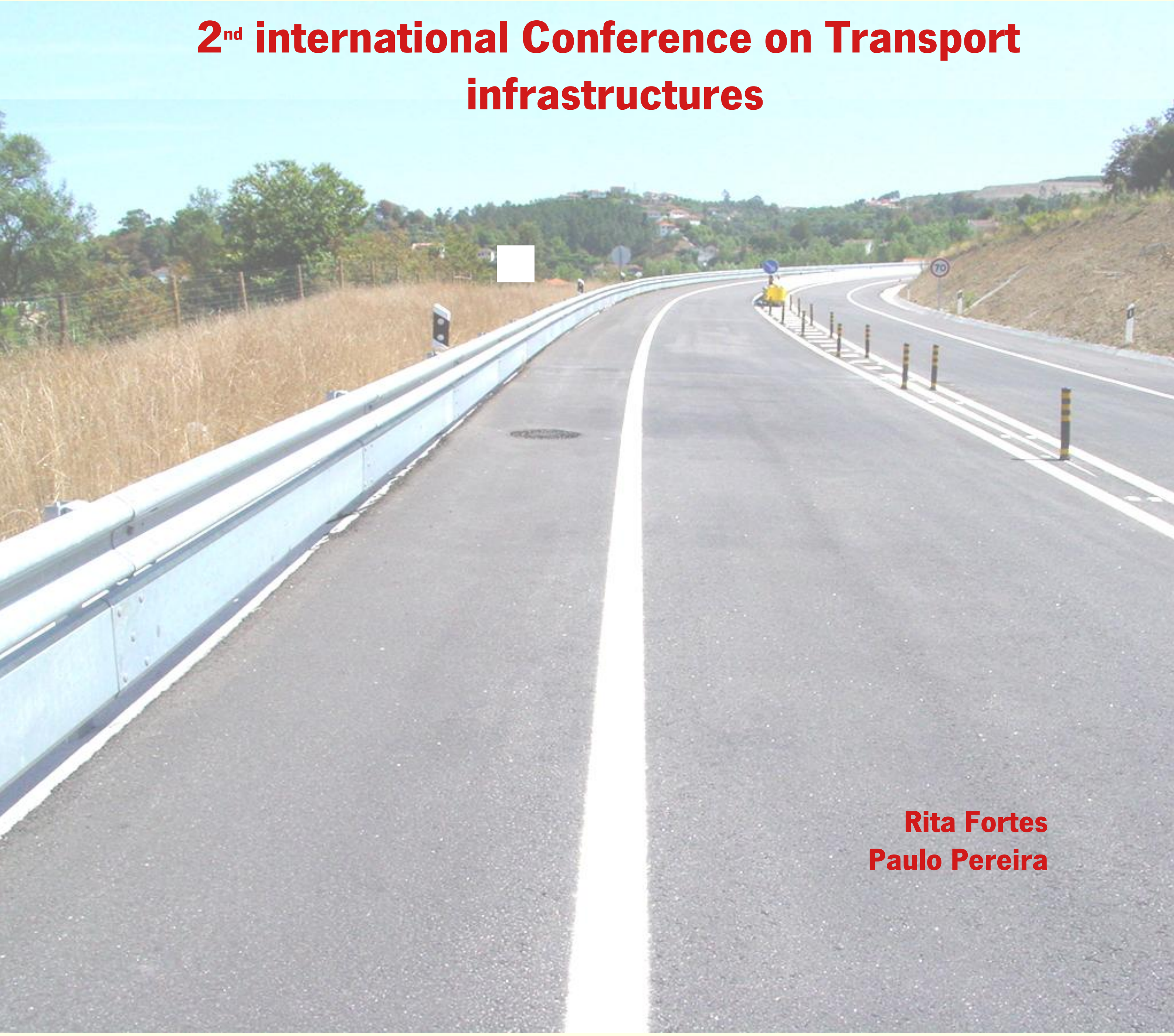
2nd international Conference on Transport infrastructures



Rita Fortes
Paulo Pereira

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Edited by

Rita Moura Fortes
Escola de Engenharia
Universidade Presbiteriana Mackenzie, Brasil

Paulo Pereira
Escola de Engenharia
Universidade do Minho, Portugal

Departamento de Engenharia Civil, Campus de Azurém, P-4800-058 Guimarães, Portugal
Telefone: +351 253 510 240; Fax: +351 253 510 217
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4º CONGRESSO DE INFRAESTRUTURA DE TRANSPORTES CONINFRA 2010

PREFACE

A suitable transport infrastructure has always been essential for the mobility of the persons and goods all over the world since remote ages.

Most of the transport infrastructures have been developed under national policy premises. There is the need to establish a single, multimodal system integrating land, sea and air transport connections which ensure the sustainability of our transport networks into the future. Environmental protection requirements are also the key to development sustainability and should not be left apart.

The worldwide awareness of the sustainability of life and nature is indicative of an important role that civil infrastructures play in guaranteeing a sustainable life quality. Once an infrastructure is constructed its life-cycle demands a great effort in terms of maintenance and rehabilitation, in order not to jeopardize the initial investment in infrastructure assets and not to degrade surrounding and global environment.

Thus, the iCTi series has been implemented with the support of a permanent organization, a technical and learned society – iSMARTi – with the aim of disseminating the most recent research in the themes approached in this international conference.

After its first Conference in China, this second ICTI welcomes you in São Paulo, Brazil.

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Changes in the Rubber Morphology Caused by the Interaction with Bitumen

J. Peralta, H. Silva & J. Pais
Territory, Environment and Construction Centre – C-TAC, University of Minho, Guimarães, Portugal
joana@civil.uminho.pt
hugo@civil.uminho.pt
jpais@civil.uminho.pt

A.V. Machado
Institute for Polymers and Composites/I3N, University of Minho, Guimarães, Portugal
avm@dep.uminho.pt

ABSTRACT: It is estimated that about 10 kg of tires are discarded per inhabitant annually. This residue can be reused as a constituent of pavements in the form of asphalt rubber (AR) binder. The use of bitumen modified with crumb rubber contributes for a sustainable development of road infrastructures through: (i) the definition of an efficient final destination for the used tires of cars and trucks; (ii) the improvement of the performance of the resulting material used in the pavement. However, the materials which constitute these AR binders and the physicochemical changes in the binder during the production of the AR are not sufficiently characterized. Thus, the main objective of this study is to characterize the changes in the morphology of the rubber particles due to the interaction with three different bitumens during the production of the ARs. The studied bitumens were selected from the same origin and obtained in the same distillation column. Initially, the bitumens were left to interact with 17.5% of crumb rubber by mass of AR binder. After that, a modified "Basket Drainage Method" was used to divide the residual bitumen and the rubber of the AR binder. The recovered rubber (RR) particles from the AR binders, and the ones filtered (FR) from the residual bitumens, were then observed in an optical microscope, being measured and weighted. The morphology of the crumb rubber particles was assessed by macroscopic and microscopic observation, and their percentages of depolymerization (ASTM D 6814-02) were also determined. It was observed that the extent of morphological changes in the rubber particles is higher when softer bitumens are used, due to swelling, splitting, depolymerization or devulcanization and eventually repolymerization or revulcanization phenomena.

KEY WORDS: Asphalt rubber, Rubber particles, Swelling, Microscopy, Morphology.

1. INTRODUCTION

Although tires are not regarded as a dangerous residue, their hollow shape usually brings sanitary problems and important difficulties in their final deposition. Furthermore, the society is wasting material from the tire with a high intrinsic value, namely its main constituent: vulcanized natural and synthetic rubber. Thus, the introduction of crumb rubber in the production of Asphalt Rubber (AR) mixtures for road pavements should be considered as a sustainable technology which transforms an unwanted residue into a new bituminous mixture with a high resistance to fatigue and fracture. However, the addition of rubber in a bituminous mixture increases its complexity, hence being essential to carry out a study to understand the interaction between its constituents in order to optimize the performance of AR mixtures.

The main objective of this study is to characterize the changes in the morphology of the rubber particles due to the interaction with three different bitumens during the production of ARs. The main results of this work are (i) the assessment of the changes in the RR and FR morphology due to the interaction with each bitumen; (ii) the quantification of the variation of rubber weight in consequence of bitumen-rubber interaction; (iii) a better understanding of the effect of bitumen constitution in the extent of rubber swelling, splitting and devulcanization.

2. LITERATURE REVIEW ON THE BITUMEN EFFECT ON RUBBER PARTICLES

The morphology of the rubber particles, before and after the interaction with bitumens of different chemical constitution, should be studied because (i) the morphology of the crumb rubber (CR) determine the form and extension of interaction with bitumen and has a significant influence in the viscosity of the AR, and (ii) bitumens with different constitution interact with rubber particles in diverse extension and rate, promoting changes in the rubber particles morphology (Peralta, 2009).

The effects of crumb rubber on asphalt rubber binders can be separated into interaction effect (IE) and particle effect (PE). The IE is the effect of the lighter fractions of the binder diffusing into the crumb rubber particles. The PE is the effect of the crumb rubber particles acting as filler in the binder (Putman and Amirkhanian, 2006). The IE is greatly influenced by the crude source of the binder and could potentially be used as an indicator of a binder's compatibility with CR (higher IE would indicate a more compatible binder). The PE is most significantly affected by the CR content of the binder (higher CR contents result in greater PE values). The effect of CR size on the PE follows the same trends.

The extent of swelling and degradation depends on the nature of the rubbers, the bitumen's chemical composition and the mixing conditions, time, temperature and degree of agitation. For example, during the AR binder production, the interaction of the crumb rubber particles with softer bitumens is clearly higher than with harder bitumens (Peralta *et al.*, 2009). In addition, these processes will determine the mechanical properties of the crumb rubber modified binders (Artamendi and Khalid, 2006). Two main types of activities that affect AR binder properties have been reported in the literature: particle swelling and detachment or depolymerization. These processes occur as the binder is subjected to different combinations of time and temperature (Jensen and Abdelrahman, 2006; Peralta *et al.*, 2009).

During the interaction process, rubber particles swell in a time and temperature dependent manner, which results in a reduction in the interparticle distance, thereby increasing viscosity. Once the rubber has swelled, if temperature is maintained too high or for too long a period, the rubber begins to disintegrate into the asphalt by partial depolymerization, causing a reduction in viscosity. Change in the viscosity of the asphalt rubber binder has traditionally been used to indicate the progress of the interaction between asphalt and rubber (Jensen and Abdelrahman, 2006). The application of higher temperatures for long periods of time, during asphalt rubber production, results in swelling followed by depolymerization (Khalid, 2005), whereas applying lower temperatures results only in swelling (Leite and Soares, 1999).

Post-vulcanization (Green and Tolonen, 1977) is an interesting phenomenon that sometimes occurs when mixing asphalt cement with rubber. During rubber processing there are sulphur and other agents that have not been entirely chemically bonded during vulcanization of the rubber. When mixing rubber with hot asphalt, the vulcanization process will be reactivated and continue for some time, depending on the interaction temperature.

3. BITUMEN-RUBBER INTERACTION AND RUBBER SEPARATION AND CHARACTERISATION

3.1. Bitumen selection and samples preparation

The bitumens used in this work were carefully selected, being collected from the same batch of production at the refinery, because the characteristics of bitumens are highly depend on their sources and processing. Three bitumens were chosen to carry out this research, namely the commercial bitumens A (10/20), I (60/70) and M (150/200), in order to understand the influence of the penetration grade in the rubber morphology.

3.2. Crumb rubber selection and samples preparation

The crumb rubber used in this work was obtained by the cryogenic process, being constituted by 30% of truck tyres and 70% of car tyres. The CR was sieved through a sequence of sieves in the laboratory, in order to use only the fraction passed through the sieve ASTM #20 (0.850 mm) and held on the sieve ASTM #40 (0.425 mm). The sieved CR obtained in the lab was washed with toluene and dried in an oven at 135 ºC. The clean and dry rubber particles where observed and measured, using an optical microscope, being also analyzed to assess its soluble percentage (ASTM D 6814-02).

3.3. Interaction with bitumen and separation of the rubber particles

The method used to produce AR and collect the desired samples of rubber was a modified "Basket drainage method" (Rahman, 2004). The used wire basket was manufactured with a square wire mesh, with an opening of 0.470 mm (observed in an optical microscope). The AR production and collection of rubber samples consisted of (i) heating about 1 kg of base bitumen at 180 ºC; (ii) introducing 21.0% w/w of crumb rubber (CR) and re-heating of the mix at 180 ºC; (iii) stirring the "asphalt rubber" at 180 ºC, at a velocity at 230 rot/min for 60 min.; (iv) suspend the basket in a oven at 180 ºC for 15 min and; (v) collecting a sample of the recovered rubber (RR) retained in the basket.

3.4. Recovery and analysis of the rubber particles

Throughout the process of AR production, the rubber particles greatly change their morphology. The changes are caused by the interaction with bitumen, which diffuses through the rubber reticulated structure causing them to swell, while transforming rubber from an elastic solid into a gel structure. Furthermore, the AR production process involves aggressive stirring at high temperatures, which favour the swelling process. When rubber particles turn into gel structures they also become more brittle, and the stirring and eventual localized depolymerisation of the gel rubber particles can break them into little pieces. The wire basket mesh was chosen to unable the rubber particles, in their initial shape, to pass through the wire basket during the drainage of the residual bitumen, but it cannot assure that little pieces of depolymerised rubber will be retained. The reported phenomena may vary when rubber particles interact with different grade base bitumens. In order to evaluate this phenomenon, the rubber particles recovered (RR) from ARs produced with the commercial bitumens and filtered from the respective residual bitumens (FR) were observed and measured with an optical microscope.

The preparation of the RR particles for microscopic observations was carried out by: (i) introducing 2.0 g of RR in a bottle with roughly 200 mL of toluene and agitation of the bottle until the rubber particles were totally separated; (ii) filtering the resulting solution with a filter paper, and washing it with toluene until the filtered solution appears absolutely clean; (iii) drying the filter paper with the resulting RR in an oven at 135 ºC and; (iv) observing and measuring the clean particles in an optical microscope.

The preparation of the FR particles (those suspended in the residual bitumen after going through the wire basket) for the microscopic observations was carried out by: (i) introducing 2.0 g of residual bitumen in a baker of 250 mL capacity; (ii) diluting the residual bitumen with small amounts of toluene while stirring the solution with a glass rod (the diluted solution is filtered through a filter paper and washed with toluene until the filtered solution appears absolutely clean); (iii) drying the filter paper with the resulting FR in an oven at 135 ºC and; (iv) observing and measuring the clean particles in an optical microscope.

4. STUDY OF THE MORPHOLOGY OF THE CRUMB RUBBER PARTICLES

4.1. Percentage of depolymerisation

The crumb rubber solubility was assessed according to ASTM D 6814-02 standard, in order to indirectly evaluate its depolymerisation potential after interacting with bitumen. Two solvents were used in this test, toluene and cycle-hexane, being the CR mass loss respectively equal to 10.9% and 10.7% (low depolymerisation potential). This mass loss can also result from the soluble additives and fillers present in the CR constitution.

4.2. Initial shape and dimension of the crumb rubber particles

The CR particles used in this work were obtained by the cryogenic gridding of used tyres. The resulting rubber particles are very small (Figure 1), thus being impossible to evaluate their shape with the naked eye.

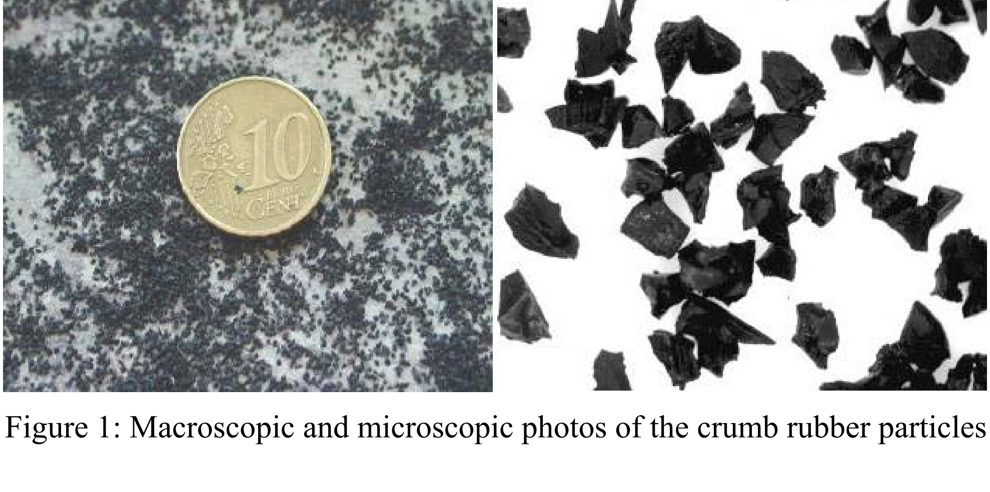

Figure 1: Macroscopic and microscopic photos of the crumb rubber particles

An optical microscope was used to assess the shape and average dimension of the CR particles. It is noticed that the rubber particles present slightly the same size, with an average equivalent diameter of 620.1 µm. The shape of the particles is irregular and angular, but their faces are almost plan.

4.3. Morphology of the recovered rubber particles

4.3.1. Rubber particles recovered from asphalt rubber A

The RR particles from the AR produced with base bitumen A (10/20) were photographed and observed by microscopic methods (Figure 2).

Figure 2: Macroscopic and microscopic photos of the RR particles of the AR produced with bitumen A

The microscopic photo shows that the RR particles present a very different shape and size in comparison with the original crumb rubber. The average equivalent diameter (Øeq = 620,648 µm) of the RR particles is similar to the CR one, but this does not mean that during the AR production the rubber particles were not affected. Actually, the rubber particles swelled and broke during the AR production with the base bitumen A. At the end of the AR production process, the rubber particles present a very irregular shape and very little pieces of rubber can be observed among larger ones. Rubber swelling was assessed using the "Basket drainage method" combined with the "Sphere AR production simulator" method (1, 3), thus being possible to evaluate the variation of the rubber weight during the AR production. During the AR production with bitumen A, the initial weight of rubber was 428.6 g and the final one was 574.4 g, indicating an increase of 25.23%. The percentage of rubber in the beginning of the AR production with bitumen A was 21.0% w/w, but the final percentage of swelled rubber particles in the AR was 70.1% w/w. This increase of 50% is coherent with earlier observations, indicating that the rubber swelling was due to the diffusion of bitumen molecules in its reticulated structure.

4.3.2. Rubber particles recovered from asphalt rubber I

Bitumen I (60/70) is the softer binder generally used to produce AR. The appearance of the RR particles from the AR produced with this bitumen is presented in Figure 3.

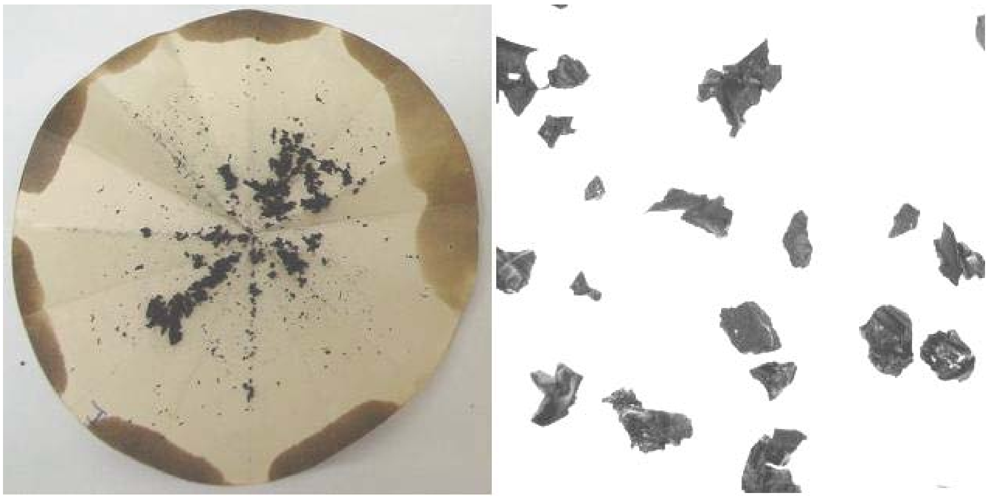

Figure 3: Macroscopic and microscopic photos of the RR particles of the AR produced with bitumen I

The microscopic photo shows a collection of rubber particles recovered from the AR produced with bitumen I. There are RR particles of several sizes, forming a continuous particle size distribution. In average, the equivalent diameter of these particles is 634.19 µm. The signs of degradation, holes and irregularities in the particles are more marked than in the RR obtained after interaction with bitumen A. The initial weight of rubber used to produce the AR with bitumen I was 210.84 g and the final weight of the RR after swelling was 379.57 g (increase in mass of 44.5%). The percentage of the rubber particles in the AR increased from 21.0%, at the beginning, to 77.87% at the end of the AR production with bitumen I.

4.3.3. Rubber particles recovered from asphalt rubber M

Usually, 150/200 pen grade bitumens (M) are not used to produce bituminous mixtures, whether rubberized or not. However, it was used in this research work about AR binders. Figure 4 presents the photos of the RR from AR produced with bitumen M.

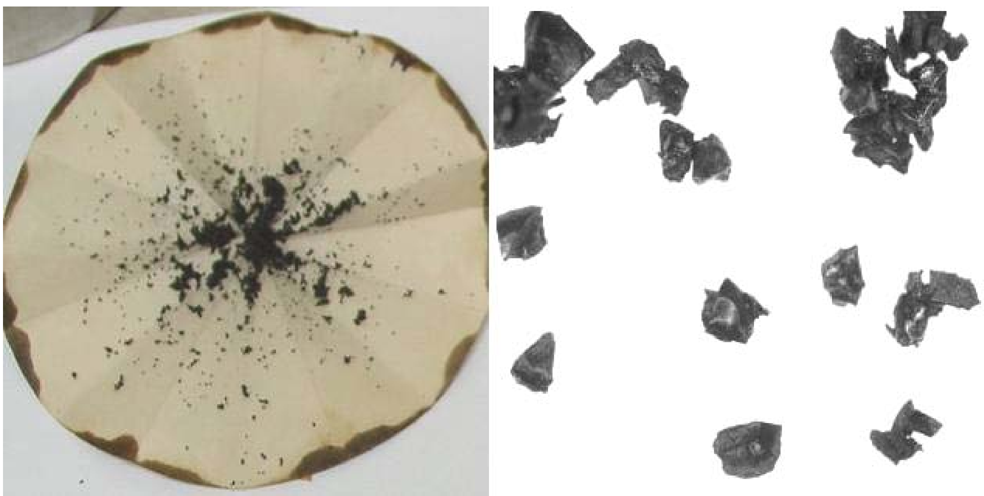

Figure 4: Macroscopic and microscopic photos of the RR particles of the AR produced with bitumen M

Surprisingly, and in contrast with the RR from the previous ARs, the rubber particles recovered from AR produced with bitumen M have slightly the same size (608.42 µm), i.e., the particle size distribution is narrow and sharp. Additionally, the rubber particles are not isolated (as the RR from the other ARs), forming little clusters. During the interaction with bitumen M, the rubber particles augment their weight in 51.6% (210.11 to 434.39 g). Thus, the final percentage of swelled rubber in the AR was 81.8%, being much higher than the initial value of 21.0%.

4.4. Morphology of the filtered rubber particles

During the AR production the rubber particles split in little pieces and some passed through the wire basket.

Therefore, to quantify and make a visual inspection of these little pieces of rubber (FR), they were observed to the naked eye and in an optical microscope. To assess the percentage of rubber in the residual bitumen, the mass of the insoluble material in the base bitumen and in the residual bitumen was determined, by using EN 12592 standard (the comparison between the two values is the mass concentration of FR in the residual bitumen).

4.4.1. Rubber particles filtered from asphalt rubber A

The photos of the filter containing the FR particles from the residual bitumen A and the microscopic image of the FR particles are presented in Figure 5.

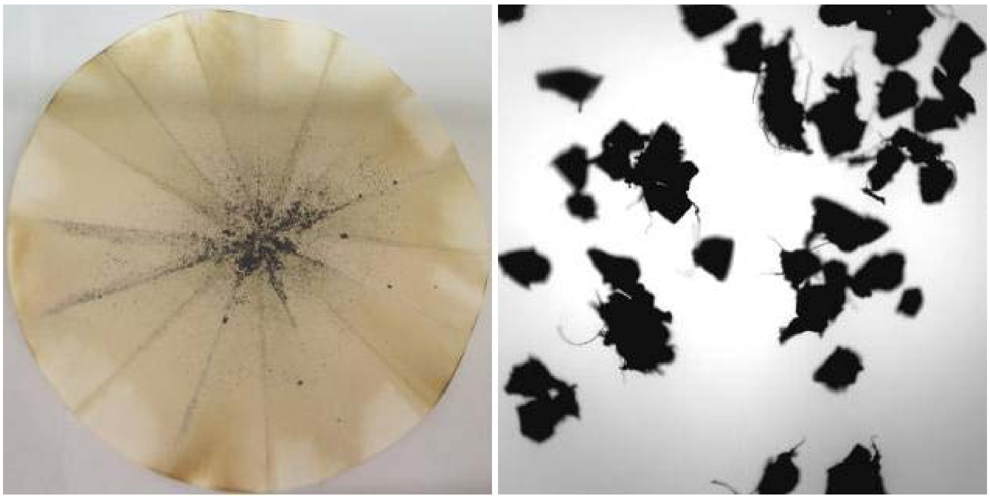

Figure 5: Macroscopic and microscopic photos of the FR from residual bitumen A

Observing the photo in the left side, it is evident that the dimension of the FR particles is clearly lower than the previously observed (RR). However, the FR particles are easily identified to the naked eye. Surprisingly, when analysing the microscopic photo, it was noticed a similarity in the size of the FR particles from residual bitumen A. Their equivalent diameter was 378.67 μm, thus being lower than the CR and RR particles. The mass concentration of FR in the residual bitumen was 1.01% w/w, thus being almost undetectable in the physical and rheological characterization of this material.

4.4.2. Rubber particles filtered from asphalt rubber I

Figure 6 presents the photos of the FR particles from residual bitumen I (filter and at an optical microscope).

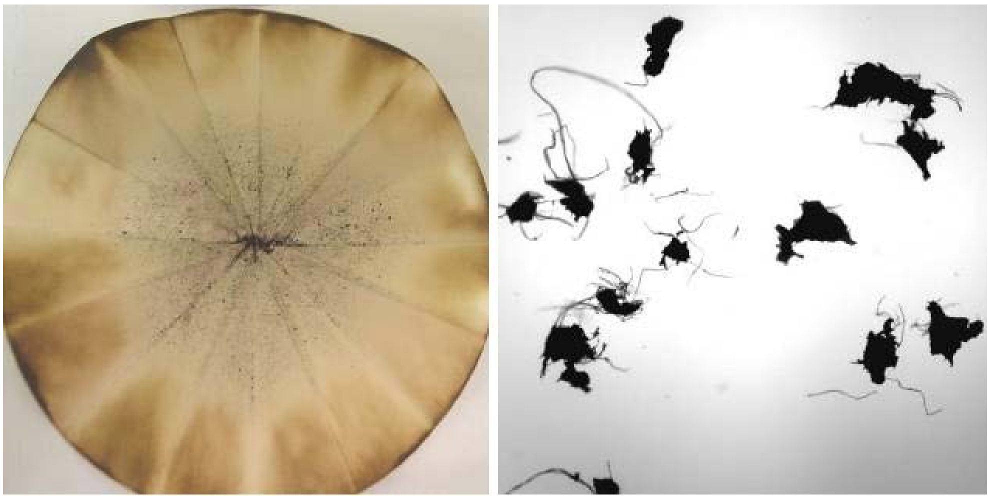

Figure 6: Macroscopic and microscopic photos of the FR from residual bitumen I

The photo of the filter clearly shows a reduction in the amount of FR particles in the residual I, when compared with the previous FR from residual bitumen A. In fact, its mass concentration in the residual bitumen I was assessed, being equal to 0.34%. At the same time, the dimension of these FR particles seems to be much lower than the one of the FR observed before (residual bitumen A). Actually, an average equivalent diameter of 278.76 µm was determined by carrying out a microscopic evaluation.

4.4.3. Rubber particles filtered from asphalt rubber M

The macro and microscopic photos of the FR particles from residual bitumen M are presented in Figure 7.

Figure 7: Macroscopic and microscopic photos of the FR from residual bitumen M

The appearance of the FR from residual bitumen M is even dustier and the quantity of rubber retained on the filter is lower than that previously observed. In fact, the residual bitumen M contains only 0.14% w/w of FR particles. Unsurprisingly, the equivalent diameter of the FR from residual bitumen M is only 282.98 µm. Furthermore, the FR particles appear to be stick to each other, as earlier observed in the RR from AR M.

5. CONCLUSIONS

Several morphological changes on CR were detected during the AR production and were related with the used base bitumen. The rubber particles swell due to the diffusion of small molecules of bitumen in the reticulated molecular structure of rubber. The swelling increases with the base bitumen penetration, until a maximum value that corresponds to the saturation of the rubber. The rate and extent of swelling of rubber particles in the AR depend on the concentration of bitumen molecules capable of diffusing into the rubber bulk. The rubber particles saturated with bitumen molecules become a brittle gel that can easily split by stirring, heating and depolymerisation. Therefore, a slight increase in the volume of rubber for bitumens with penetrations lower than 70 dmm was observed, although for softer bitumens the average dimension of the rubber particles decreased.

During AR binder production, the interaction of crumb rubber particles with softer bitumens is clearly higher, and the morphology of the rubber particles is clearly more influenced by those bitumens than by harder ones. Apparently the rubber particles in contact with softer bitumens swell much more, splitting and releasing a great amount of very small elements. Thus, the particles filtered from residual bitumen A are the largest and the ones filtered from residual bitumens I and M are the smaller. When softer bitumens are used, rubber particles can swell to their saturation and re-vulcanization and re polymerization among the rubber particles can occur.

REFERENCES

Artamendi, I., Khalid, H.A., 2006. *Diffusion Kinetics of Bitumen into Waste Tyre Rubber*. Journal AAPT, Vol. 75, Savannah, Georgia, pp. 133-164.

Green, E.L., Tolonen, W.J., 1977. *The Chemical and Physical Properties of Asphalt-Rubber Mixtures*. FHWA-AZ-HPR14-162, Arizona DoT, Report ADOT-RS-14 (162), USA.

Jensen, W., Abdelrahman, M., 2006. *Crumb Rubber in Performance-Graded Asphalt Binder*. Nebraska Department of Roads, Final Report, SPR-01 (05) P585, University of Nebraska-Lincoln, USA.

Khalid, H.A., 2005. *Recent Research on Use of Rubber in Asphalt*. WRAP Rubber in Roads Seminar, University of Liverpool, UK.

Leite, L.F.M., Soares, B.G., 1999. *Interaction of Asphalt with Ground Tire Rubber*. Petroleum Science and Technology, 17 (9), pp. 1071-1088.

Peralta, J., 2009. *Study of the Interaction between Bitumen and Rubber*. Thesis submitted to the University of Minho for the degree of Master of Science, Department of Civil Engineering, University of Minho, Portugal.

Peralta, J., Silva, H.M.R.D., Pais, J., Machado, A.V., 2009. *Rheological and Functional Evaluation of the Interactions between Bitumen and Rubber*. 7th International RILEM Conference, Rhodes, Greece.

Putman, B.J., Amirkhanian, S.N., 2006. *Crumb Rubber Modification of Binders: Interaction and Particle Effects*. AR2006 Conference, Palm Springs, USA.

Rahman, M.M., 2004. *Characterisation of Dry Process Crumb Rubber Modified Asphalt Mixtures*. Thesis submitted to the University of Nottingham for the degree of Doctor of Philosophy, University of Nottingham, School of Civil Engineering, UK.