes as spheres or cubes. The 3D images of twenty aggregates of the same grading class (21.4/31.5 mm) were captured with the FAROARM PLATINUM with laser scan head LINE PROBE III (measurement accuracy 0.03 mm). The resulting images were treated with Meshlab version 1.3.3 applying 50,000 meshes and they provided the measurements of the actual surface and volume of the grains. An “average grain” was defined as the aggregate characterised by the surface and volume calculated as the average of the twenty samples scanned. Subsequently all the samples were scaled into different fractions using the Meshlab software. The detailed procedure for obtaining the scale factors is reported elsewhere [31]. Once the characteristics of the “average grain” for every fraction were obtained, the following equations were used [31]:

$$V_{il \text{ kg}} = \frac{1}{\rho_a} \quad (3)$$

**Table 1** Summary of mineral aggregate, filler and binder characteristics

Property	Test method	Value
Aggregate characteristics (Famsa)		
Density (kg/m <sup>3</sup> )	EN 1097-6	2741
Polished stone value	DIN EN 1097-8	60–62
Los Angeles	SN EN 1097-2	11–15
Affinity with bituminous binders	SN 670 460	$U = 85.1$ % (Adequate adherence of bitumen on aggregates is ensured for the majority of uses)
Phyllosilicate content	SN 670 115	From 1.9 to 4.9% in mass (admissible)
Filler characteristics (limestone)		
Density (kg/m <sup>3</sup> )	EN 1097-7	2705
Rigden voids (%)	EN 1097-4	32
Methylene blue (clay content)	EN 933-9 (C837-09)	4.00
Property	RAP binder Value	Virgin bitumen Value
Extraction and recovery (%) EN 12697-1 and 3	5.60	–
Penetration grade @25°C [10 <sup>-1</sup> mm] SN EN 1426	26	82
Softening points R&B (°C) SN EN 1427	64.6	47.8
Penetration index (PI) (–) SN EN 12591	0.4	–0.6
SARA fractions (Iatroscan)		
Saturates (%)	5.18	6.69
Aromatics (%)	10.51	24.64
Resins (%)	57.69	41.23
Asphaltenes (%)	26.63	27.54

$$N_{i1 \text{ kg}} = \frac{V_{i1 \text{ kg}}}{v_i} \quad (4)$$

$$\alpha_i = N_{i1 \text{ kg}} \cdot s_i \quad (5)$$

where  $\rho_a$  is the density of aggregates (same for all) ( $\text{kg}/\text{m}^3$ ),  $V_{i1 \text{ kg}}$  is the volume of 1 kg of aggregates of  $i$ -fraction ( $\text{m}^3/\text{kg}$ ),  $v_i$  is the volume of “average” grain of the  $i$ -fraction ( $\text{m}^3$ ),  $N_{i1 \text{ kg}}$  is the number of “average” grains in every kg of aggregates ( $\text{kg}^{-1}$ ),  $s_i$  is the surface area of the “average” grain of the  $i$ -fraction ( $\text{m}^2$ ) and  $\alpha_i$  is the surface area factor of aggregates for the  $i$ -fraction ( $\text{m}^2/\text{kg}$ ).

Subsequently, multiplying the surface area factors ( $\alpha_i$ ) by the percentage in mass of the aggregates in the grading curve and summing all the contributions allows the calculation of the surface of virgin aggregates ( $\text{SSA}_{\text{agg}}$ ) ( $\text{m}^2/\text{kg}$ ) to be covered by the virgin bitumen.

$$\text{SSA}_{\text{agg}} = \sum \alpha_i \cdot (p_{i+1} - p_i) \quad (6)$$

where  $(p_{i+1} - p_i)$  is the percentage by weight of aggregates smaller than size  $i + 1$  and larger than size  $i$ ;  $\alpha_i$  are the new surface area factors determined with the above procedure. Thus, the volume of bitumen required to cover all the aggregates is defined as:

$$V_{\text{bit-agg}} = \bar{t}_{\text{opt-agg}} \cdot \left\{ \sum \alpha_i \cdot (p_{i+1} - p_i) \right\} \quad (7)$$

where  $\bar{t}_{\text{opt-agg}}$  is the optimal average thickness of virgin bitumen around the aggregates selected from literature as being equal to 10 microns to minimize the effect of ageing [5].

### 3.2 Filler

The physical characteristics of the limestone were determined by carrying out several tests:

- Particle Size Distribution (PSD) with laser diffraction. This test provides several measurements: the sieve sizes at which 10, 50 and 90 % of the filler sample passes ( $d_{[v,0.1]}$ ,  $d_{[v,0.5]}$ ,  $d_{[v,0.9]}$ ) and the average diameter ( $d_{[4,3]}$ ). The PSD measurements also provide a value of the specific surface area ( $\text{SSA}_{\text{PSD}}$ ) based on the hypothesis that all the particles are spherical.
- Brunauer, Emmett and Teller (BET) measurements for determining the agglomeration factor

( $F_{\text{ag}}$ ). The BET procedure was carried out with a Micromeritics Gemini 2360.

The agglomeration factor ( $F_{\text{ag}}$ ) is an indication of the degree of filler agglomeration and it allows the comparison between powders and treatments.  $F_{\text{ag}}$  is calculated with the following equations:

$$d = \frac{6}{\text{SSA}_{\text{BET}} \cdot \rho_f} \quad (8)$$

$$F_{\text{ag}} = \frac{d_{[v,0.5]}}{d_{\text{BET}}} \quad (9)$$

where  $\text{SSA}_{\text{BET}}$  is the specific surface area measured by nitrogen absorption (BET method) [ $\text{m}^2/\text{g}$ ],  $\rho_f$  is the filler density [ $\text{g}/\text{cm}^3$ ],  $d_{\text{BET}}$  is the average diameter calculated with BET [ $\mu\text{m}$ ] and  $d_{[v,0.5]}$  is the median diameter found with PSD [ $\mu\text{m}$ ].

- Environmental scanning electron microscope (ESEM). The images captured with this technique helps to visualise the shape of the filler particles and how they differ from spheres. Moreover, it is possible to verify whether the particles are clearly detectable, or if they are agglomerated.

The information obtained from these tests raised the question of how to consider the filler surface for calculating the bitumen content. Indeed, the BET method provides the specific surface area ( $\text{SSA}_{\text{BET}}$ ) of the completely dispersed powder whereas in reality, when filler is used in asphalt mixtures is not completely dispersed in the bitumen. Hence, the actual surface to be covered with bitumen does not correspond to that measured with the PSD and BET instruments. The agglomeration factor is an indicator for the tendency of the filler to create clumps. However, further studies are needed to understand how this factor can be used to obtain a suitable surface for mix design calculations.

In view of these unsolved problems the study was concentrated on the filler-bitumen mastic which has the role of filling the interstitial voids, thus regulating the stiffness of the asphalt mixtures [32]. It is well known that the mastic stiffness varies depending on the filler mineralogy and concentration. Recently, the impact of filler concentration on mastic stiffness was thoroughly investigated by Faheem and Bahia [33] who proposed a conceptual model for the introduction of filler with the binder.

The model identifies two states: diluted and concentrated. In the diluted region the filler particles

are separated by free bitumen volume. The bridge of bitumen between the particles is sufficiently thick to ensure almost constant stiffness. When the amount of filler increases above a certain threshold, the mastic enters the concentrated state, where the consumption of the free bitumen causes the sudden increase of stiffness.

The *critical filler concentration* ( $\phi_c$ ) represents the transition between the diluted and the concentrated states and, as suggested by Faheem and Bahia, it represents the maximum allowable limit of filler in the mastic. The *critical filler concentration* is obtained with the following equation [34]:

$$\phi_c = 83.2 \text{ RV}(\%) + 4.79 \text{ MBV} \quad (10)$$

where RV(%) is the Rigden voids, MBV is the Methylene Blue Value (clay content) (–) and  $\phi_c$  is the Critical filler concentration (% by volume).

The *critical filler concentration* was used to calculate the minimum bitumen quantity necessary to maintain the mastic in a diluted phase (Eq. 12). This allows the mastic to maintain the required stiffness and suitable consistency in the bituminous mixtures.

$$V_{\text{filler}} = \frac{W_{\text{filler}}}{\rho_f} \quad (11)$$

$$V_{\text{bit-filler}} = \frac{V_{\text{filler}} \cdot \left(1 - \frac{\phi_c}{100}\right)}{\frac{\phi_c}{100}} \quad (12)$$

where  $V_{\text{filler}}$  is the filler volume ( $\text{m}^3/\text{kg}$  of mixture),  $W_{\text{filler}}$  is the filler weight ( $\text{kg}/\text{kg}$  of mixture),  $\rho_f$  is the filler density ( $\text{kg}/\text{m}^3$ ),  $\phi_c$  is the critical filler concentration (% by volume) and  $V_{\text{bit-filler}}$  is the minimum volume of bitumen required to maintain mastic in diluted phase ( $\text{m}^3/\text{kg}$  of mixture).

### 3.3 Modelling of RAP clustering

As mentioned above, the old re-activated binder trapped in the RAP material becomes softer during the re-heating, acting as glue and facilitating cluster formation between the small-size RAP particles. This clustering has been discussed in detail by the authors [28, 35], considering old and new RAP clusters. In order to readjust the target grading curve taking into account the formation of new RAP clusters during a new mix phase a clustering prediction model was

developed considering only the new clusters and involving two variables: the RAP fraction size and RAP percentage.

A particular procedure was carried out to determine the amount of new clusters forming during a new mixing phase considering different fractions and percentages of RAP. The virgin aggregates and RAP material were sieved separately for the following fractions: 0.063/0.5, 0.5/1, 1/2, 2/4, 4/8, 8/16 mm.

The estimation of the amount of clusters was conducted using two fractions at a time, for each couple of fractions the same steps were applied. Hereafter an example of the procedure is reported (50 % of 0.063/0.5 mm RAP and 50 % of 0.5/1-mm virgin aggregates).

- 200 g of 0.5/1-mm virgin aggregate fraction was heated at 200 °C for one hour.
- 200 g of 0.063/0.5-mm RAP fraction was heated at 135 °C for one hour. It is important to heat the RAP as little as possible as the heating ages the already aged binder further [36].
- The two fractions were mixed together for two minutes by hand and left in the oven for one hour at 160 °C to bring the mixture to the selected fabrication temperature. Note that the cluster phenomenon could be affected by the fabrication temperature, the mixing energy and time. The fabrication temperature was kept constant for every mixture preparation. In this way, the probable effect of different type of heating might have on the thickness and state of the RAP bitumen was avoided.
- Once the mix was removed from the oven and cooled, it was sieved again with the 0.5-mm sieve. Part of the RAP was retained by the sieve (0.5 mm) because the RAP activated binder within the RAP was adhering to other RAP particles or virgin aggregates, creating new clusters that could not pass through the sieve. This is also why the virgin aggregate fraction just above that of the RAP was chosen. Indeed as soon as the RAP particles started to stick together they did not pass through the sieve and were retained in the upper fraction.
- The RAP material passing through the sieve (0.5 mm) was weighted after the procedure and the difference in mass between the initial passing RAP material was reported as a value of the percentage of new clusters for the fraction.

The procedure was carried out using several RAP percentages and RAP fractions. Once the values of cluster percentages in mass for every combination had been obtained a multiple regression analysis was conducted. A second-degree model with interaction terms was applied (Eq. 13). The variables have been standardized to bring them on a common scale.

$$C'_i = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j} \sum \beta_{ij} x_i x_j + \varepsilon \quad (13)$$

where  $C'_i$  is the percentage in mass of RAP new clusters of weight of RAP-i-fraction [%],  $k$  is the number of variables,  $\beta_0$  is the constant term,  $\beta_i$  and  $\beta_{ii}$  are the coefficients of first- and second-degree terms respectively,  $x_i$  and  $x_i^2$  are the first- and second-degree terms respectively,  $\beta_{ij}$  is the coefficients of interaction terms,  $x_i x_j$  is the interaction terms and  $\varepsilon$  is the unknown error.

The clustering model was introduced in the mix design calculation to readjust the proportions of every fraction of virgin aggregate in order to maintain the same target grading curve after the mixing process.

### 3.4 Verification in the laboratory

Once all the contributions (virgin aggregates, filler and RAP) were investigated, a formula was developed for estimating the optimal bitumen amount to be added to the RAP mixtures.

Subsequently, verification was undertaken on 50 % RAP laboratory mixtures with 4.5, 5, 5.5 and 6 % of 70/100 unmodified virgin bitumen by weight of the total mixture. The mix design method was applied to a Swiss standard binder course AC B 16 (i.e. asphalt concrete with maximum nominal size 16 mm). The target grading curve is reported in Table 6.

The following tests were performed to verify that the optimal bitumen amount calculated with the proposed formula really represents a valid estimation of the measured one are:

- Marshall tests
- Gyratory compaction test
- Indirect Tensile Strength Test (ITST) at 15 °C.

Marshall tests were conducted on cylindrical samples with a diameter of 102 mm and a height of 64 mm. In order to compact the samples fifty blows

were given on each side of the specimen. The testing load applied increases until it reaches a maximum, and then begins to decrease. The maximum load is recorded just before it starts to decrease.

The optimum asphalt binder content is finally selected based on the combined results of Marshall stability and flow as well as air voids content. The minimum values required by the Swiss standard S 640 431-1b-Na for the Marshall stability and the flow and air voids are respectively 7.5 kN, 2–4 mm and 3–6 %.

The gyratory compactor has in many cases replaced the Marshall hammer compaction. The following conditions for the gyratory compactor were set:

- Sample size: 150-mm-diameter cylinder approximately 115 mm in target height
- Compaction pressure: 600 kPa
- Speed of rotation: 30 gyrations/min
- Inclination from the vertical axes: 1.25°
- Compaction temperature: 135 °C

The Superpave gyratory compactor establishes three different gyration numbers:  $N_{\text{initial}}$ ,  $N_{\text{design}}$  and  $N_{\text{max}}$ .  $N_{\text{design}}$  is the parameter considered in this study. It is the standardized number of gyrations used in design in accordance with the indicated amount of traffic. Typically  $N_{\text{design}} = 100$  for 20-year traffic loading higher than 3 million of equivalent single axle loads (ESALs) equal to 8.16 kN.

For the ITST, specimens with the same dimensions as for the Marshall tests were used. The deformation rate during loading was 0.85 mm/s. From this test it was possible to determine the tensile strength  $\sigma_t$  (Eq. 14).

$$\sigma_t = \frac{2P}{\pi Dh} \quad (14)$$

where  $\sigma_t$  is the tensile strength (MPa),  $P$  is the load [kN],  $D$  is the specimen diameter (mm) and  $h$  is the specimen height (mm).

## 4 Results and specific application

### 4.1 Virgin aggregates

Twenty 3D images of aggregates were captured with the laser scan and analysed with Meshlab 1.3.3. 50,000 meshes were applied and the boxes containing the aggregates were drawn in order to scale the grains



correctly. Examples of the images and their digital representations are shown in Fig. 1.

After having scaled all the samples in every grading class, an “average grain” was obtained for every fraction. The “average grain” is the aggregate characterised by the average surface and volume of all the samples in the same grading class. Thus, using Eqs. 3, 4, 5 and 6 the new surface area factors and the total SSA of aggregates were calculated (Table 2).

Knowing the total SSA of aggregates, it is possible to calculate the bitumen for covering the aggregates with a selected optimal bitumen film thickness of 10 microns according to AC B 16 grading curve (Table 2). This first step excludes the filler contribution included in the next section.

#### 4.2 Filler

The characteristics of the filler (PSD, BET and  $F_{ag}$ ) are summarised in Table 3 and Fig. 2.

From Table 3 and Fig. 2 it can be seen that the filler is composed of different shapes and elongated particles (Fig. 2b). This is one of the reasons why the  $SSA_{PSD}$  and  $SSA_{BET}$  are significantly different. Indeed, PSD measurements are based on the assumed spherical particles. Moreover, the particles are not completely dispersed in the fluid and the filler has a high tendency to agglomerate ( $F_{ag} = 51.79$ ). This is confirmed also by the ESEM image where the particles

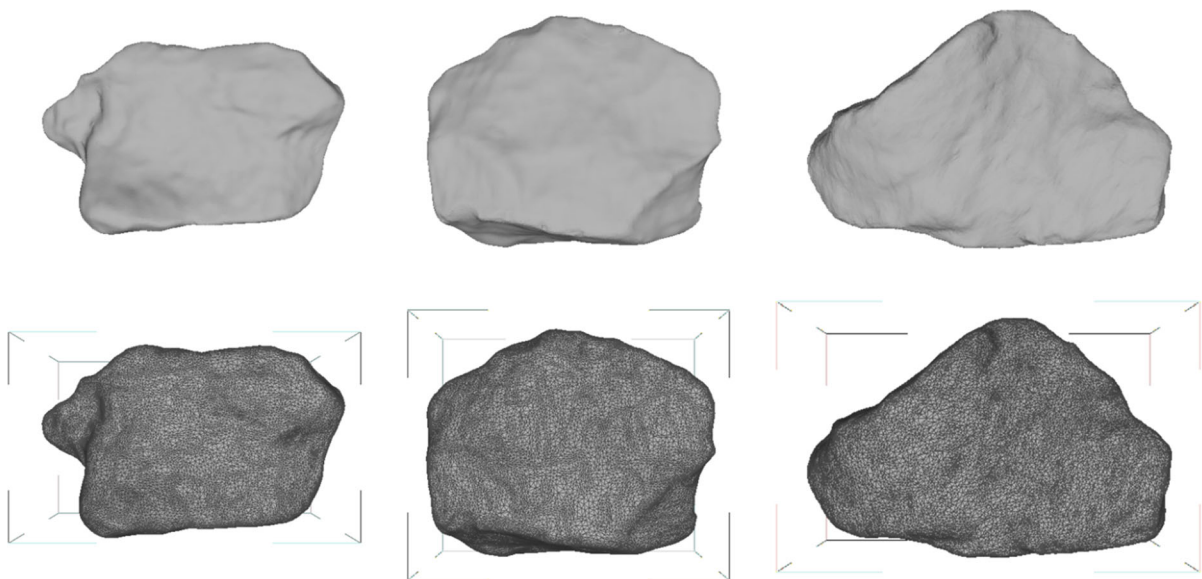
overlap and are not clearly recognisable. On the one hand, the SSA measure of BET is the more reliable because it takes into account the actual shapes of the particles that are completely dispersed. On the other hand, this value cannot be used in the calculation because it does not correspond to the effective SSA of the filler in the mixture due to its tendency to agglomerate.

To overcome this problem in the present study the *critical filler concentration* ( $\phi_c$ ) was used for defining the bitumen required for covering the filler. This represents the maximum allowable limit of filler in the mastic before reaching the cohesive strength state. The critical filler concentration and volume of bitumen necessary for maintaining the mastic in a diluted phase in case of AC B 16 are summarised in Table 4.

#### 4.3 RAP clustering prediction model

The mass of new RAP clusters for different fractions and several percentages of RAP are summarised in Table 5.

All the combinations in Table 5 tested according the procedure explained in Sect. 4.3 allowed the quantification of the new RAP clusters for every RAP fraction and percentage of RAP. Therefore, a second-degree multiple regression analysis and the determination of the coefficients of the model (Eq. 15) were carried out:



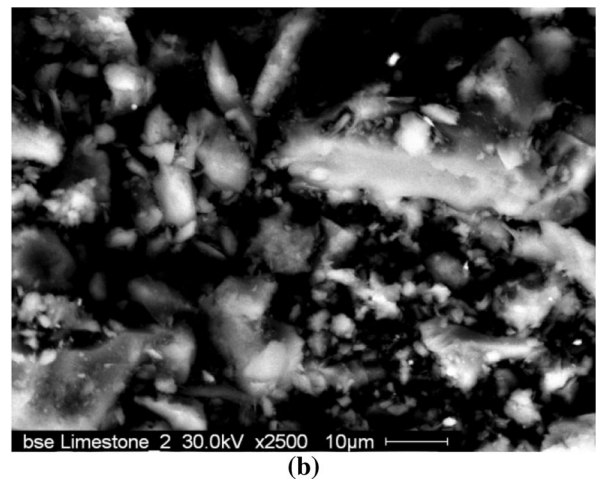
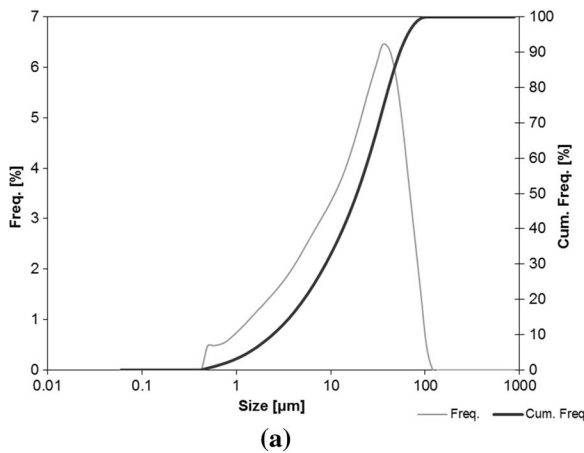
**Fig. 1** Example of 3D images captured with laser scan technology and digital representations of the grains

**Table 2** Calculated values of new surface area factors ( $\alpha_i$ )

Fractions (mm)	$s_i$ (m <sup>2</sup> )	$v_i$ (m <sup>3</sup> ) Equation (3)	$N_{i1}$ kg (kg <sup>-1</sup> ) Equation (4)	$\alpha_i$ (m <sup>2</sup> /kg) Equation (5)	$(p_{i+1} - p_i)$ (%) Equation (6)	SSA <sub>agg</sub> (m <sup>2</sup> /kg) Equation (6)
22.4/31.5	3.56E-03	1.30E-05	28.03	0.0999	—	—
16/22.4	1.98E-03	5.00E-06	72.96	0.1444	4	0.00577
8/16	6.73E-04	9.44E-07	386.24	0.2599	20	0.05199
4/8	2.43E-04	1.64E-07	2223.06	0.5395	25	0.13487
2/4	7.73E-05	2.86E-08	12,735.20	0.9842	18	0.17715
1/2	2.63E-05	5.00E-09	72,955.72	1.9171	10	0.19171
0.5/1	9.28E-06	8.73E-10	417,939.18	3.8776	7	0.27143
0.25/0.5	3.42E-06	1.52E-10	2,394,235.23	8.1934	2	0.16389
0.125/0.25	1.16E-06	2.66E-11	13,715,781	15.8527	4	0.63420
0.063/0.125	3.95E-07	4.74E-12	77,012,279	30.4403	3	0.91321
Total aggregate surface						2.54422
$\bar{r}_{opt-agg}$ [micron] (selected from literature)						10
$V_{bit-agg}$ (m <sup>3</sup> /kg)						2.5442E-05

**Table 3** Summary of filler characteristics

$d_{[v,0.1]}$ (μm)	$d_{[v,0.5]}$ (μm)	$d_{[v,0.9]}$ (μm)	Mean diameter $d_{[4,3]}$ (μm)	SSA <sub>PSD</sub> (m <sup>2</sup> /g)	SSA <sub>BET</sub> (m <sup>2</sup> /g)	$F_{ag}$
2.44	18.93	52.81	25.42	0.3526	6.0676	51.79

**Fig. 2** a PSD filler size distribution and b ESEM image of limestone filler**Table 4** Calculated value of bitumen volume necessary to maintain mastic in diluted phase

Percentage of passing material in target grading curve (%)	$\phi_c$ (%) by volume)	Filler density $\rho_f$ (kg/m <sup>3</sup> )	$V_{filler}$ (m <sup>3</sup> /kg of mixture)	Equation (11) $V_{bit-filler} = \frac{V_{filler} \cdot (1 - \frac{\phi_c}{100})}{\frac{\phi_c}{100}}$ (m <sup>3</sup> /kg of mixture)
7	46	2705	2.5878E-05	3.064E-05

**Table 5** Summary of results of percentage of new clusters in mass for different RAP fractions and percentages of RAP

Fraction (mm)	Percentage of RAP (%)	Percentage of new clusters in mass of the weight of the RAP fraction (%) <sup>a</sup>
0.063/0.5	10	24.55
	30	55.05
	50	64.34
	70	45.35
	90	13.40
0.5/1	10	30.00
	30	58.80
	50	70.20
	70	59.25
	90	32.80
1/2	10	–
	30	39.25
	50	46.00
	70	24.80
	90	31.35
2/4	10	8.05
	30	11.75
	50	12.95
	70	9.14
	90	8.55

<sup>a</sup> Average of at least three repetitions

$$C'_i = 49.96 - 16.94x_1 - 1.47x_2 + 2x_1x_2 - 11.59x_1^2 - 24.08x_2^2 \quad (15)$$

where  $C'_i$  is the percentage in mass of RAP new clusters of weight of the RAP-*i*-fraction (%),  $x_1$  is the standardised size of RAP fraction (0.063/0.5, 0.5/1, 1/2, 2/4 mm) and  $x_2$  is the standardised percentage of RAP (%).

The above model provides the percentage of RAP new clusters in mass for every RAP fraction once the standardised input parameters (type of fraction and percentage of RAP) are established.

The ANOVA conducted to evaluate the goodness of fit of the model presented in Eq. 15 showed  $p$  value  $\ll 0.05$ . In order to include the clustering model in the mix design calculation it is necessary to weight the clustering according to the percentage of RAP material for every fraction:

$$C_i = C'_i \cdot \text{RAP}_{i\_fraction} \quad (16)$$

where  $C_i$  is the percentage of the total mass of new RAP clusters for the *i*-fraction,  $\text{RAP}_{i\_fraction}$  is the mass of the *i*-fraction in the RAP grading curve. The RAP grading curve was then readjusted, taking into account the clustering according to the following equation:

$$\% \text{RAP}_{i\_fraction \text{ readjusted}} = (p_{i+1} - p_i - C_{i+1} + C_i) \quad (17)$$

where  $\% \text{RAP}_{i\_fraction \text{ readjusted}}$  is the percentage of RAP material between *i* and *i* + 1 sieve readjusted after the formation of new RAP clusters,  $p_{i+1}$  is the percentage by weight passing through (*i* + 1)-sieve (%),  $p_i$  is the percentage by weight passing through *i*-sieve (%),  $C_{i+1}$  is the percentage of mass of particles clustered and retained in (*i* + 1)-sieve calculated in Eq. 16 (%) and  $C_i$  is the percentage of mass of particles clustered and retained in *i*-sieve calculated in Eq. 16 (%).

Applying the same surface area factors as those calculated for the virgin aggregates, it is possible to determine the surface of RAP ( $\text{SSA}_{\text{RAP}}$ ) after the new clustering occurred during the mixing process:

$$\text{SSA}_{\text{RAP}} = \sum_i^n \alpha_i (p_{i+1} - p_i - C_{i+1} + C_i) \quad (18)$$

where  $n$  is the number of fractions.

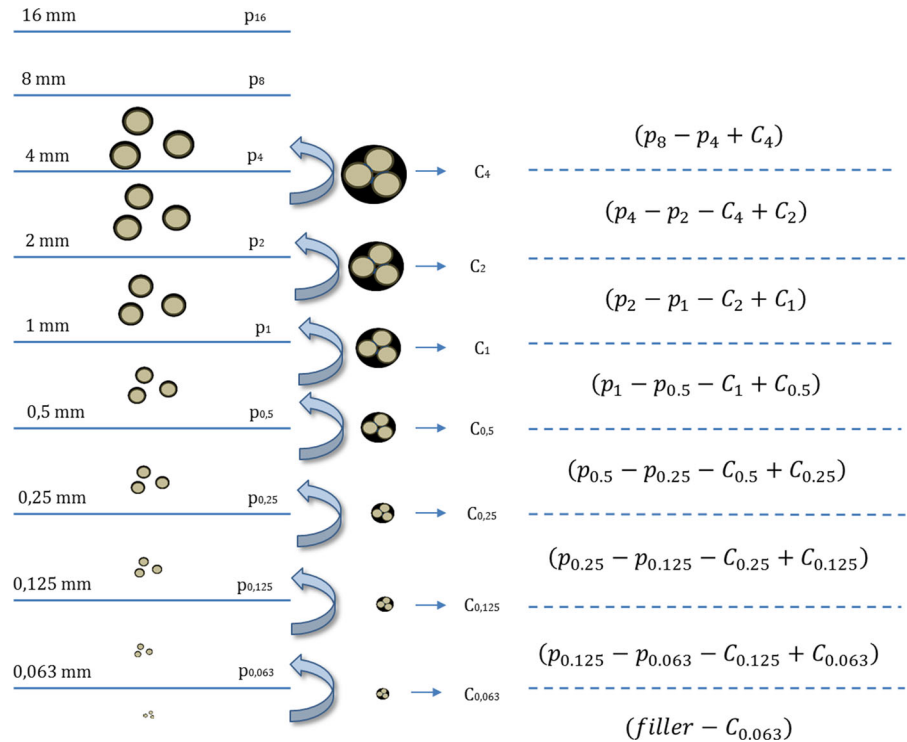
The main concept behind the adjustment (see Fig. 3) of the RAP grading curve (Eq. 17) after the new clustering occurred during the mixing process is the subtraction of the clustered particles that move from the  $\text{RAP}_{i\_fraction}$  to the upper fraction and the addition of the clusters that move into the  $\text{RAP}_{i\_fraction}$  from the lower one. With the readjusted grading curve it is possible to calculate the surface of RAP (Eq. 18). To clarify the conceptual model, a schematic representation is shown in Fig. 3. Note that the cluster phenomenon occurs only among small-size RAP particles [35]. Thus the phenomenon is considered only up to a particle size of 4 mm.

#### 4.4 Final theoretical formulation and laboratory verification

After defining the contributions of all the components (aggregates, filler and RAP) through Eqs. 7, 12 and 18 the bitumen amount to be added to the RAP mixtures can be calculated as follows:



**Fig. 3** Schematic representation of readjustment of RAP grading curve based on cluster phenomenon



$$V_{\text{bit}} = \bar{t}_{\text{opt-agg}} \cdot \left\{ \left[ (1 - \% \text{RAP}) \sum_i^n \alpha_i [(pt_{i+1} - pt_i) - (pr_{i+1} - pr_i - C_{i+1} + C_i)] \right] + \left[ \% \text{RAP} \sum_i^n \alpha_i (pr_{i+1} - pr_i - C_{i+1} + C_i) \right] \right\} + \frac{V_{\text{filler}} \cdot (1 - \frac{\phi_c}{100})}{\frac{\phi_c}{100}} \quad (19)$$

where  $V_{\text{bit}}$  is the estimation of optimal bitumen to add to mixture ( $\text{m}^3/\text{kg}$ ),  $\bar{t}_{\text{opt-agg}}$  is the optimal average thickness of virgin bitumen around aggregates chosen equal to 10 microns (m),  $\% \text{RAP}$  is the RAP percentage in asphalt mixture (-),  $\alpha_i$  is the surface area factor of aggregates passing through  $i + 1$  sieve and retained in  $i$ -sieve ( $\text{m}^2/\text{kg}$ ),  $pt_{i+1}$  is the percentage of material passing through  $(i + 1)$ -sieve in target grading curve of AC B 16 (%),  $pr_{i+1}$  is the percentage of material passing through  $(i + 1)$ -sieve in RAP curve (%),  $pt_i$  is the percentage of material passing through  $i$ -sieve in target grading curve (%),  $pr_i$  is the percentage material passing through  $i$ -sieve in RAP curve (%),  $C_{i+1}$  is the percentage of mass of particles clustered and retained

in  $(i + 1)$ -sieve (%),  $C_i$  is the percentage of mass of particles clustered and retained in  $i$ -sieve (%),  $V_{\text{filler}}$  is the volume of filler ( $\text{m}^3/\text{kg}$ ) and  $\phi_c$  is the critical filler concentration.

$(pt_{i+1} - pt_i) - (pr_{i+1} - pr_i - C_{i+1} + C_i)$  represents the amount of virgin aggregates to add to the mixtures for every fraction in order to readjust the target grading curve after the RAP clustering occurring during a new mix. Thus, Eq. 19 can be written as:

$$V_{\text{bit}} = \bar{t}_{\text{opt-agg}} \cdot \left\{ \left[ (1 - \% \text{RAP}) \sum_i^n \alpha_i (pa_{i+1} - pa_i) \right] + \left[ \% \text{RAP} \sum_i^n \alpha_i (pr_{i+1} - pr_i - C_{i+1} + C_i) \right] \right\} + \frac{V_{\text{filler}} \cdot (1 - \frac{\phi_c}{100})}{\frac{\phi_c}{100}} \quad (20)$$

where  $pa_{i+1}$  is the percentage of virgin aggregates passing through  $(i + 1)$ -sieve to add to mixture (%) and  $pa_i$  is the percentage of virgin aggregates passing through  $i$ -sieve to add to mixture (%).

Thus, if 50 % of RAP is used in AC B 16, the theoretical calculation leads to the results in Table 6.

**Table 6** Summary of calculated values for estimation of virgin bitumen to add to the 50 % RAP mixture AC B 16

Sieve (mm)	RAP grading curve percentage passing (%)	50 % RAP percentage passing (%)	%RAP ( $pr_{i+1} - pr_i$ ) (%)	%RAP( $pr_{i+1} - pr_i - C_{i+1} + C_i$ ) (%)	$\alpha_i$ (m <sup>2</sup> /kg)	RAP surface for every fraction (m <sup>2</sup> /kg)
22.4	100.0	50.0	–	–	–	–
16.0	100.0	50.0	–	–	–	–
8.0	72.8	36.4	13.6	13.6	0.260	0.035
4.0	37.6	18.8	17.6	21.4	0.540	0.116
2.0	16.6	8.3	10.5	12.03	0.984	0.118
1.0	6.7	3.4	5.0	2.4	1.917	0.046
0.500	2.4	1.2	2.2	0.6	3.878	0.023
0.250	0.9	0.5	0.8	0.0	8.195	0.000
0.125	0.3	0.2	0.3	0.04	15.855	0.006
0.063	0.1	0.1	0.1	0.00	30.440	0.000
Sieve (mm)	Target grading curve percentage passing (% by weight)	RAP percentage passing readjusted after cluster (% by weight)	Grading curve of virgin aggregates required to maintain target curve (% by weight)]	(1 – %RAP) ( $pa_{i+1} - pa_i$ ) (% by weight)	$\alpha_i$ (m <sup>2</sup> /kg)	Surface of virgin aggregates for every fraction (m <sup>2</sup> /kg)
RAP total surface (m <sup>2</sup> /kg)						
22.4	100.0		50.0	–	–	–
16.0	96.0	50.00	45.9	4.1	0.144	0.006
8.0	76.0	36.45	39.5	6.4	0.260	0.017
4.0	51.0	15.09	35.9	3.6	0.540	0.019
2.0	33.0	3.05	29.9	6.0	0.984	0.059
1.0	23.0	0.64	22.4	7.6	1.917	0.146
0.500	16.0	0.05	16.0	6.4	3.878	0.248
0.250	14.0	0.05	14.0	2.0	8.1945	0.164
0.125	10.0	0.00	10.0	4.0	15.855	0.634
0.063	7.0	0.00	7.0	3.0	30.440	0.913
Virgin aggregate surface (m <sup>2</sup> /kg)						2.206
Total surface (RAP + virgin aggregates) (m <sup>2</sup> /kg)						2.551
Volume of bitumen to cover virgin aggregates and RAP [m <sup>3</sup> /kg] (with $\bar{t}_{opt} = 10$ micron)						2.551E–05
Volume of bitumen to cover filler (m <sup>3</sup> /kg)						3.043E–05
Total virgin bitumen volume (m <sup>3</sup> /kg)						5.594E–05
Bitumen density (kg/m <sup>3</sup> )						1030
Percentage of virgin bitumen to add to the aggregate + RAP mixture (% by weight of the total mixture)						5.43

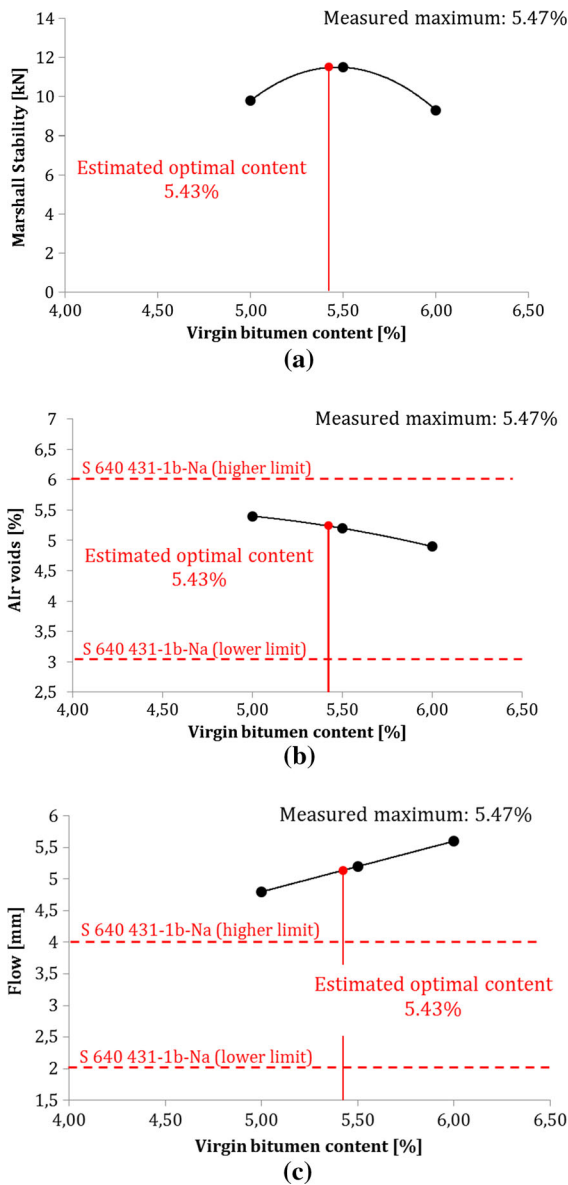
Note that the surface area factors estimated for the calculation of the SSA of virgin aggregates were used also for calculating the SSA of RAP. Even if the source of RAP was controlled and composed of aggregates of the same origin, this represents an approximation for the following reasons:

- RAP aggregates result from different stages: milling, transport and stock. Thus, the RAP is not completely controllable.

- The aged bitumen might change the SSA and an important proportion of aggregates can be rounded at the end of the pavement service life.

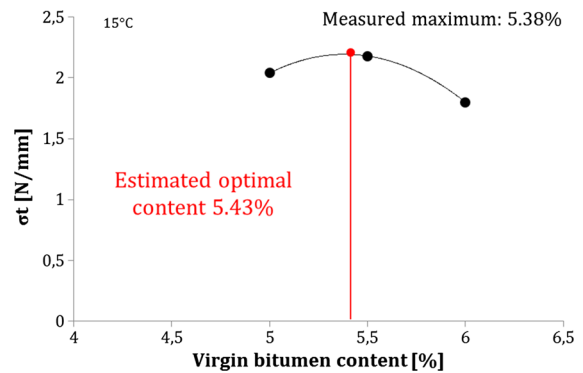
Nevertheless, the authors decided to use the same surface area factors estimated for the virgin aggregates with the above mentioned procedure because it represents a less strong approximation than considering the aggregates as spheres or cubes as in the previous methods [11, 12].





**Fig. 4** Marshall test results for AC B 16 with 50 % of RAP. The bitumen content refers to the virgin bitumen to add to the mixture

The percentage of bitumen calculated in Table 6 represents the estimation of the optimal binder quantity to add to the mixture after readjusting the grading curve due to the RAP cluster phenomenon occurring during a new mix phase. Indeed, Eq. 20 allows manipulating the size and the amount of virgin aggregates to adjust the curve, knowing the change caused by the RAP clustering. Moreover, it would



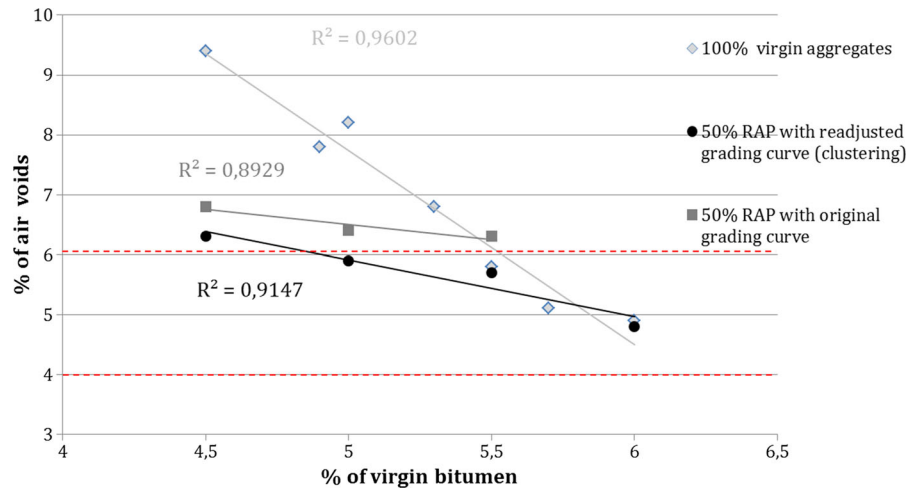
**Fig. 5** ITST results ( $\sigma_I$ ) at 15 °C for AC B 16 with 50 % of RAP. The bitumen content refers to the virgin bitumen to add to the mixture

potentially be possible to use different bitumen film thicknesses for different particle sizes. The density is assumed to be constant for all the aggregates.

To verify the theoretical results obtained, laboratory tests were conducted. The results of Marshall and ITS tests are reported in Figs. 4 and 5.

The Marshall tests conducted (Fig. 4) show that the bitumen quantity calculated with the proposed methodology (5.43 %) represents a reliable estimation of the measured optimal value (5.47 % of the asphalt mixture). The requirements for Marshall stability and air voids (Fig. 4a, b) are met, instead the flow (Fig. 4c) exceeds the admissible range, expected to be between 2 and 4 mm. This is because the old binder in the RAP becomes softer during heating and acts as a lubricant, increasing the flow during load application. The optimal bitumen quantity according to the ITST (Fig. 5) at 15 °C is 5.38 % of the mixture. The theoretical calculation provides also in this case a reliable estimation of the optimal bitumen quantity measured; indeed the values differ only by 1.1 %. The quantity of the total bitumen estimated (virgin bitumen + aged binder) in the mixture is high (8.23 %). This should be considered when life cycle cost analysis of this solution is performed. Indeed, according to recent studies [37] there is no energy gain in use of RAP if the bitumen is hardened. This raises the problem of how the old binder can be reactivated performing the same function as in its original application. Indeed, the concept of asphalt recycling involves the use of the RAP aggregates and binder and it is environmentally preferable to asphalt reuse, i.e.

**Fig. 6** Gyratory air void content for AC B 16 asphalt mixtures with 100 % of virgin aggregates, with 50 % of RAP without considering clustering and 50 % of RAP considering clustering for readjustment of grading curve



the old bitumen performs a lesser function than in the original application.

Figure 6 shows a comparison of the compactability trend as a function of the bitumen quantity for AC B 16 mixtures with 100 % of virgin aggregates; with 50 % of RAP without changing the virgin aggregate proportions after the RAP particle agglomeration (original grading curve), and a mixture with 50 % of RAP where the proportions of virgin aggregates have been changed considering the RAP clustering by readjusted grading curve.

From Fig. 6 it is obvious that the compactability of virgin and RAP mixtures is significantly different. For low bitumen content (4.5 and 5 %) the effect of the aged binder in both RAP mixtures is significant because it acts as lubricant increasing the compactability. As the amount of virgin binder increases the air voids decrease more rapidly in case of a virgin mixture. The effect of the aged binder in RAP mixtures becomes less evident and is probably masked by the presence of intergranular voids trapped in the RAP clusters. The air voids trapped in the old RAP clusters, i.e. clusters present before the new RAP mixing, are not attainable by the bitumen. Thus, even if virgin bitumen is added, the air voids cannot be filled. Moreover with the formation of new RAP clusters mixing the RAP a reduction in the small-size particles occurs. This causes a change in the final grading curve and an increase of air voids. Thus, for mixtures with 50 % of RAP without the proposed readjustment of the grading curve, it appears difficult to achieve an acceptable air void content (<6 %). Instead, if clustering is taken into account and specific

amounts of virgin aggregates are added to replace the agglomerated small-size RAP particles, the compactability of the mixture improves and an acceptable range of air voids is obtained. A consequence of these conclusions is that it is essential to break the old RAP clusters and avoid the presence of intergranular voids to reach the acceptable voids range. One possible solution could be to select an appropriate mixing energy during the asphalt mixture fabrication.

## 5 Conclusions and recommendations

The effects of RAP particle agglomeration on the expected grading curve and consequently on the optimal dosage of virgin bitumen were analysed in order to establish a new analytical mix design method for mixtures with high RAP content. The present study led to the development of the theoretical formula for the optimal bitumen quantity in the mixture.

With the methodology proposed it is possible to manipulate the virgin aggregates to readjust the grading curve, knowing the change caused by the RAP clustering. Moreover, potentially it would be possible to use different bitumen film thicknesses for different sizes of particles.

Laboratory verification was carried out and the results showed that:

- The theoretical formula estimates a reliable value of optimal bitumen content in case of an AC B 16 mixture with 50 % of RAP (5.43 %) that is

comparable to the measured values obtained with Marshall (5.47 %) and ITST (5.38 %). All the requirements are met except for the flow in the Marshall test. This is because the aged binder becomes softer during heating and acts as a lubricant, increasing the flow during load application.

- The compactability, measured with the gyratory compactor, for RAP mixtures is different from that of virgin mixtures. The reduction of the amount of small-size particles due to the clustering and intergranular voids trapped in the old RAP clusters prevents the achievement of an acceptable air voids content. The RAP particle agglomeration causes a change in the final grading curve and an increase of air voids. The mixtures designed with the readjustment of the grading curve and a calculation of the specific amounts of virgin aggregates for every fraction to replace the agglomerated small-size RAP particles exhibit improved compactability (acceptable voids content). Nevertheless, these results show that it is essential to select an appropriate mixing energy during asphalt mixture fabrication in order to break the old RAP clusters and prevent the presence of intergranular voids in the old clusters.

The methodology represents a first step towards a new mix design approach for mixtures with high percentage of RAP. Based on the results it is considered successful in estimating the value of the optimal bitumen quantity. The advantage of applying this methodology is the reduction of the laboratory testing efforts since the estimated value of the optimal bitumen quantity represents a reliable prediction of the measured one. Moreover, the formula allows the control of the bitumen film thickness around the different components of the asphalt mixture (mineral aggregates, RAP and filler). Thus, the optimal bitumen film thickness around the aggregates can be changed depending on the needs and it would potentially be possible to use different bitumen film thicknesses for different particle sizes. Finally, knowing the RAP grading curve and establishing the RAP percentage it would be possible to calculate the agglomeration of RAP particles for every fraction and to readjust the target grading curve modifying the amount of virgin aggregates for every fraction. Nevertheless, additional work is needed to validate the methodology using other materials (RAP, virgin aggregates and filler),

other types of mixtures and other fabrication temperatures. Even if the methodology presented in this paper is valid only for the materials used for this work, it could be adopted for other materials after their characterization.

In particular RAP clustering needs to be investigated with RAP originating from other sources and for different fabrication temperature to define which parameters have an effect on cluster formation. Moreover, the asphalt mixtures resulting from the proposed mix design methodology should be tested to evaluate the stiffness, permanent deformation and fatigue resistance.

To establish a repeatable procedure in the laboratory it was necessary to control all the variables for keeping the mixing conditions consistent. Nevertheless, it should be noted that passing from the laboratory scale to the plant scale the considered parameters, and therefore the entire procedure, might be subjected to variations. For this reason, in future works it is fundamental to verify and adapt the methodology testing RAP asphalt mixtures produced in the plant.

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