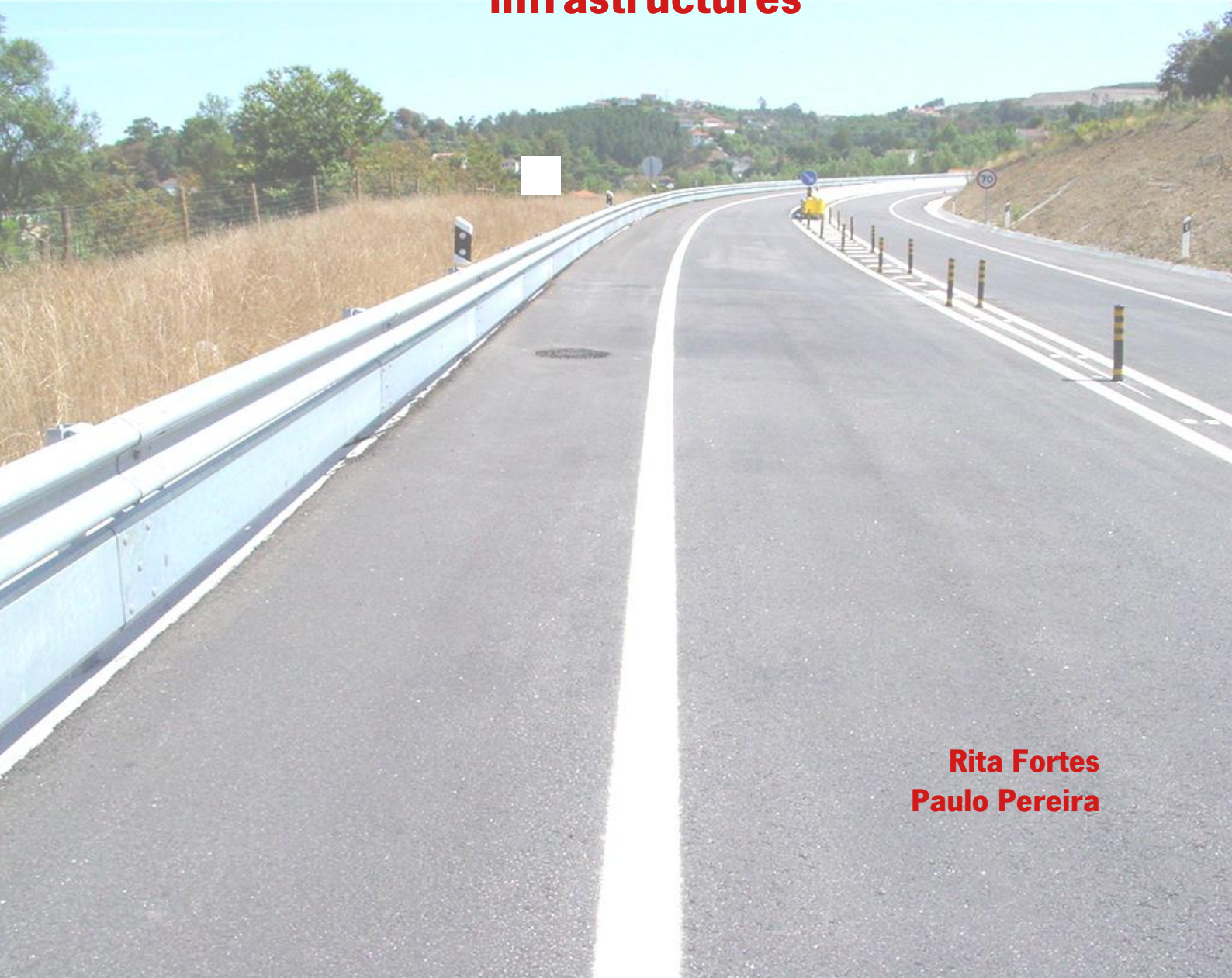




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Paulo Pereira**

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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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Monitoring Rubber Swelling and De- and Re-vulcanization through AR Elastic Recovery

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ABSTRACT: Healthy economies require an appropriate transportation system, in which roadways are an essential part. Therefore, the construction and maintenance of road pavements should be long lasting and have a significant impact on the economic vitality of a nation. The elastic recovery is a property characteristic of the elastomers, or binders modified with those polymers, being used to classify them and especially to survey the polymerization reactions in rubber production and the crosslink density during the vulcanization process. In this work, a large range of pen grade bitumens from the same source were used to produce different asphalt rubber binders (ARs). Then, the elastic recovery (ER) of each AR binder was determined while the rubber used to produce the AR was recovered, weighted and observed by optical microscopy. Finally, the elastic recovery of the AR binder was related to the corresponding weight percentage and dimensions of the rubber particles recovered from the final AR binder. The main conclusion draw from this study is that the analysis of the elastic recovery of the AR binder can give very useful information about the magnitude of rubber swelling, de- and re-vulcanization and final degradation of the rubber networks. The results of the elastic recovery of the AR binders and the recovered rubber properties also suggested that it possible to select a bitumen to produce AR binders with enhanced characteristics.

KEY WORDS: Elastic recovery, Rubber, Dimensions, De/revulcanization, De/repolymerization.

1. INTRODUCTION

The construction and maintenance of road pavements should be long lasting and have a significant impact on the economic vitality of a nation. Beyond the traditional constituents of the bituminous pavement layers, the use of crumb rubber recycled from used tyres should be studied as a form of environmental protection and as a pavement performance enhancer.

Billiter et al. (1997a) referred that the improved adhesion of asphalt-rubber binder (AR) promotes chip retention when it is used as a chip seal. Thus, rubber modified pavement improves skid resistance even under icy conditions. Furthermore, the practice of utilizing rubber in bitumen is environmentally friendly since it eliminates a solid waste and is able to reduce traffic noise.

The obvious benefit of adding rubber to bitumen is that the rubber imparts elasticity to the binders, which helps increase pavement fatigue life or fatigue resistance, as well as reduces reflective cracking. This elasticity also makes the pavement more flexible, thus improving the pavement's susceptibility to low-temperature cracking. It has been reported that rubber also imparts tensile strength, ductility, toughness, resiliency, tenacity and thus, durability to the binder modification (Martinez *et al.*, 2006; Neto *et al.*, 2003; Way, 2003; Gopal *et al.*, 2002; Coomarasamy and Hesp, 1998).

Elastic recovery describes the ability of a bitumen binder to elongate when tension is applied and to recover its original shape when the tension is released. This property is important in both fatigue and rutting resistance. Additionally, elastic recovery is a property that is indicative of the quality of polymer components in bitumen binders (Jensen and Abdelrahman, 2006). The elastic recovery of the asphalt-rubber binders tends to increase as the rubber particle size decreases. Finer rubber particles result in higher ductility than larger particles and, in accordance, toughness increases as particle size decreases (Jensen and Abdelrahman, 2006).

Some authors referred some breakings in cross-links (devulcanization) due to heating during the curing process (Billiter *et al.*, 1996). This phenomenon induces a progressive decrease in viscosity, which is considered, in some papers, as the initial degradation of the polymer chains. Both swelling and devulcanization are present during the asphalt rubber curing, even for the so-called dry process. The predominance of one of the phenomena relies on the processing parameters and essentially on blending temperature and digestion time (Ould-Henia and Dumont, 2006).

The effect of the interaction conditions on the development of the high temperature properties can be summarized as follows (Jensen and Abdelrahman, 2006):

- The temperature controls the activity of the interaction process. There are two main activities within the interaction process, namely swelling and depolymerisation. Interaction temperature affects the process by controlling the time when swelling is replaced with depolymerization;
- Shearing energy can be very effective in controlling crumb rubber particle sizes during the interaction process. Higher shear turns coarser particles into fine particles and help stabilize crumb rubber binder properties.

The absorption/swelling capacity of a rubber particle is related to the crude source, the penetration grade of the binder and the nature of the crumb rubber (Rahman, 2004; Airey *et al.*, 2003; Shen *et al.*, 2009). Two main types of activities that affect AR binder properties have been reported in the literature: particle swelling and detachment or depolymerization.

Rubber particles might swell to 3-5 times their original volume (Massucco, 1994). Moreover, the extent of rubber swelling in asphalt varies according to the temperature and time of rubber-asphalt contact, chemical composition of asphalt, rubber type, and particle size (Stroup-Gardiner *et al.*, 1993).

The scrap tire rubber used as an asphalt modifier is in a vulcanized state. Swelling accounts for a partial degeneration of the polymer properties (Lougheed and Papagiannakis, 1996). By using high temperature and high shear, the rubber particles can be devulcanized and depolymerized in the asphalt. It has been found that the devulcanization and depolymerization of rubber is higher when asphalts are rich in aromatics (Billiter *et al.*, 1996). Increased rubber dissolution was determined to improve the low- and intermediate-temperature rheological properties of a binder (Billiter *et al.*, 1997b). If the rubber is not partially devulcanized, it produces a heterogeneous AR binder with the rubber acting mainly as a flexible filler (Giavarini, 1994). However, excessive devulcanization brings about the deterioration of binder properties. An improvement in the elasticity of the binder may be achieved by additional cross-linking of the polymer in the asphalt-rubber binder (re-vulcanization). Xiang

et al. (2009) reported that a fine and perfect polymer reticular structure was formed when crumb rubber powder was used to produce AR.

These literature references suggest that a good relation may be observed between the changes in rubber morphology and concentration, during AR production, and the elastic recovery (ER) of AR binder. Thus, monitoring of the elastic recovery of AR binders can be an easy and useful method to assess the optimum parameters of interaction between bitumen and rubber, in order to achieve the best AR binder performance

Therefore, the aim of this work is to assess the elastic recovery of AR binders and relate them to the corresponding changes in the rubber concentration and dimensions. A large range of base bitumens was used in order to carry out this study, and thus the type of base bitumen was one of the main parameters evaluated.

2. MATERIALS AND METHODS

2.1. Materials

In order to minimize any variability in the characteristics of the materials due to their source and processing, bitumens were collected from the same batch of the La Rábida refinery of CEPESA in Huelva, Spain.

Asphalt rubber properties are very dependent on the used bitumen. Thus, in this work, several grade bitumens were initially chosen in order to evaluate their influence on the AR binder characteristics. Due to the difficulty of the refinery to supply other bitumens than those from their commercial line (10/20, 40/50, 60/70 and 150/200), these commercial bitumens were combined in different proportions in order to obtain eight base bitumen samples, as presented in Table 1.

Table 1: Proportions of commercial bitumen used to obtain the eight base bitumen samples

Sample labeling		A	B	E	I	J	M	N	O
Commercial bitumen	10/20	1.00	0.75			0.25			
	40/50		0.25	1.00					
	60/70				1.00	0.75		0.25	0.50
	150/200						1.00	0.75	0.50

The laboratorial characterisation of these base bitumen samples was carried out in order to evaluate some physical empiric properties, namely the penetration at 25 °C (EN 1426), ring and ball softening point (EN 1427) and the dynamic viscosity (EN 13302).

The crumb rubber used in this work was produced by the cryogenic process and, according to the supplier, was obtained by the grinding of 30% of truck tires and 70% of car tires. The crumb rubber size ranges between 0.18 and 0.60 mm (sieves ASTM #80 to #30). These sizes represent the AR mixtures typically produced and applied in Portuguese roads.

The supplied crumb rubber was sieved so as to select and use only the fraction passed in sieve ASTM #20 (0.850 mm) and retained in sieve ASTM #40 (0.425 mm). The crumb rubber sieved in the laboratory was washed with toluene and dried in an oven at 135°C. After cleaning the rubber particles, they were analyzed by optical microscopy (carried out to assess the rubber particles form and size), using an Olympus BH-2 microscope and a magnification of 100x (10x × 10x).

2.2. Methods

The method used to produce AR and collect the desired samples of AR, of recovered swelled rubber and of residual bitumen was the “basket drainage method” (Rahman, 2004). The asphalt rubber production facility is constituted by several equipments, assembled in a laboratory ventilation chamber.

The wire basket used in the AR production facility was manufactured with a wire mesh, which was previously observed in an optical microscope to measure its average opening dimension (# 0.370 mm).

The process used to produce and collect the several samples of AR, of recovered swelled rubber and of residual bitumen consisted of heating of 1 kg of each base bitumen at 180°C (collecting a sample). Then, 21% (w/w) of crumb rubber were added to the bitumen, and the resulting AR binder was continuously heated at 180 °C for 60 minutes (digestion time), while stirring the binder at 230 rot/min. A sample of the produced asphalt rubber binder (AR) was collected and finally, the wire basket with the AR binder was suspended in an oven at 180°C for 15 minutes, so as to separate and weight its constituents (recovered swelled rubber and residual bitumen). Finally, the elastic recovery of the AR binder sample was determined.

In order to prepare the recovered rubber particles for microscopic observations, 2.0 g of recovered rubber were put in a container with approximately 200 mL of toluene, and the container was slightly shaken until the rubber particles were totally separated. The resulting solution was filtered with a filter paper and washed again with toluene until the filtered solution became completely clean. Finally, the filter paper with the resulting recovered rubber was dried in an oven at 135 °C and, ultimately, analyzed by optical microscopy.

The recovered and filtered samples for microscopy tests (carried out to assess the rubber particles size) were also obtained by using standard EN 12592, repeating the procedure several times if necessary until the adequate number of rubber particles (about 150 in this study, according to standard ISO 9276) were obtained.

The washed and dried rubber particles were analyzed by optical microscopy using an Olympus BH-2 microscope and a magnification of 100x (10x × 10x). The rubber particle size was quantified using a method of image analysis (Leica Quantimet 550). The results were obtained after studying typically 150 rubber particles.

3. RESULTS AND DISCUSSION

The swelling of the rubber increases with the base bitumen penetration, going to a maximum value that corresponds to the saturation of the rubber particles. When this point is achieved, the rubber particles are in the form of a gel and the diffusion process of bitumen molecules into rubber reticules cannot continue. The stirring and the high temperatures cause the rupture of the particles, exposing the centre of each particle to the bitumen, thus maintaining the swelling process. Simultaneously, parts of the gel rubber particles are partly or completely depolymerized, depending on the concentration of small molecules in the bitumen. In fact, when the swelling process of the rubber particles is complete, splitting and degradation of the rubber gel particles can occur in the AR binder. Therefore, it is evident that, as the AR production process takes place, the relative importance of the rubber particles in the binder increases. In fact, the evolution of the rubber weight concentration (%) in the AR and in the residual bitumen, as a function of the penetration of the used base bitumen, was quantified and compared with the concentration of the crumb rubber in the beginning of the ARs production process (17.35 % w/w).

Figures 1, 2 and 3 show the quantitative representation of the results obtained in the tests performed to assess the changes in the morphology of the rubber particles during the production of AR binders with various pen grade bitumens. Those Figures are a summary of the observations made in this work, allowing a holistic understanding of the AR production process from the point of view of the rubber particles.

Therefore, the points representing the evolution of the equivalent diameter of the crumb rubber initially added to the 4 commercial base bitumens, after being recovered from the AR, are presented in Figure 1 in function of the rubber mass concentration (%) in the AR binder.

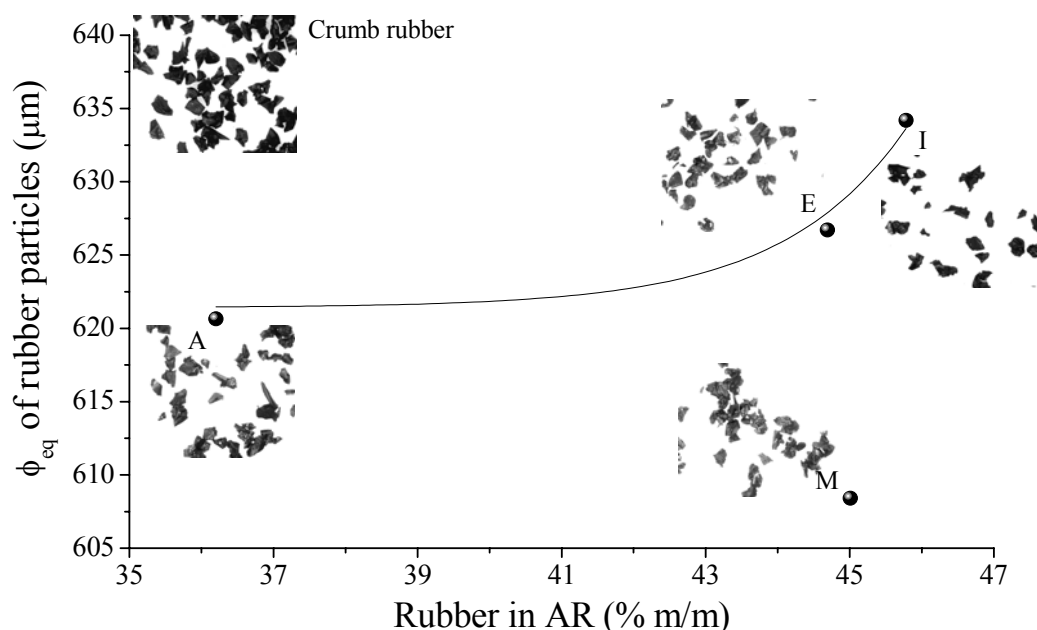


Figure 1: Correlation between the changes in the concentration and dimension of rubber in AR produced with four commercial bitumens

The same crumb rubber, with an equivalent diameter (ϕ_{eq}) of 620.10 μm , was always used on the production process of the different AR binders.

The swelling of the rubber increases with the penetration of the base bitumen, trending to a maximum value that corresponds to the saturation of the rubber particles. When this point is achieved, the rubber particles are in the form of a gel and the diffusion process of bitumen molecules into rubber reticules cannot continue. These observations show that during the AR production process the rubber particles swell and transform into a fragile gel. The stirring and high temperature caused the rupture of the particles, exposing the centre of each particle to the bitumen, aiding the prolongation of the swelling process. Simultaneously, parts of the gel rubber particles are partly or completely depolymerized, depending on the concentration of little molecules in the bitumen. In fact, when the swelling process of the rubber particles is complete, the phenomena that occur in the AR are the splitting and degradation of the rubber gel particles. However, re-polymerization and re-vulcanization may concurrently occur.

Attending on the facts previously exposed, it is evident that, as the AR production process takes place, the relative importance of the rubber particles in the binder increases. Therefore, the evolution of the rubber mass concentration (%) in the AR binder, in function of the used base bitumen, was quantified and compared to the concentration of the crumb rubber in the beginning of the AR production process (17.35 % w/w).

During the AR binder production, the interaction of the crumb rubber particles with softer bitumens is clearly higher than with harder bitumens, and thus the morphology of the rubber particles is clearly more influenced by softer bitumens. Apparently the rubber particles in

contact with softer bitumens swell much more, splitting and releasing a great amount of very small elements. Furthermore, the formation of smaller particles increases the rubber volume fraction in AR binder, inducing the re-vulcanization/re-polymerization, as shown in the rubber particles recovered from AR M (Figure 1).

The elastic recovery is a property characteristic of the elastomers, being used to classify them and especially to survey the polymerization reactions in rubber production and the crosslink density during the vulcanization process. The introduction of rubber in the bituminous binders confers to the pavement, among others, the capacity of recovering the form after being subjected several times to deformation without showing signs of fatigue.

The graphic presented in Figure 2 was drawn in order to analyze the effect of the final ϕ_{eq} of the rubber particles (recovered from the four ARs produced with the commercial bitumens) in the resultant elastic recovery of the AR binder.

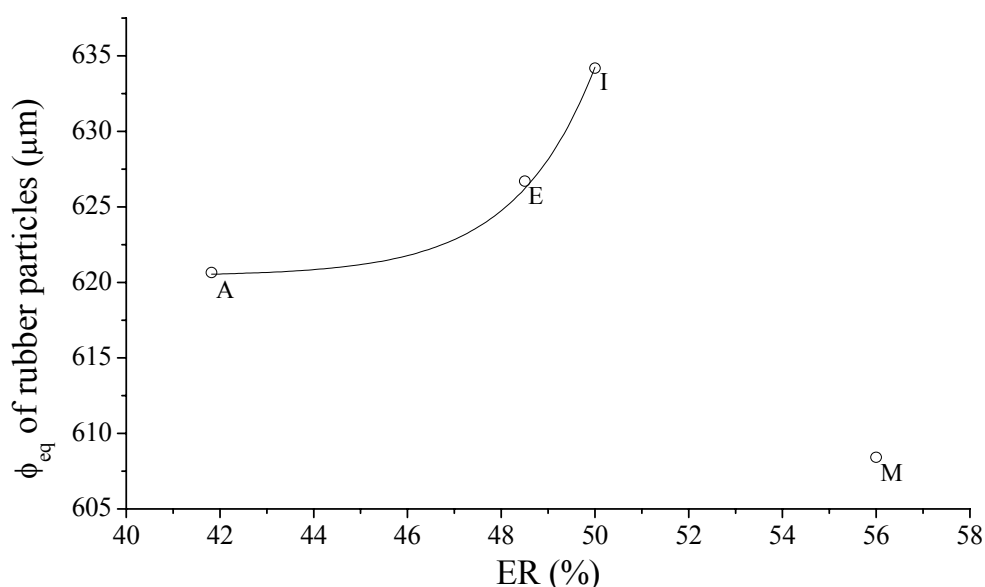


Figure 2: Correlation between the rubber particles dimensions and the elastic recovery of the AR binders produced with four different commercial bitumens

By analyzing the previous graphic, it was observed that the rubber swelling phenomenon increases the value of AR elastic recovery. In fact, the larger the rubber particles present in the AR are, the higher is the elastic recovery of the AR binder. Furthermore, the rubber particles swelling phenomenon is proportional to the quantity of small molecules in the AR, and the swelling process only ends when the rubber particles are saturated and/or when there is no more molecules to be absorbed.

The equivalent diameter of the rubber particles recovered from the AR binders (ϕ_{eq}) can be related with the elastic recovery by an exponential trending line, but only until the saturation and splitting of the crumb rubber, which occurs when rubber interacts with softer base bitumens (e.g. bitumen M). Actually, when crumb rubber interacted with very soft bitumens (e.g. bitumen M), it was noticed that the elastic recovery is still increasing in spite of the reduction of the ϕ_{eq} . This behaviour is similar to that observed in Figure 1, but appears to be somewhat contrary to the one usually reported in the literature, which indicates that the elastic recovery is a function of the rubber particles size and content.

In order to clarify the previous observations, and essentially to analyze if the rubber content is more influential than the particles size, a large range of pen grade bitumens were used to produce AR binders and their final elastic recovery and rubber concentration were determined (Figure 3).

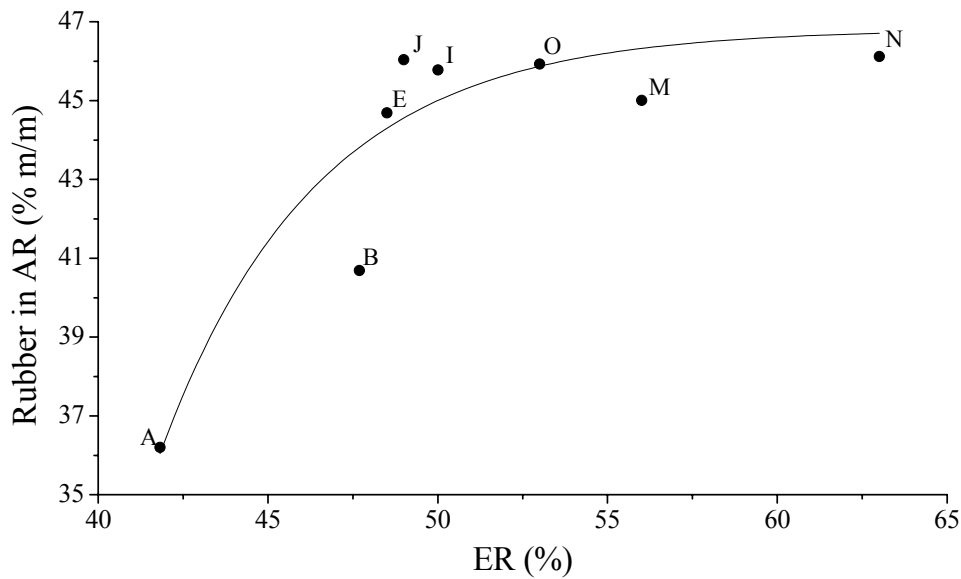


Figure 3: Correlation between the elastic recovery and the rubber final concentration in the AR binders produced with eight different bitumens

The higher interaction between the rubber particles and softer base bitumens enhances the elastic recovery of the resultant AR binder, but only until a certain point. In this study, when the softest base bitumen (M) was used, the interaction with rubber particles was maximized. Nevertheless, the elastic recovery was lower than that verified with bitumen N.

From the obtained results it was observed that the final rubber percentage in the AR binder has an exponential relation with the respective elastic recovery. However, when softer bitumens (I, O, M and N) are used, the rubber percentage in the AR binder is roughly the same in spite of the significant changes in the elastic recovery.

These observations indicate that when the rubber saturation is achieved, the rubber particles start to split, which increases the volume fraction of rubber in bitumen. This fact, in addition to the stirring and high temperature used during the production of the AR binder, may cause processes of re-polymerization and re-vulcanization, thus increasing the length of the polymer chains.

The rubber particles are constituted by polymer chains that can be compared to springs. When a spring is compressed or tensioned, it recovers its initial form, and the longer is the spring the higher is the recovered length. This is the reason why AR binders with the same rubber percentage, but constituted by rubber particles with lower ϕ_{eq} , present a higher elastic recovery.

Conversely, when very soft bitumens (e.g. bitumen M) are used, a decrease in the elastic recovery of the corresponding AR binders can occur due to a simultaneous decrease in the rubber percentage and ϕ_{eq} of the rubber particles. This means that irreversible phenomena of degradation and solubilization of the rubber particles in the recovered bitumen is occurring during the production of the AR.

This inflection point of the curve relating the rubber percentage with the elastic recovery can be used as a criterion to select base bitumens to be used in the production of AR binders with improved elastic recovery performance. The elastic recovery results may also be useful to determine the optimum temperature, interaction time, initial rubber content and dimensions among other production parameters. However, since all these parameters are co-dependent, they must be studied separately so as to understand their individual contribution to optimize the AR binder behavior.

4. CONCLUSIONS

Any study of bituminous materials constituents and behavior is a very complex task, since bitumen composition and performance vary significantly according to its origin and processing of the crude oil. Therefore, the results presented above may not be applicable to all paving grade bitumens. The main conclusions drawn from this study are the following:

- for paving grade bitumens usually applied to produce AR binders, the elastic recovery can be a very effective parameter to assess the changes in the rubber percentage and $\emptyset_{eq}$ during the AR production;
- although the rubber percentage and $\emptyset_{eq}$ are nearly constant when AR binder are produced with softer bitumens (with penetrations higher than 70), the elastic recovery is still increasing due to rubber splitting and re-polymerization or/and re-vulcanization;
- the elastic recovery decreases when very soft bitumens (e.g. bitumen M) are used, as well as the rubber percentage and $\emptyset_{eq}$, indicating an irreversible degradation of rubber;
- the maximum value of elastic recovery was observed for bitumen N (25% of 60/70 plus 75% of 150/200), which is the best base bitumen to use in order to obtain AR binders with improved elasticity;
- the elastic recovery results may be used to determine the best production parameters of AR binders with optimized performance.

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