ROAD MATERIALS AND PAVEMENT DESIGN

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Volume 7 – Issue 2/2006

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Volume 7 – Issue 2/2006

Road Materials and Pavement Design

Road Materials and Pavement Design an International Journal

Vol. 7 No 2/2006 – April-June

Road Materials and Pavement Design is indexed in:
Science Citations Index Expanded, ISI Alerting Services
Current Contents (Engineering Computing, and Technology)

11, rue Lavoisier – F-75008 Paris
ISSN 1468-0629
Head of publication: Patrick Fenouil

Lavoisier
14, rue de Provigny – F-94236 Cachan cedex
tel. : 33 (0)1 47 40 67 00
fax : 33 (0)1 47 40 67 02

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Road Materials and Pavement Design

an International Journal

Road Materials and Pavement Design

Contents

Volume 7 – No. 2/20(

Influence of Crumb Rubber and Digestion Time on the Asphalt Rubber Binders

Silvrano A. Dantas Neto* — Márcio M. Farias*
Jorge C. Pais — Paulo A.A. Pereira** — Jorge B. Sousa*****

** Post-Graduation Program in Geotechnics, University of Brasilia, SG 12, Asa Norte*
70910-900, Brasilia, Federal District, Brasil

silvrano@yahoo.com.br
muniz@unb.br

*** University of Minho, Department of civil engineering*
4800-058, P-Guimarães

jpais@civil.uminho.pt
ppereira@civil.uminho.pt

**** Consulpav*
Taguspark, Tecnologia I, nº 26, P-2780 Oeiras

jmbsousa@aol.com

ABSTRACT. *The behavior of asphalt binders modified with recycled crumb rubber depend several factors, such as: rubber content and type, temperature and time employed during digestion process. Some of these aspects are investigated in this paper by means of a seri tests performed on asphalt-rubber produced* via *the wet process. Crumb rubber manufact by ambient grinding and cryogenic processes were used in this work. Reductio penetration and sharp increase of viscosity, softening point and resilience were observe increasing rubber contents. The results show that the Brookfield viscosity limits the cr rubber content incorporated into straight binder, once it tends to become too high abo certain critical amount of incorporated rubber. The effect of digestion time on the viscosi the modified binders depended in this research on the rubber content. For high ru contents, there seems to exist a critical time after which viscosity tends to decrease continuing digestion process. A rubber-binder interaction model, which could explain results, is proposed. The influence of rubber type occurred especially in terms of Brook viscosity and softening point. It was observed a decrease in these properties for the asp rubber binders produced with crumb rubber obtained by cryogenic process in relatio those produced by the grinding process.*

KEYWORDS: *Asphalt-Rubber, Recycled Rubber, Asphalt Binders.*

1. Introduction

Research continues in the quest to find materials that could enhance the properties of asphalt mixes, thus reducing road maintenance costs. Addition of polymers has become common practice in several countries, with the main objective of improving flexibility and reducing thermal susceptibility of modified asphalt binders. However, that inevitably adds to the final cost of the product.

Besides performance improvements, there are increasing concerns about developing environment friendly products. In this context, there is growing interest in asphalts modified with crumb rubber recycled from used tires, also known as asphalt-rubber or rubber-asphalt, since this constitutes an important source of consumption for a huge mass of used tires produced annually.

The rubber used to produce tires comprises a mixture of different components, such as, styrene-butadiene rubber of high molecular weight, natural rubber, polymers, thermoplastic elastomers and carbon particles, among others (Horodecka *et al.*, 2000).The rich diversity of polymers enhances the properties of conventional asphalt binders, when crumb rubber is incorporated.

The behavior of modified asphalt-rubbers depends on several factors, such as, the origin, fabrication process and grain size distribution of the crumb rubber, the type of base conventional binder used in the mixture, and the temperature and time of the mixing process or digestion. Several authors have investigated the rheological and physical properties of binders modified with rubber, for rubber contents below 20% by weight (Anderson *et al.*, 2000).

In this work, a conventional base binder having a penetration grade of 50/70 was mixed with crumb rubber produced via milling at ambient temperature for different rubber contents and also with rubber obtained *via* cryogenic process for a rubber content of 21% by weight. The digestion temperature, which corresponds to the mixing temperature of crumb rubber and straight binder, was fixed at 190°C forvariable digestion times. The binder-rubber mixtures, produced using the wet process, were subjected to conventional tests, such as, penetration, softening point, Brookfield viscosity and resilience.

The objective of the present work was to investigate the main characteristics of crumb rubber that affect the physical properties of modified binders. The principal factors studied were the crumb rubber type and content incorporated to conventional asphalt binder.

2. Literature review

Rubbers used in the production of tires have different constituents, but the main component is vulcanized rubber. During the vulcanization process, rubber is heated in the presence of sulphur and activating and accelerating agents. This process

produces cross-links in the individual molecules of polymers, which confers to rubber a rigid three-dimensional structure. The strength of the rubber is proporti to the number of such links.

The constituents of passenger vehicle tires are shown in Figure 1:

a) Tread: tire part which is directly in contact with the pavement, its composi (rubber and special chemical agents) provides great wear resistance;

b) Belts: usually made from steel, the belts are used to reinforce the area ur the tread. These belts provide puncture resistance and help the tyre stay flat so th makes the best contact with the road;

c) Polyester Carcass: composed of nylon or polyester cords, provides mechan resistance to the tire;

d) Sidewall: The sidewall provides lateral stability for the tyre, protects the bc plies and helps keep the air from escaping;

e) The Bead Bundle: The bead is a loop of high-strength, steel cable coated w rubber. It gives the tire the strength it needs to stay seated on the wheel rim.

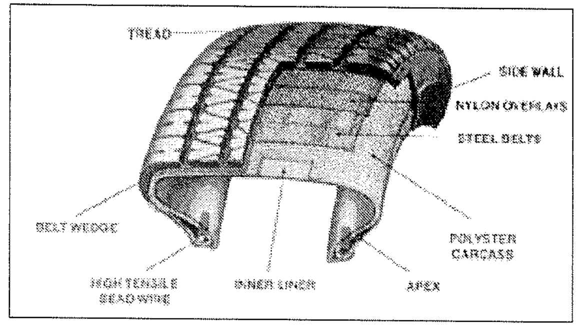


Figure 1. *Components of the tire*

A passenger vehicle tire is made approximately of 85% rubber, 15% steel fib and a polyester carcass. After its lifetime usage the rubber percentage in a tire still be about 83%, and the steel fiber quantities will be unaltered. On the other ha truck tires are composed of greater percentages of rubber, mainly, natural rubl These differences will affect the mechanical behavior of rubber asphalts produ from either truck or passenger vehicle tires.

Grinding or cryogenic processes can be used to produce the crumb rubber will be incorporated in the asphalt[A1] rubber. The grinding process is basic tearing and crushing the old tires at ambient temperature. A combination of grinc or granulators followed by sieves, transport conveyers and different kinds magnets are used to crush and extract the steel of the carcass. The grinding proc method has been widely adopted and is also more productive to obtain the crumb rubber The final product is generally an irregular particle, with high specific surface. When working with granulators, more regular particles with lower specific surface can be obtained.

The cryogenic process is carried at very low temperatures (-87°C to -198°C). In this case, the rubber of the tires is dipped into liquid nitrogen. At very low temperatures, the rubber becomes very brittle and it can be easily pulled apart on a press, into the desired particle dimensions. These particles of crumb rubber have lower specific surface than the ones obtained by the grinding process.

The use of binders modified with rubber started in the 1940's (Mohammad *et al.*, 2000). However, only in the 1960's the process of manufacturing asphalt-rubber known as wet process or McDonald process was developed and patented by Engineer Charles McDonald. From 1964, the Arizona Department of Transportation (ADOT) started using binders modified with crumb rubber in several maintenance projects and in the production of asphalt hot mixes (Way, 2000).

There are two processes for producing asphalt-rubber, known as the wet process and the dry process. In the wet process, a straight binder is initially preheated to around 190°C in a tank under hermetic conditions and then transported to a blending tank, where crumb rubber is added. The digestion process, which is the incorporation of rubber in the conventional binder, continues for a period of 1 to 4 hours, at a temperature of 190°C. the process is facilitated by a mechanical agitation produced by a horizontal shaft (Visser *et al.*, 2000).

In the dry process, particles of crumb rubber are added to preheated mineral aggregates before the addition of the straight bituminous binder (Visser *et al.*, 2000). Aggregates are heated to temperatures of approximately 200°C, then crumb rubber is added and mixed for about 15 seconds until a homogeneous mixture is obtained. Straight binder is then added in a conventional mixing plant.

In the dry process, modified mixes rather than modified binders are produced, since there is no digestion of the rubber by the conventional binder. The time of contact between the rubber and the binder in the dry process is relatively short and not enough to produce all necessary reactions between the two materials (Horodecka *et al.*, 2000; Oliver, 2000). The dry process does not fit the conventional definition of asphalt-rubber binders, and the product would be more appropriately described as aggregate-rubber.

Physical properties of asphalt binders are generally expressed in terms of penetration grade, softening point, thermal susceptibility, viscosity and ductility, among others. Previous studies with asphalt-rubbers, incorporating up to 20% of crumb rubber by weight, have demonstrated that there is an increase in viscosity in comparison with conventional binders (Anderson *et al.*, 2000). Other studies (Sebaaly *et al.*, 2000) show that the incorporation of rubber into asphalt binders enhances their viscoelastic properties.

3. Materials and methods

3.1. *Basic products and asphalt-rubber binders*

Crumb rubber, recycled from unserviceable tires using the ambient grinding the cryogenic processes were used in this study. The following grain sizes of cru rubber modifiers were used:

– CRM 1: particle diameter between 0.5 – 2.0 mm from ambient grinding proce

– CRM 2: particle diameter between 0.5 – 2.0 mm from cryogenic process.

Figure 2 shows the grain size distribution curves for the two rubber ty described in Table 1. The grade envelope, prescribed by ADOT for crumb rubbe be used in the production of asphalt-rubber, is also shown in the Figure 2.

Table 1. *Grain size distributions*

Sieve size		% Passing			
inch	Mm	ADOT		CRM 1	CRM 2
N° 4	4.75	100	100	100	100
N° 8	2.36	100	100	100	100
N° 10	2.00	100	100	97.4	97.3
N° 16	1.18	65	100	51.9	49.7
N° 30	0.60	20	100	20.0	24.6
N° 50	0.30	0	45	7.9	4.1
N°200	0.075	0	5	0	0

Base asphalt with penetration grade of 50/70 was used to mix with the cru rubbers previously described. Table 2 presents the results of standard characteriza tests preformed for the conventional binder.

Table 2. *Characterization of the conventional binder[A2]*

Physical Properties	AC 50/70
Penetration, ASTM D 5-95 (1/10 mm)	52,0
Softening point, ASTM D36-97 (°C)	50,6
Brookfield viscosity at 175°C, ASTM D 4402-87 (cP)	87,5
Resilience, ASTM D5329 (%)	14,0

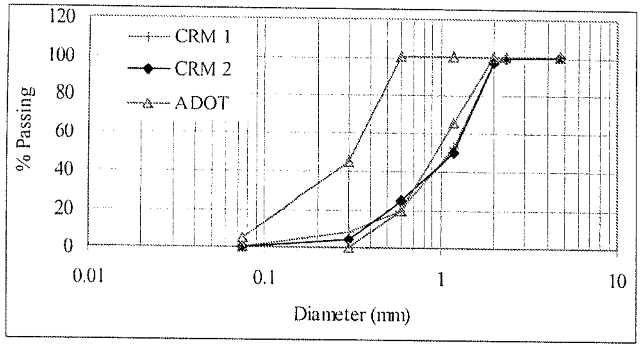


Figure 2. *Grain size distribution curves for CRM 1 and CRM 2*

The following straight binder and crumb rubber combinations were used to produce asphalt rubber binder using the wet process:

- Combination C1: binder AC 50/70 + CRM 1;
- Combination C2: binder AC 50/70 + CRM 2;

For the combination C1, the following combinations of production variables were also investigated:

- Rubber content (% of total weight): 10, 15, 17, 19, 21, 23, 25 and [A3] 30;
- Digestion time (minutes): 15, 30, 45, 60, 120, 180, 240 and 300;
- Temperature of digestion (°C): 190.

The influence of rubber type (cryogenic or grinding process) was investigated by comparing asphalt rubber combinations C1 and C2 for a crumb rubber content of 21% by weight and digestion temperature of 190°C. The samples obtained from combination C2 were collected for the same digestion times described before for combination C1

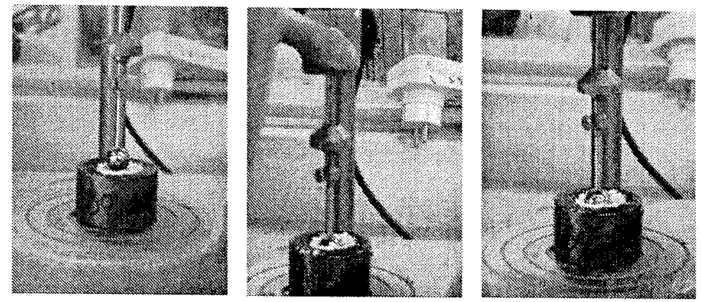
3.2. *Testing methods*

The following tests were performed to study the physical properties of the asphalt-rubbers produced for this research:

- Penetration (ASTM D5);
- Softening point (ASTM D36);
- Resilience (ASTM D5329);
- Apparent Brookfield viscosity (ASTM D2196).

The tests described above were used to obtain the physical properti modified binders under high and low temperatures. The resilience test, standar by ASTM D5329, measures the elastic recovery of samples of modified bi under compression.

The resilience test (ASTM D5329) consists of applying a total displaceme 10 mm to a binder sample at a temperature of 25°C and then releasing the lo obtain the elastic rebound. The sample is compressed at a rate of 1 mm/s by n of a rod that acts on a metallic sphere (17 mm diameter). Then compressi released and the elastic recovery that takes place after 20 s is measured expressed a percentage of the initial displacement (10 mm). Figure 3 illustrate test sequence from the unloaded stage (a), then maximum compression (b) finally the elastic recovery (c).



(a) (b) (c)

Figure 3. *Resilience test (ASTM D5329)*

Brookfield viscosity test seems to be the most convenient to define the properties of binders modified with rubber under high temperatures, since impossible to measure asphalt-rubber viscosity with most conventional visc apparatus currently available due to presence of particles of rubber. Brook viscosity is obtained by applying a torque with a spindle of fixed dimensions, w is immersed in the binder sample at a desired temperature. The standard AS 6114/97 fixes the specific conditions for Brookfield viscosity tests on asphalt-ru samples.

A Brookfield viscometer model DV-II+, shown in Figure 4, was used in research. A spindle number 27 and a rotational speed of 25 rpm were adopted.

The specified configurations used in this research were designed to invest each important variable that may interfere in the properties of the produced asp rubbers. Figure 5 shows the equipments used in the production of asphalt-ru binders. These comprise an oven, equipped with temperature control (Figure and an assembly of engine and paddle (Figure 5b) that facilitates blending o conventional binder and the crumb rubber. The paddle velocity was chosen in order to produce a homogeneous mixture and its values ranged from 250 to 300rpm. The amount of asphalt rubber binder produced in each batch is about 3kg.

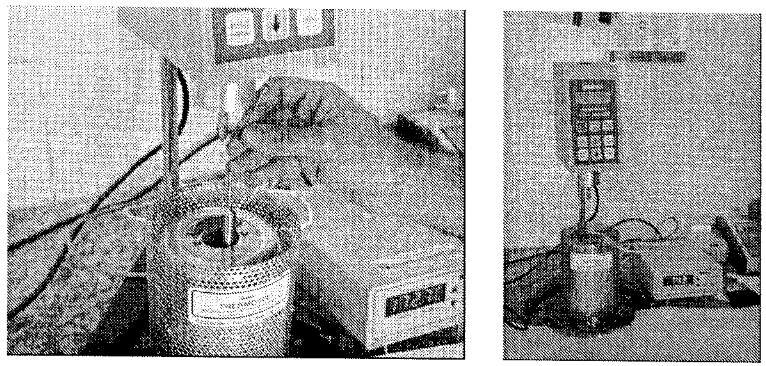


Figure 4. *Brookfield viscosimeter apparatus*

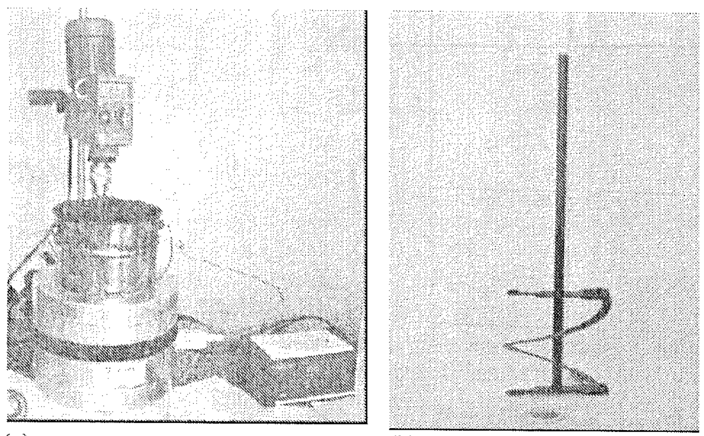


Figure 5. *Equipment used for the production of asphalt-rubber binders*

Table 3 presents the target values specified in ASTM D6114-97 standard for asphalt binders modified with crumb rubber recycled from used tires.

Table 3. *Target physical properties of asphalt-rubber (ASTM D6114-97)*

Physical Property	Unit	Range
Apparent Brookfield viscosity, 175°C (ASTM D2196)	cP	1500 - 5(
Penetration, 25°C, 100g, 5s (ASTM D5)	mm/10	25-75
Softening point (ASTM D36)	°C	> 57,2
Resilience, 25°C (ASTM D5329)	%	> 25

4. Results and discussion

4.1. *Influence of the rubber content in the physical properties of asphalt-rubl*

In order to investigate the influence of the rubber content on the ph properties of asphalt-rubber, the results of tests performed with combination C be discussed. Figures 6 to 10 show the results of penetration, Brookfield visc softening point and resilience for asphalt-rubber C_1 produced for different dig times and rubber contents at a digestion temperature of 190°C. In the same fi the results for samples of straight binder (AC 50/70), collected at different tim also shown for comparison.

The test results with the straight binder show a gradual decrease in the val penetration and an increase in softening point for samples collected at di digestion times at the same digestion temperature (190°C), as observed in Fig and 7, respectively. These results indicate that the aging of the conventional l is taking place due to the volatilization of light fractions. The evaporation of light components, controlled by the temperature, is one of the primary mecha of aging in asphalt binders used (Epps, 1997).

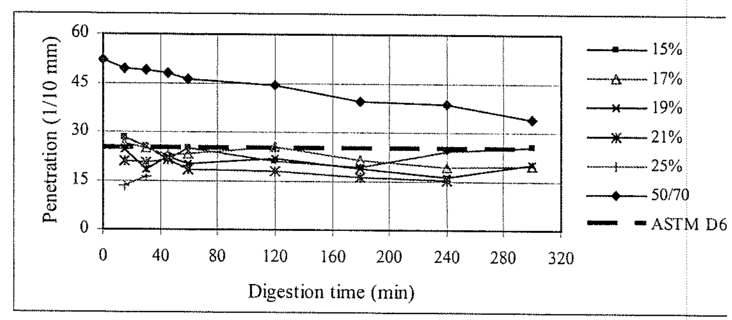


Figure 6. *Penetration of samples of asphalt-rubber combination C_1.*

According to results shown in Figure 6, the penetration grade of asphalt-rubber binders decreases with the increase of rubber content. For all rubber percentages and production times investigated in this research, the lower limit penetration of 25 (0.1mm) suggested by ASTM D6114 was not satisfied. However, it was always possible to meet all other recommendation for appropriate combinations of rubber content and process time. This might suggest either that penetration is not the most appropriate test for this kind of standard or that a more complacent lower limit (say 20 0.1mm) should be adopted.

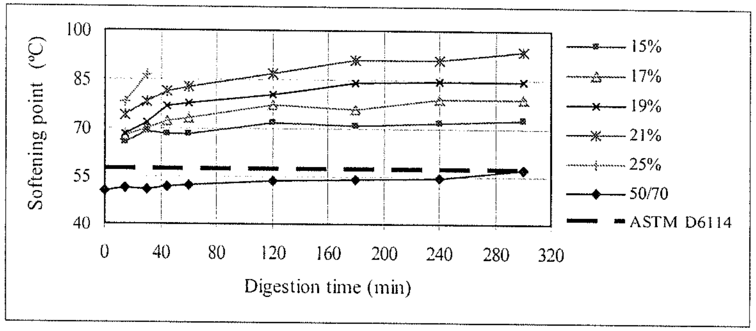


Figure 7. *Softening point of samples of asphalt-rubber combination C_1.*

The results of the penetration test do not allow definitive conclusions of the influence of digestion time on this property. The results present a significant scattering and are highly dependent on the superficial distribution of crumb rubber grains in the tested samples. That again suggests that penetration tests are not the most appropriate for determining the consistency of asphalt-rubber binders. If it should be used, perhaps the number of determinations should be increased to allow for the computation of more representative mean values.

The results of the Brookfield viscosity tests are presented in Figures 8 and 9. The viscosity of the resulting asphalt-rubber binder is controlled by several factors, related to the base materials and mixture process. The factors related to base materials are: the origin, type and composition of the base asphalt; the origin, type and composition of the crumb rubber; the grain size distribution of the crumb rubber. Variables related to the mixture process include: relative proportion of rubber and base asphalt; mixture time and mixture temperature. Therefore, guidelines should be set so that producers of asphalt-rubber can choose the appropriate base materials, find the proper dosage between the ingredients, and specify the appropriate process of mixing.

Regarding the rubber content and the mixing time, the results of Figures 8 and 9 indicate the following:

– For a fixed mixing time, viscosity increases steadily and at faster rates increasing percentages of crumb rubber. For the materials and conditions use this research, and for the usual process time of 45-60 minutes, the maximum lim 5000 cP suggested by ASTM D6114 was achieved for about 19% of crumb rubb

– For a fixed percentage of crumb rubber, the mix viscosity increases increasing mixing time, but at decreasing rates. For higher percentages of cr rubber, there is a limit for this viscosity gain, which is followed by viscosity For the condition of this research, viscosity loss was observed for mixing time a 120 minutes and crumb rubber content above 19%.

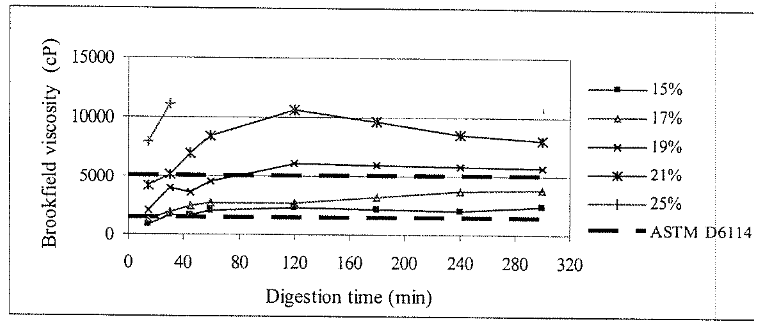


Figure 8. *Brookfield viscosity at 190°C of samples of asphalt-rubber combination*

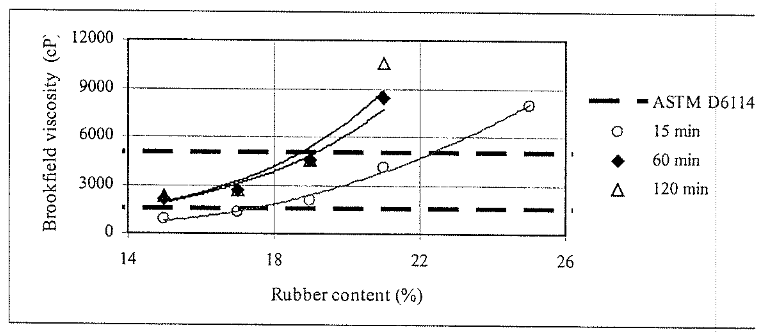


Figure 9. *Brookfield viscosity at 190°C of samples of asphalt-rubber combinatio C_1 for digestion times of 15, 60 and 120 minutes*

The gain in viscosity with the addition of crumb rubber possibly includ physical and a chemical component. The physical component refers to the visc gain that results from the inclusion of any particles, as presented in (Specht *et* 2003), similar to what happens to asphalt mastic with the introduction of inert particles or cement filler. This process is possibly dominant at early stages of the asphalt-rubber mixing process.

Then some kind of chemical process takes over in terms of importance. A possible model of chemical interaction was proposed in Holleran *et al.* (2000), which suggests that the asphaltenes and light fractions of the conventional asphalt binder and the rubber particles interact to form a gel-coated particle. The rubber particles would swell in a process similar to what occurs in polymer-asphalt systems. That paper also suggests that this system is not thermodynamically stable and leads to significant change of properties over time.

Takallou *et al.* (2003) argues that the rubber acts exclusively as an additive, rather than a modifier. He also contests the idea of particle swelling or any kind of permanent reaction and states that the process is reversible, although the work does not clearly described how this is accomplished. However, these conclusions are partially based on results from asphalt-rubbers obtained with the dry process, in which mixing time is short and it is still possible to separate the phases physically.

A recent study, using binders obtained *via* the wet process, shows that physical particle addition alone is not enough to explain the viscosity gain in samples of asphalt rubber that reacted for sufficient time (Specht *et al.*, 2003). Therefore, irrespective of particle swell or not, it seems that some reaction takes place, even though this does not rule out the possibility that this reaction might be reversible as claimed in Takallou *et al.* (2003).

The authors here favor a binder-rubber interaction model, which includes physical, chemical and thermal components. The chemical component should include the absorption (even if temporary) of light fractions from the base asphalt matrix by the rubber particles. This explains the increase of viscosity of the binder matrix for increasing mixing time, until a kind of saturation point. The thermal component involves some kind of softening of the rubber particles, possibly due to devulcanization. This would explain the loss of viscosity after reaching a peak for increasing time or temperature of exposure of the asphalt-rubber mix to heat.

The results of softening point tests, presented in Figure 7, show an increase in softening point with the increase of rubber content. There is a sharp initial elevation of the softening point and this increase up to about 120 minutes of digestion time. It is believed that this increase of softening point is related to absorption of light fractions from the asphalt matrix by the rubber particles. After some time the increase of softening point for all AR binders show approximately the same tendency as that of the conventional straight binder. This suggests that the later steady increase of softening point with time (now at a much lower rate) is related to the loss of light oils in the base asphalt matrix.

All the results in Figure 7 easily satisfy the recommendations of ASTM D6114 with respect to the minimum value of softening point equal to 57.2°C.

The results of resilience tests for the AR and conventional binders are show Figure 10. Resilience increases for increasing amount of incorporated crumb ru and all values easily satisfy the lower limit of 25% suggested by ASTM D6 Most of the resilience gain takes place at the initial stages, and then resili increases slightly and may be followed by some decrease as in the viscosity res These observations suggest that the physical presence of the rubber particles ha major impact in resilience as measured according to ASTM D5329.

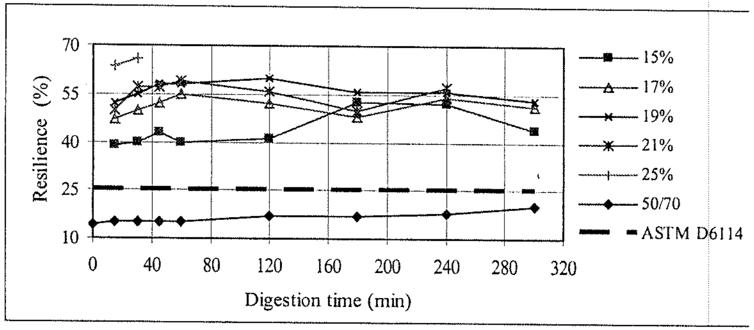


Figure 10. *Resilience of samples of asphalt-rubber combination C_1*

The rubber particles have high elastic recovery and that reflects immedi after addition of the rubber into the base asphalt. This impact is proportional tc amount of rubber incorporated. As mixing time progresses, the smaller resili gain can be credited to the reaction of light fractions with the rubber grains and to the loss of these fractions as can be observed for the neat asphalt. Eventual lo resilience, as well as viscosity, is credited to the softening of the rubber grain explained in the model previously proposed. This is clear from the result Figure 10, after 300 minutes of mixing time.

4.2. *Influence of the type of rubber in the physical properties of asphalt-rubbe*

Figures 11 to 14 present the results of physical properties of asphalt-ru obtained from the blending of conventional binder AC 50/70 and crumb rub produced according to different processes: rubber CRM 1 produced with amb grinding and rubber CRM 2, produced with the cryogenic process. A percen of 21% of rubber by weight was incorporated into the conventional binder and mixture produced at a digestion temperature of 190°C.

The results in Figures 11 and 12 show that the use of crumb rubbers obta with the cryogenic process produced modified asphalt-rubbers with lc Brookfield viscosity and lower softening points than those produced by grin process. No significant change of penetration or resilience was observed, irrespective of the process of fabrication of the crumb rubber used in the asphalt-rubber (Figures 13 and 14).

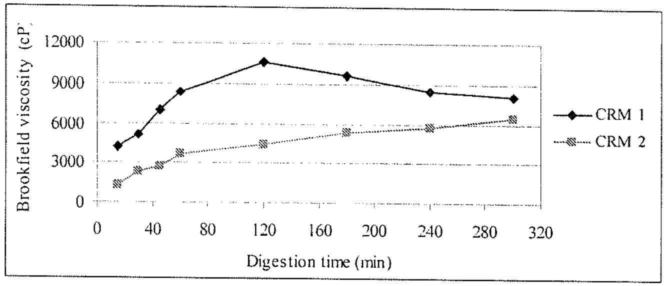


Figure 11. *Brookfield viscosity of samples of asphalt-rubber combination C_2*

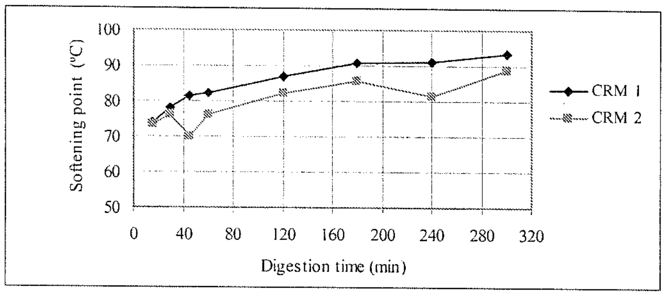


Figure 12. *Softening point of samples of asphalt-rubber combination C_2*

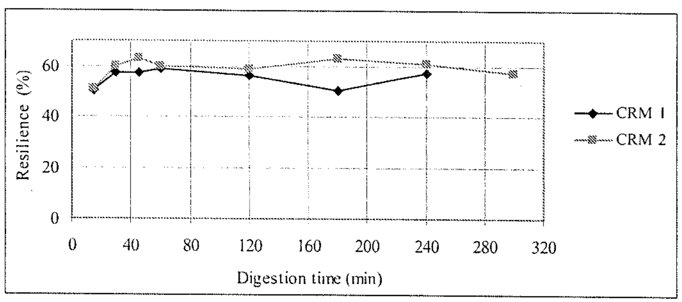


Figure 13. *Resilience of samples of asphalt-rubber combination C_2*

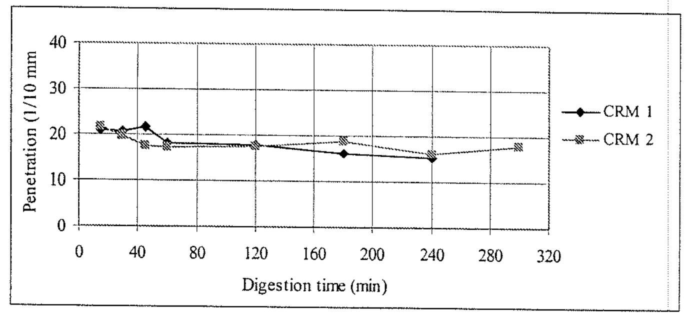


Figure 14. *Penetration of samples of asphalt-rubber combination C_2*

The cryogenic process produces crumb rubber with a smaller specific su and grains with a well-defined and regular shape, in opposition to irregular g produced in the ambient grinding process, as showed in Figure 15. The effect o crumb rubber production process (cryogenic or ambient grinding) on the part texture can be noticed at simple touch, when manipulating the rubber gr Ambient grinded rubber grains have a smooth surface, due to the tearing process the other hand, cryogenic have sharp angular edges like glass cubes, due tc breaking process of nitrogen frozen tires.

The reduction of specific surface of the regular grains of crumb rubber, obta with the cryogenic process, diminishes their capacity to interact with conventional binder. Therefore the absorption of light fractions by the cryog rubber grains is less intense, thus resulting in AR binders with lower Brook viscosity and softening points when compared to those prepared using rubber g ground at ambient temperature.

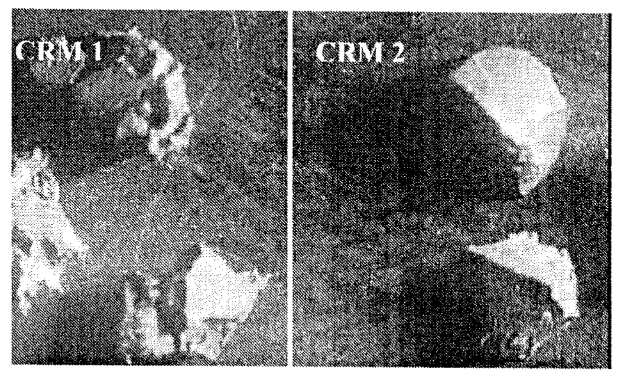


Figure 15. *Form of the grains of crumb rubber obtained by the grinding prc (CRM 1) and cryogenic process (CRM 2)*

The possible effect of grain morphology on the specific surface can be even more appreciated when the grain size distribution curves of CRM 1 and CRM 2 are compared, as shown in Figure 2. Despite having the same grain size distribution, the results of viscosity tests suggest a higher specific surface for the crumb rubber obtained *via* ambient grinding when compared to the cryogenic process.

As high viscosity is a limiting factor regarding binder workability, the use of cryogenic rubber might be useful to obtain asphalt rubbers with suitable viscosity while still incorporating a high amount of crumb rubber. However, potential environmental benefits with the incorporation of higher rubber contents should be weighted against eventual cost increase of more expensive cryogenic rubbers, supposing that both binders will result in asphalt mixes with suitable mechanical properties.

5. Conclusions

The physical properties of asphalt-rubber binders are controlled by several factors, related to the base materials and mixture process. Therefore, guidelines should be set so that producers of asphalt-rubber can choose the appropriate base materials, find the proper dosage between the ingredients, and specify the appropriate process of mixing.

Brookfield viscosity is the determining physical property of asphalt rubber binders, since they set a practical limit of workability. The limits for other properties suggested in ASTM D6114 appear too harsh for penetration tests and too easy to accomplish in softening point and resilience tests.

Higher contents of crumb rubber incorporated into the conventional binder leads to the production of modified asphalt-rubbers with lower penetration grade, higher Brookfield viscosity, higher softening point and higher resilience for all digestion times investigated.

Penetration tests presented significant scattering and are highly dependent on the superficial distribution of crumb rubber grains in the tested samples, which suggests that these tests are not the most appropriate for determining the consistency of asphalt-rubber binders. If they must be used, perhaps the number of determinations should be increased to allow for the computation of more representative mean values.

Results of Brookfield viscosity tests show that, for a fixed mixing time, viscosity increases steadily and at faster rates with increasing percentages of crumb rubber in the mix. For a fixed percentage of crumb rubber, the mix viscosity also increases, but at decreasing rates, with increasing mixing time. For higher percentages of crumb rubber, there is a limit for this viscosity gain, which is followed by viscosity loss.

The gain and later loss of viscosity possibly includes physical, chemica thermal causes. The physical effect of addition of crumb rubber particles is pronounced in the early mixing stages. This is followed by gain in viscosity c the absorption of light fractions from the base asphalt mixes by the rubber grai the process of heat transfer is continued further, then the rubber particles soften, thus resulting in viscosity reduction.

The use of crumb rubber manufactured with the cryogenic process fo production of asphalt-rubber generates a mixture with lower viscosity and softening point, if compared to asphalt-rubber that incorporate rubbers proc with the grinding process at ambient temperature. This is due to the morpholo cryogenic rubber grains, which result in a smaller specific surface. Other prope such as penetration and resilience, were not affected.

Acknowledgements

This work is part of the doctor thesis developed at Pos-graduate Progra Geotechnics of the University of Brasilia and with support of the Departme Civil Engineering of the University of Minho, in Portugal. It is supported grants from CAPES (Coordenação de Aperfeiçoamento de Pessoal de Superior) and CNPQ (Conselho Nacional de Desenvolvimento Científi Tecnológico). The authors are also thankful to the companies that supplie materials used in this work, among them Cepsa (supplier of the binders), Bi and Recipneu (suppliers of the granulated rubber).

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Received: 3 March 2004
Accepted: 15 December 2005