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# The effect of prolonged storage time on asphalt rubber binder properties

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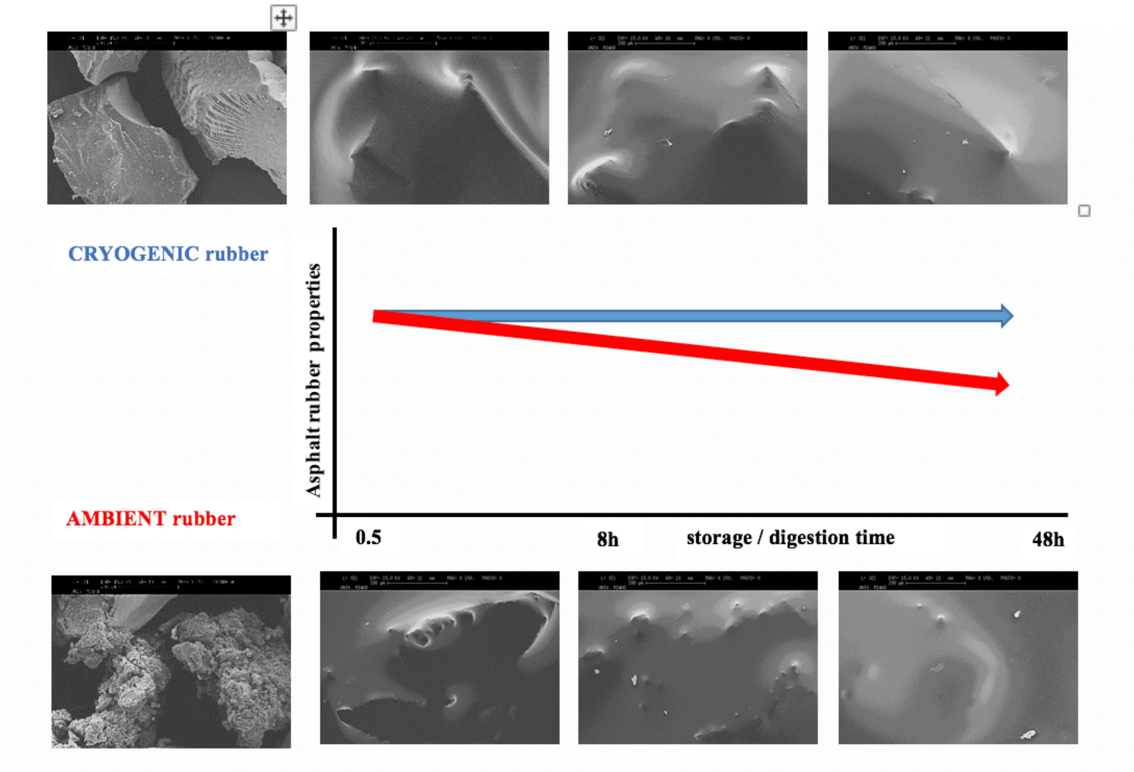
## HIGHLIGHTS

- We study some asphalt rubbers with different crumb rubber types and base asphalts.
- We assess the asphalt rubbers performance-related characteristics.
- We analyse the asphalt rubber surface through scanning electron microscopy images.
- We assess the asphalt rubbers mechanical performance.
- We conclude about the effect of prolonged digestion time of asphalt rubber performance.

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# The effect of prolonged storage time on asphalt rubber binder properties

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# The effect of prolonged storage time on asphalt rubber binder properties


## Abstract

This study wants to provide fundamental understanding of prolonged storage time on asphalt rubber binder properties by performing an investigation on the variation of conventional properties, rheology and morphology of four asphalt rubbers maintained at 180C in low shear for different digestion/storage times up to 48 hours. The analysed asphalt rubbers were manufactured by combining two different asphalt binder grades, pen 35/50 and pen 50/70, with both cryogenic and ambient crumb rubber. Results have shown that keeping asphalt rubber agitated at the above mentioned processing conditions, up to 48 hours, is significantly detrimental when an ambient crumb rubber is used, while it seems not to negatively affect the asphalt rubber produced with cryogenic rubber crumbs. Instead, no remarkable change is recorded when asphalt rubbers are produced with the same rubber type and by changing the base asphalt binders, namely pen 35/50 and pen 50/70.



**Keywords:** asphalt rubber; digestion time; storage stability, rheology; scanning electron microscopy; crumb rubber


## 1. Introduction

Over the last decades, the use of crumb rubber from scrap tires in hot asphalt mixes has become a frequent practice in road construction. The use of asphalt rubber has advantages such as the development of environmental friendly products (Dantas Neto et al., 2006), improvement of mechanical performance of asphalt paving mixes (Minhoto et al., 2005, Minhoto et al., 2008, Moreno et al., 2011; González et al., 2012), reduced ageing of the asphalt mixtures (Lo Presti 2013), lower maintenance and conservation costs (Jung & Way , 2002; Sousa et al., 2001; Kirk & Holleran, 2000), reduction of the noise level (McNerney et al., 2000), more safety guarantees due to long-term colour contrast for pavement markings because rubber acts as a pigment (López et al., 2008) and reduction of the effect of water projection during the rainy season (Fontes et al., 2010). Beyond that, it was proved that the use of crumb rubber as a modifier improves the penetration index, low temperature ductility and temperature susceptibility of the asphalt rubber (Xiang et al., 2009; Mashaan & Karim, 2013).

There are two methods to obtain crumb rubber from ground used tires: ambient grinding and cryogenic grinding process. In the ambient grinding process, scrap tires are grinding at or above ambient temperature. The particles obtained with this process have an irregular structure with different shapes and high specific surface area. The other method to obtain the crumb rubber is through cryogenic grinding where liquid nitrogen is used to freeze the recycled tire rubber (usually between -87ºC and -198ºC) until it becomes brittle. Then, it is reduced to smooth and regular particles with lower surface area than those obtained by ambient grinding (Neto et al., 2006; Lo Presti, 2013).

The incorporation of the rubber into asphalt mixes can be accomplished by two processes: dry or wet process. In the dry process the crumb rubber is usually added to the aggregate prior to the addition of the asphalt. Thus, 1 to 3% of the aggregate in the asphalt mix is replaced by crumb rubber (Caltrans, 2005). In the wet process, the crumb rubber is blended with the asphalt to produce a crumb rubber modified asphalt, usually named asphalt rubber, that is then mixed with the aggregates (Moreno et al., 2011; Mitchell et al., 2009; Bahia & Davies, 1994). In this process, the asphalt is preheated to a temperature of 176ºC – 226ºC in a tank under hermetic conditions where the crumb rubber is added. The resulted blend is kept at elevated temperatures (150 to 218ºC) for a designed period of typically 45 to 60 minutes to allow an interaction between the rubber and the asphalt (Caltrans, 2005). Related to the effectiveness of the two modification processes, some researchers indicated that the wet process is more effective than dry process as the wet process mixes are more consistent and have better performances than the dry process mixes (Volle, 2000; Hunt, 2002). One of the reasons that may indicate the better performance of wet process mixes is that in the dry method the interaction between the asphalt and the crumb rubber is less than in the mixes produced by the wet process where the interaction between those two components is complete before the mixing with the aggregates.

Digestion time or reaction time is the expression used to describe the time needed to promote the interaction between the binder and the modifier agent (rubber in this case) when mixed at high temperatures (Caltrans, 2003).

During digestion process the asphalt rubber swells because rubber absorbs the light fractions of asphalt (Peralta et al., 2010; Mitchell et al., 2009, Subhy et.al, 2015, Lo Presti et al. 2014). The swelling process is one of the key factors to successfully prepare the asphalt rubber and continues usually for 1-4 hours (Thives et al., 2013). It was proved that the swelling increases rapidly in the beginning and then stabilizes, depending on temperature (Dong et al., 2012). This phenomenon allows the increase of the rubber particles, which leads to a reduction of the distance between particles, increasing the viscosity and stiffness of asphalt rubber in comparison with conventional binders (Hicks & Epps, 2000; Anderson et al., 2000). According to Lo Presti (2013), Peralta et al. (2010) and Nejad et al. (2012), if the mixing time is too long or mixing temperature is too high the swell is replaced by depolymerisation/devulcanization which causes dispersion of the rubber into the asphalt and consequently reduction of viscosity.

There are several factors affecting the interaction between asphalt and crumb rubber. On the part of the asphalt mix components, the interaction is affected by the chemical and physical characteristics of the crumb rubber and the asphalt, the percentage of crumb rubber added to the mix, its size and grinding method and the physical and chemical properties of the asphalt. The processing conditions such as, shear strength, temperature and mixing time are the external factors affecting the interaction (Nejad et al., 2012; Jeong et al., 2010; Moreno et al., 2011; Cong et al., 2013, Lo Presti & Airey, 2013). Several studies proved that the increase of temperature and digestion time improves the asphalt rubber properties to periods up to 2 hours and in the temperature range of 160-200 ºC (Lee et al., 2008; Neto et al., 2003).

Some of the main parameters used to study the asphalt rubber behaviour are penetration index, which allows to indirectly evaluate the stiffness of the asphalt rubber at 25 ºC; the softening point with the ring and ball method to evaluate the deformability of the asphalt rubber or its performance to elevated temperature; resilience to evaluate the elastic recovery of the asphalt rubber; and rotational viscosity that allows to understand the flow resistance and the coating ability of the asphalt rubber. Several studies evaluated the addition of crumb rubber in the asphalt using these tests, especially high service temperature viscosity due to its influence on mixture compaction and asphalt rubber workability during storage and pumping process (Wang et al., 2012). Recently, Thives et al. (2013) used the Scanning Electron Microscopy to study the interaction between the asphalt and the rubber in some asphalt rubbers to verify the homogeneity of the mix, proving that this technique can also be used to define the digestion time of the asphalt rubber.

The asphalt rubber has been studied using rheological studies that allow knowing parameters such as shear complex modulus, G*, used to evaluate the asphalt resistance to deformation when exposed to repeated shear strain and phase angle, δ, which gives indications about the viscous and elastic properties of the asphalt rubber (AASHTO, 1998). Beyond that, the results of rheology include storage shear modulus, G’, and loss shear modulus, G’’. G’ is the shear complex modulus multiplied by the cosine of the phase angle expressed in degrees and represents the energy stored during a loading cycle. G’’ is the shear complex modulus multiplied by the sine of the phase angle expressed in degrees which represents the component of the complex modulus that is a measure of the dissipated energy during a loading cycle (AASHTO, 1998). With the results obtained in rheology tests it is possible to determine parameters such as $G^*/\sin(\delta)$, a measure of permanent deformation resistance which means that a higher value of $G^*/\sin(\delta)$ means better resistance. Beyond that, $G^* \times \sin(\delta)$ is related to fatigue response (SHRP, 1994).

The use of asphalt rubber in the wet process requires specialised equipment that allows continuous agitation of the material. Another option could be to produce the asphalt rubber and immediately send it to the mixer, so to avoid issues linked with the poor storage stability of the material. In both cases, currently one of the major points of debate is how long can we hold asphalt rubber in a digestion tank? Also, is the effect similar if we use cryogenic crumb rubber rather than a more conventional ambient crumb rubber? Sometimes there is equipment breakdown, and contractors do not wish to discard a large quantity of asphalt rubber or asphalt rubber mix. 48 hours seems to be the preferred solution from contractors, thus, the objective of this paper is to realize the effect of long digestion time (up to 48 hours) in the performance of the asphalt rubber manufactured with both ambient and cryogenic crumbs, assessing its mechanical behaviour, performance related behaviour and internal structure.

## 2. Objective

This work studied four different asphalt rubbers produced with a digestion time up to 48 hours. The asphalt rubbers were composed with two conventional asphalt binders (35/50 and 50/70 pen asphalt) mixed with two crumb rubbers, an ambient crumb rubber and a cryogenic crumb rubber. The behaviour of the asphalt rubbers was studied by the evolution of rheological properties over time as well as their morphology through Scanning Electron Microscopy. More conventional characterisation, namely penetration, softening point, resilience and viscosity, was also carried out. The study of the effect of long digestion times was undertaken for 5 samples of each asphalt rubber corresponding to 30 minutes, 8, 24 and 48 hours of digestion time.

## 3. Materials and Methodology

The materials used in this work include two conventional asphalt binders: an hard A35/50 with a penetration grade of 35/50 mm/10 and a soft A50/70, with a penetration grade of 50/70 mm/10. These two asphalt binders allow to produce asphalt rubbers with different interactions with the crumb rubber because hard bitumen has lesser interaction with crumb rubber compared to soft bitumen. The penetration and softening point for these asphalt binders are presented in Table 1.

Table 1. Properties of conventional asphalts.

| Properties | Standard | A35/50 | A50/70 |
|---|---|---|---|
| Penetration at 25 ºC, 100 g, 5 s (0.1 mm) | EN 1426 | 42 | 61 |
| Softening Point (ºC) | EN 1427 | 54 | 51 |

For each asphalt binder, the evolution of shear complex modulus (G*) and phase angle (δ) with temperature is presented in Figure 1 and Figure 2, respectively. These figures allow to verify that in fact asphalt binder A35/50 is stiffer than A50/70 and presents a lower phase angle. These differences are more evident for low temperatures up to 60ºC.

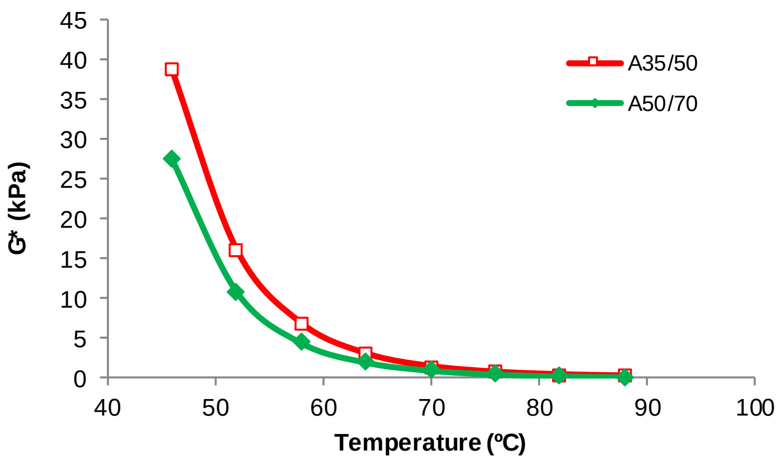


Figure 1. Evolution of G* with temperature for asphalt binders A35/50 and A50/70

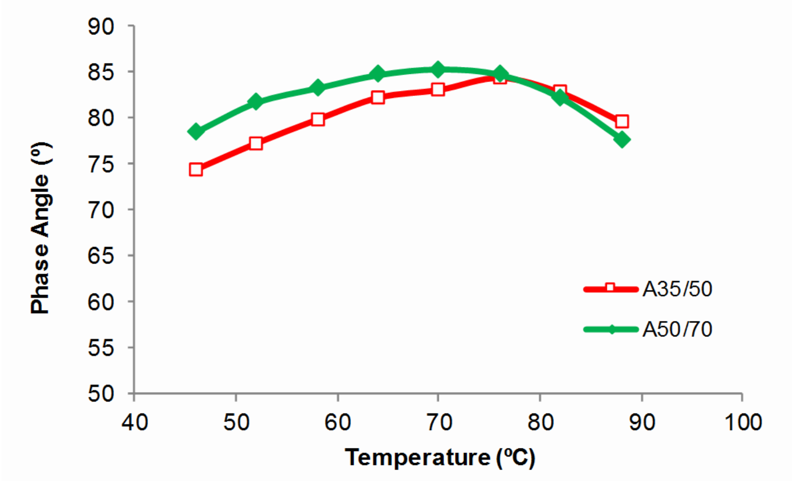


Figure 2. Evolution of phase angle with temperature for asphalt binders A35/50 and A50/70.

Two different crumb rubbers were used in this work: namely a Cryogenic grinding crumb Rubber (RC) and an Ambient grinding crumb Rubber (RA). These crumb rubbers presented the similar gradation, with a maximum dimension of 0.8 mm, but differences in the particles morphology, as shown in Figure 3 and Figure 4 where scanning electron microscopy images of the two crumb rubbers used in this work are presented, allowing to see the morphology and shape of the rubber particles. The particles of rubber were magnified 22x, 150x and 700x. Physically, the main difference between the crumb rubbers obtained using the cryogenic and ambient grinding process is the morphology of the resulting particles. The particles obtained with ambient process generally have a porous or fluffy appearance. On the other hand, the surface of the particles of crumb rubber obtained from the cryogenic process are glasslike; therefore, it has a rather low surface area compared to ambient crumb rubber with a similar gradation.

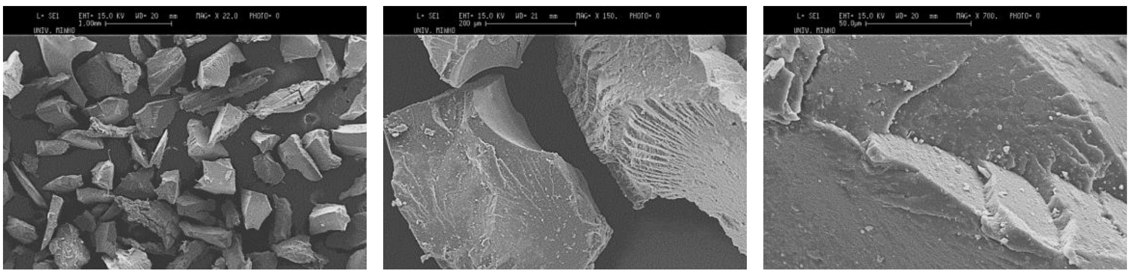

Figure 3. Scanning electron microscopy images of cryogenic crumb rubber: 22x (left), 150x (center) and 700x (right).

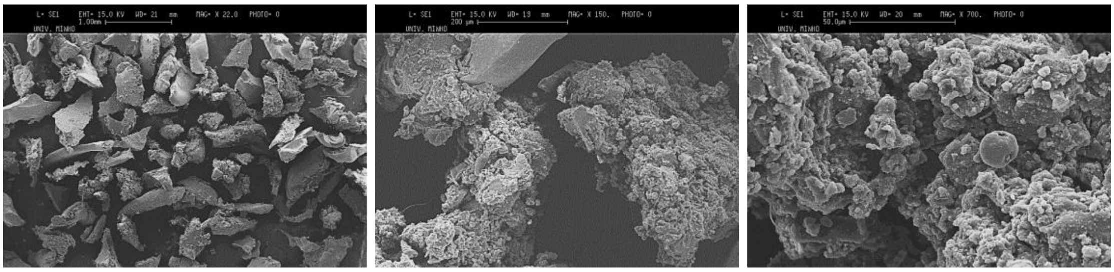

Figure 4. Scanning electron microscopy images of ambient crumb rubber: 22x (left), 150x (center) and 700x (right).

The asphalt rubber production was carried out in laboratory in a small equipment composed by a helix and an engine that facilitates blending of the asphalt binder and the crumb rubber. The helix velocity was chosen in order to produce a homogeneous mixture and ranged from 250 to 300 rpm.

With the two asphalt binders and the two crumb rubbers, four different modified asphalt rubbers were produced as follows:

(1) Asphalt rubber A35/50RC: asphalt rubber with asphalt A35/50 and rubber RC;
(2) Asphalt rubber A35/50RA: asphalt rubber with asphalt A35/50 and rubber RA;
(3) Asphalt rubber A50/70RC: asphalt rubber with asphalt A50/70 and rubber RC;
(4) Asphalt rubber A50/70RA: asphalt rubber with asphalt A50/70 and rubber RA.

All asphalt rubbers used in this work were produced with a crumb rubber content of 19% by mass of total asphalt rubber, digestion temperature of 180 ºC and digestion time up to 48 hours. Over the 48 hours of digestion time, samples were collected at 30 minutes, 8, 24, 30 and 48 hours.

The evolution of the asphalt rubber's properties with the digestion time was studied through the following testing program:

(1) Morphology by means of Scanning Electron Microscopy (SEM) to identify the homogeneity of the asphalt rubber;
(2) Rheological tests to assess the evolution of the shear complex modulus and phase angle;
(3) Conventional tests typical for asphalt rubber: penetration, softening point temperature, resilience and rotational apparent viscosity.

## 4. Results and Discussion

### 4.1. Influence of digestion time on Morphology

The objective of the Scanning Electron Microscopy (SEM) is to verify the modification of the asphalt rubber during the digestion time process. Thus, several samples of asphalt rubber were analysed which included cryogenic and ambient crumb rubber and both types of asphalt binders (35/50 and 50/70). SEM images allow to have an indication of the surface of the material and all conclusions are obtained from the configuration of that surface in terms of the number and size of the irregularities.

In Figure 5 the results of SEM for a magnification of 150x for asphalt rubber with cryogenic crumb rubber and asphalt binder 35/50 with a digestion time of 30 minutes, 8 hours and 48 hours are presented, being possible to verify that at the beginning of the digestion time the crumb rubber has defined edges that at 8 hours of digestion time are nearly rounded. The observation of the sample digested during 48 hours shows that there was a large interaction between the crumb rubber and the asphalt binder since the crumb rubber is more dispersed into the asphalt and its size was reduced. The same conclusion is taken through the observation of Figure 6 for the results of SEM of asphalt rubber with ambient crumb rubber.

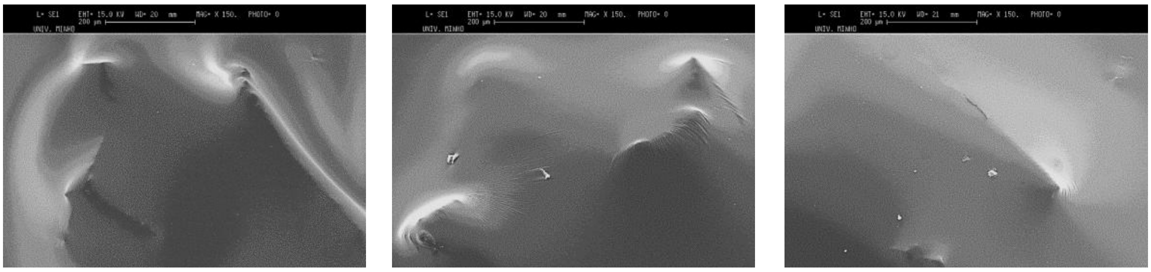

Figure 5. Scanning electron microscopy images (150x) for A35/50RC at 30 minutes (left), 8 hours (centre) and 48 hours of digestion time (right).

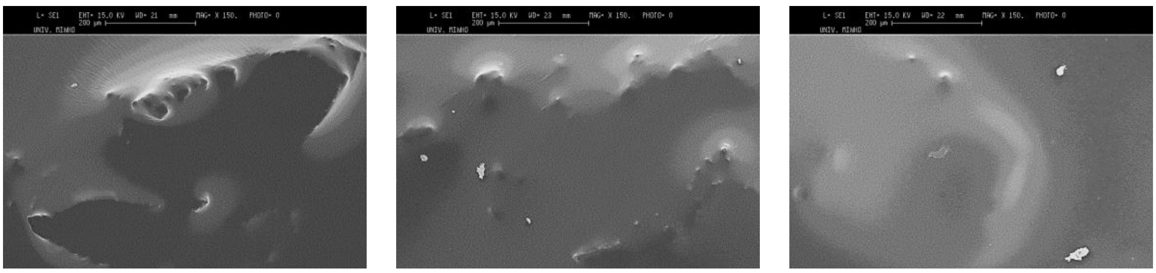

Figure 6. Scanning electron microscopy images (150x) for A35/50RA at 30 minutes (left), 8 hours (centre) and 48 hours of digestion time (right).

The results of SEM of A50/70 modified with cryogenic and ambient crumb rubber are presented in Figure 7 and Figure 8, respectively. Similarly, to the A35/50, it is clear that the increase of digestion time increases the interaction between the asphalt binder and the crumb rubber. After 30 minutes of digestion time the edges of the rubber particles are perceptible, which doesn’t happen when the digestion time is 24 or 48 hours. For higher digestion time the crumb rubber is totally digested into the asphalt binder. Beyond that, comparing the behaviour of both asphalt rubbers, it is possible to understand the difference in the morphology of the cryogenic and ambient crumb rubber, where the cryogenic crumb rubber has defined edges and the ambient crumb rubber has a fluffy appearance. These changes have also been observed by Peralta et al. (2012) in his study about the changes in rubber due to its interaction with bitumen.

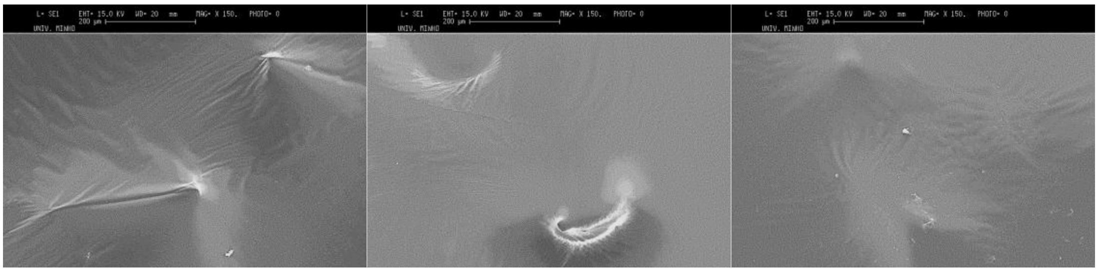

Figure 7. Scanning electron microscopy images (150x) for A50/70RC at 30 minutes (left), 24 hours (centre) and 48 hours of digestion time (right).

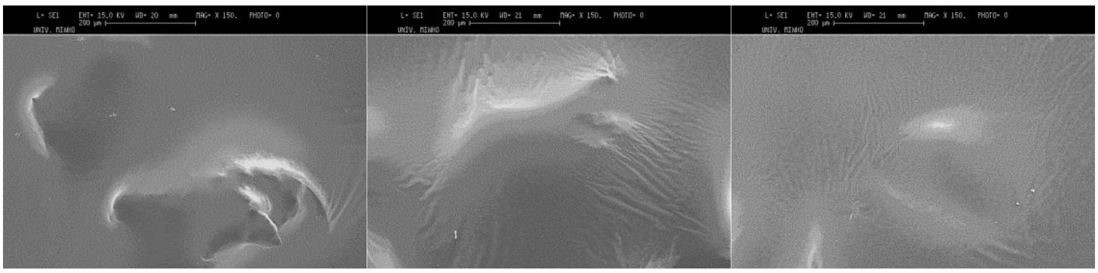

Figure 8. Scanning electron microscopy images (150x) for A50/70RA at 30 minutes (left), 24 hours (centre) and 48 hours of digestion time (right).

**4.2. Influence of digestion time on Rheology**

The rheological study was made with a dynamic shear rheometer by using a 2 mm gap between the 25mm parallel plates. Besides that, researches indicated that the results variability is smaller when the asphalt rubber is tested with a 2 mm gap because of the smaller contact of the rubber particles with both of the parallel plates (Putman & Amirkhanian 2006; Tayebali et al. 1997, Subhy et al. 2015, Brovelli et al., 2013). The results obtained with the dynamic shear rheometer included the shear complex modulus (G*) and phase angle (δ). The results presented in this work refer to a testing frequency of 10 Hz and a shear strain of 12%.
The shear complex modulus and phase angle of the asphalt rubber produced with the asphalt binder A35/50 and cryogenic crumb rubber are presented in Table 2 and represented in Figure 9 and Figure 10.
The results of the shear complex modulus and phase angle show differences with the digestion time mainly in terms of phase angle. G* is almost constant during the time whereas the phase angle has a clear decrease with the digestion time, following a logarithm variation with the digestion time. For long digestion times, the asphalt rubber becomes more elastic.
The results show that as the temperature increases the influence of the digestion time in shear complex modulus decreases, since G* tends to stabilize during the digestion time. The evolution of phase angle depends on temperature too. At high temperatures, the phase angle decreases with the increase of digestion time, which means that the asphalt rubber becomes more elastic and at low temperatures the digestion time doesn't have a big influence on the phase angle, especially after 8 hours of digestion time where it remains almost constant.

Table 2. Shear complex modulus and phase angle for asphalt binder A35/50 modified with cryogenic crumb rubber.

| Time (h) | Shear complex modulus (MPa) | | | | | | | Phase angle (degree) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Temperature (ºC) | | | | | | | Temperature (ºC) | | | | | | |
| | 46 | 52 | 58 | 64 | 70 | 76 | 82 | 46 | 52 | 58 | 64 | 70 | 76 | 82 |
| 0.5 | 150 | 90 | 52 | 31 | 20 | 9 | 4 | 46 | 49 | 52 | 57 | 60 | 72 | 77 |
| 8 | 135 | 79 | 46 | 29 | 16 | 10 | 6 | 49 | 49 | 49 | 50 | 55 | 61 | 67 |
| 24 | 168 | 93 | 53 | 31 | 19 | 12 | 7 | 48 | 48 | 49 | 50 | 52 | 57 | 63 |
| 30 | 154 | 87 | 50 | 30 | 16 | 10 | 7 | 49 | 47 | 48 | 50 | 52 | 57 | 61 |
| 48 | 181 | 102 | 59 | 35 | 22 | 13 | 8 | 47 | 48 | 47 | 47 | 49 | 51 | 55 |

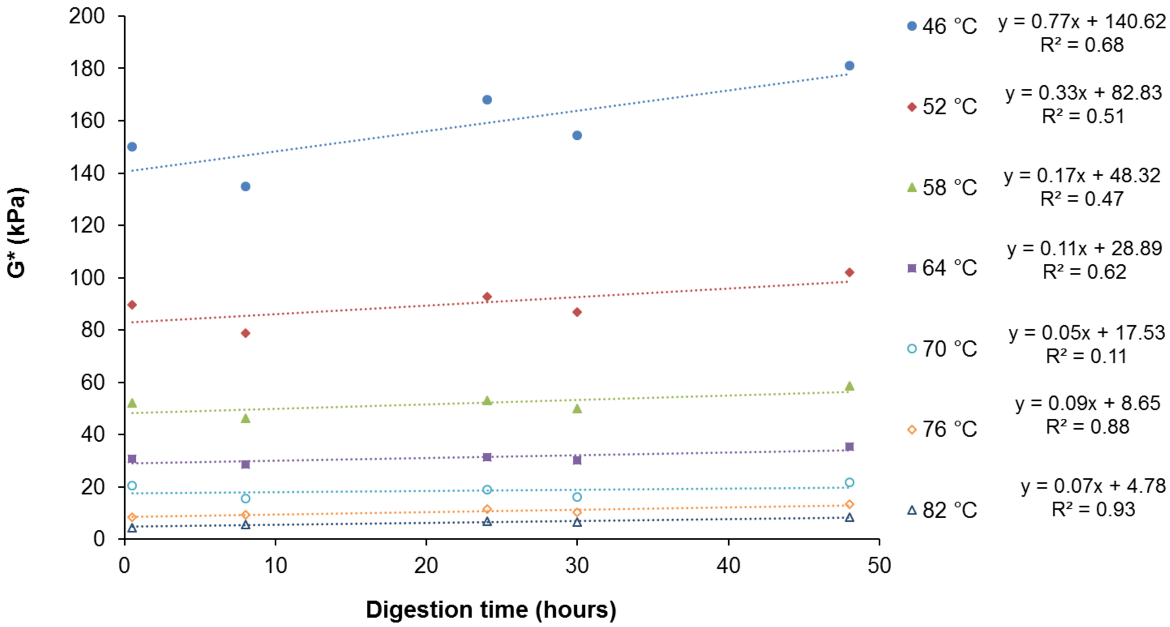


Figure 9. Evolution of shear complex modulus with temperature and digestion time of asphalt binder A35/50 modified with cryogenic crumb rubber.

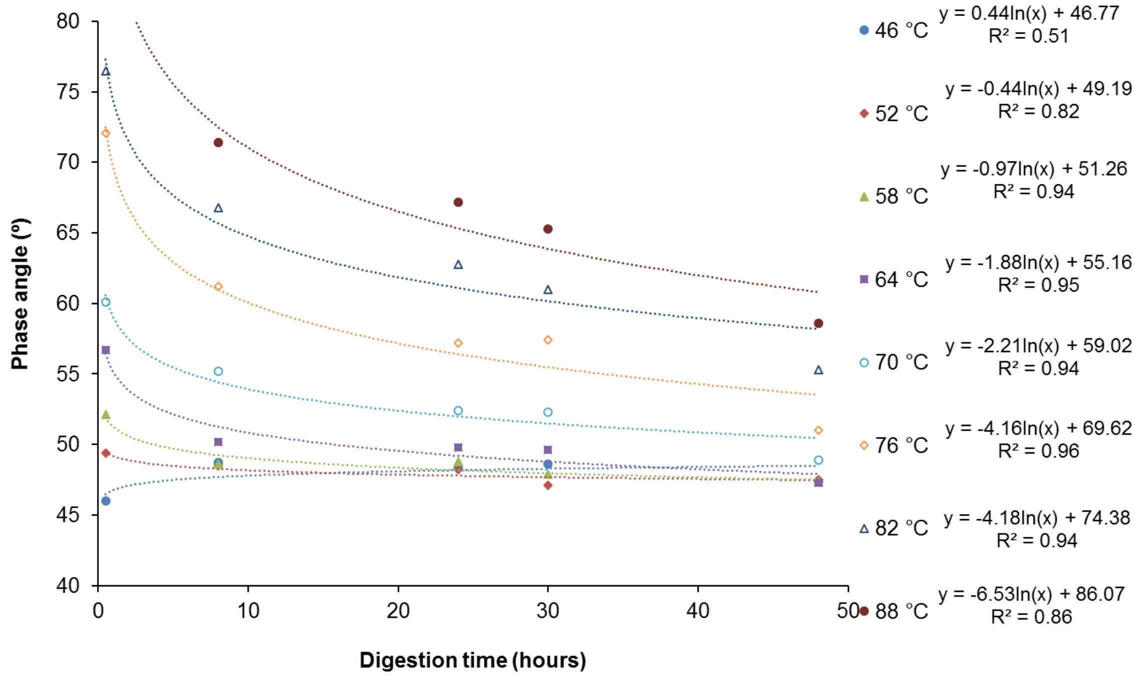


Figure 10. Evolution of phase angle with temperature and digestion time of asphalt binder A35/50 modified with cryogenic crumb rubber.

The evolution of the shear complex modulus in asphalt A35/50 modified with ambient crumb rubber, presented in Figure 11 is similar to the cryogenic crumb rubber. However, the shear complex modulus is more constant during the digestion time compared to the same results for the cryogenic rubber, without significant differences during the time.

The phase angle in asphalt A35/50 modified with ambient crumb rubber, presented in Figure 12, presents a huge decrease after the initial digestion time but it trends to a constant value after 8 hours of digestion time. The values of shear complex modulus as well as of phase angle are indicated in Table 3.

Table 3. Shear complex modulus and phase angle for asphalt binder A35/50 modified with ambient crumb rubber.

| Time (h) | Shear complex modulus (MPa) | | | | | | | Phase angle (degree) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Temperature (ºC) | | | | | | | Temperature (ºC) | | | | | | |
| | 46 | 52 | 58 | 64 | 70 | 76 | 82 | 46 | 52 | 58 | 64 | 70 | 76 | 82 |
| 0.5 | 171 | 98 | 57 | 35 | 17 | 10 | 6 | 47 | 47 | 49 | 51 | 58 | 64 | 69 |
| 8 | 152 | 88 | 52 | 31 | 19 | 13 | 9 | 49 | 48 | 47 | 48 | 50 | 52 | 55 |
| 24 | 148 | 84 | 48 | 29 | 18 | 12 | 8 | 50 | 50 | 50 | 50 | 50 | 52 | 53 |
| 30 | 160 | 90 | 52 | 32 | 20 | 12 | 8 | 50 | 50 | 50 | 50 | 51 | 53 | 53 |
| 48 | 166 | 93 | 56 | 35 | 22 | 14 | 9 | 49 | 50 | 50 | 49 | 50 | 51 | 52 |

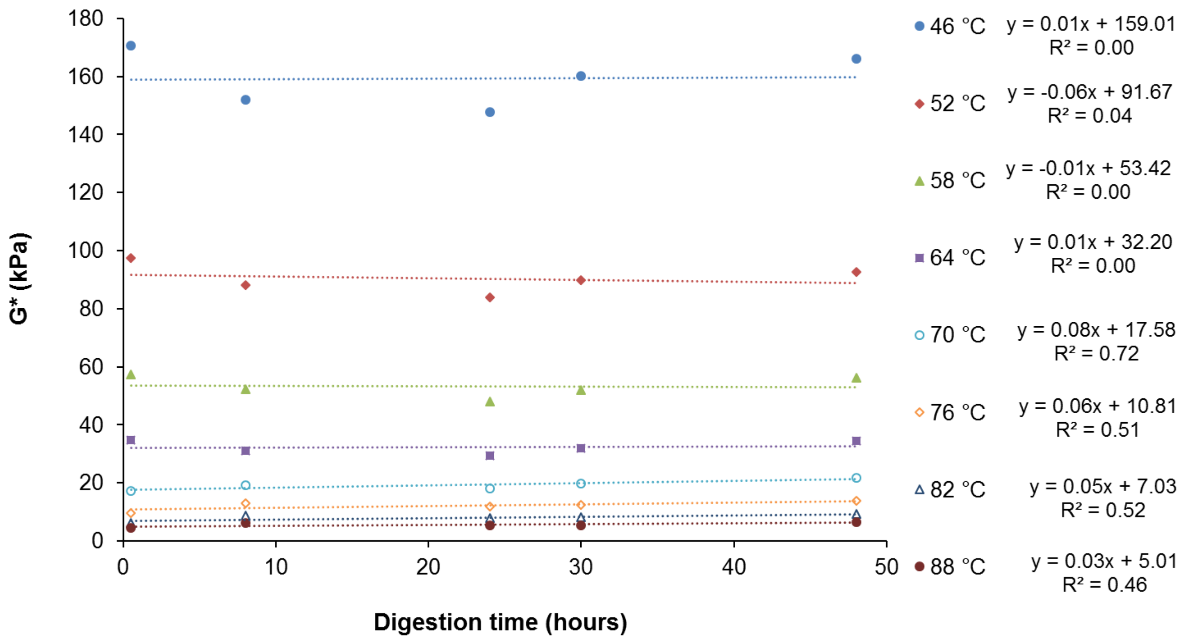


Figure 11. Evolution of shear complex modulus with temperature and digestion time of asphalt binder A35/50 modified with ambient crumb rubber.

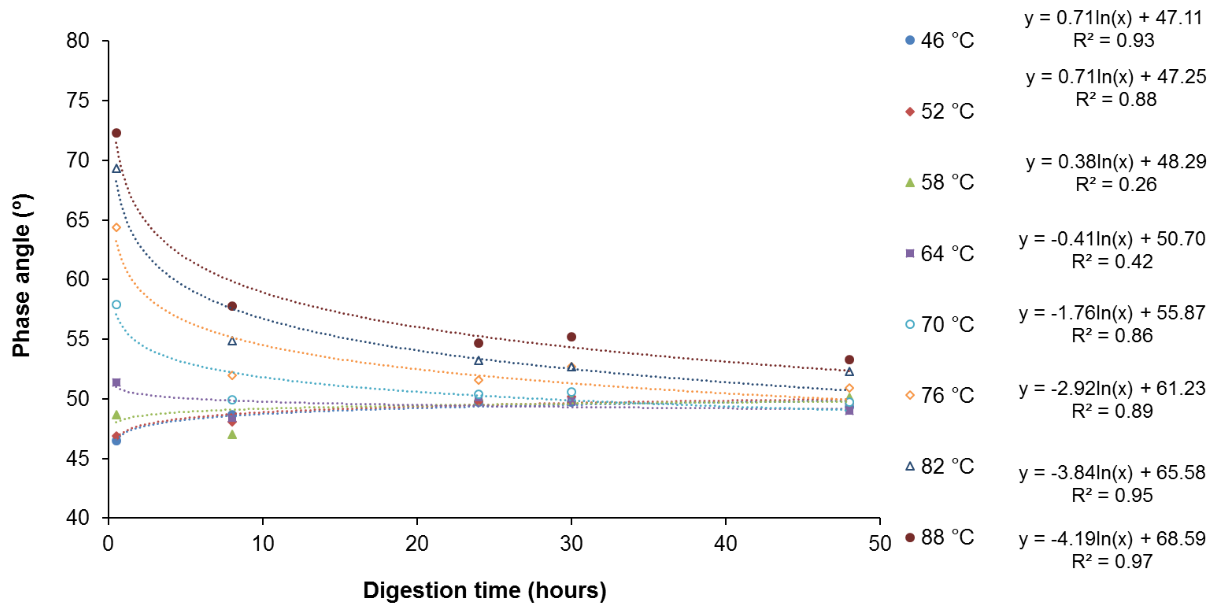


Figure 12. Evolution of phase angle with temperature and digestion time of asphalt binder A35/50 modified with ambient crumb rubber.

The relation between shear complex modulus and phase angle of the asphalt rubber produced with asphalt A50/70 and cryogenic rubber with temperature and digestion time is presented in Table 4 and represented in Figure 13 and Figure 14. For this asphalt rubber it is not visible a dependence of the shear complex modulus with the digestion time. In fact, for low temperatures there is a small decrease of the complex modulus with the digestion time up to 8 hours but for the other tested temperatures, the modulus is almost constant. It is observed a small increase of the modulus for long digestion time for low tested temperatures.

In terms of phase angle, the obtained results show some different behaviours, namely a logarithmic decrease of the phase angle with the digestion time for high temperatures; a large decrease of the phase angle after the initial digestion time; for low testing temperatures the phase angle is not influenced by the digestion time.

Table 4. Shear complex modulus and phase angle for asphalt binder A50/70 modified with cryogenic crumb rubber.

| Time (h) | Shear complex modulus (MPa) | | | | | | | Phase angle (degree) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Temperature (ºC) | | | | | | | Temperature (ºC) | | | | | | |
| | 46 | 52 | 58 | 64 | 70 | 76 | 82 | 46 | 52 | 58 | 64 | 70 | 76 | 82 |
| 0.5 | 101 | 58 | 27 | 15 | 8 | 4 | 2 | 51 | 54 | 64 | 70 | 75 | 79 | 82 |
| 8 | 91 | 52 | 30 | 18 | 10 | 5 | 3 | 49 | 50 | 52 | 55 | 62 | 69 | 74 |

| | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 24 | 92 | 52 | 29 | 18 | 10 | 6 | 4 | 50 | 50 | 51 | 53 | 57 | 63 | 68 |
| 30 | 100 | 57 | 32 | 17 | 10 | 6 | 4 | 50 | 50 | 51 | 54 | 57 | 61 | 65 |
| 48 | 109 | 61 | 34 | 20 | 12 | 7 | 4 | 49 | 50 | 51 | 52 | 55 | 59 | 63 |

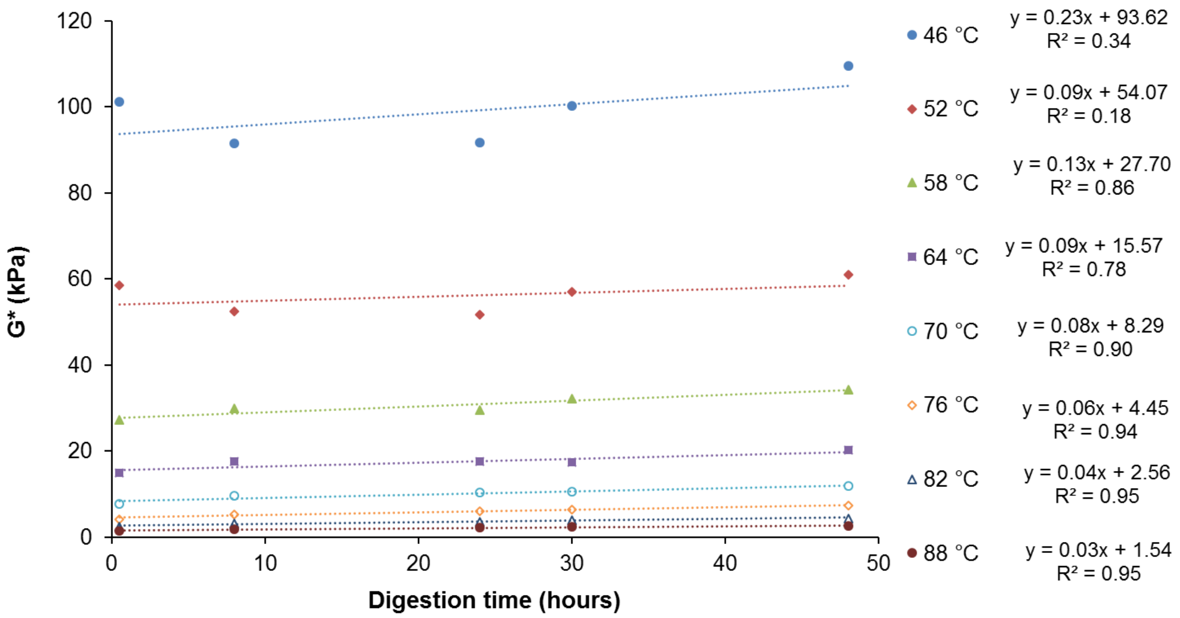


Figure 13. Evolution of shear complex modulus with temperature and digestion time of asphalt binder A50/70 modified with cryogenic crumb rubber.

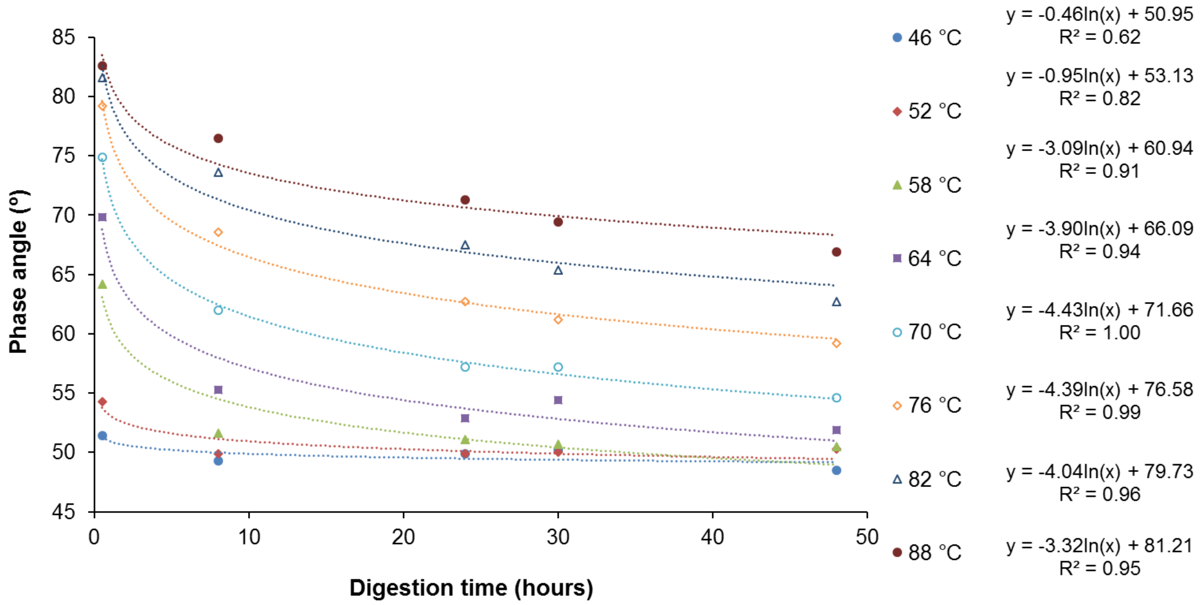


Figure 14. Evolution of phase angle with temperature and digestion time of asphalt binder A50/70 modified with cryogenic crumb rubber.

The results of the asphalt rubber produced with the asphalt binder A50/70 and ambient crumb rubber are presented in Table 5 and represented in Figure 15 and Figure 16, and, unlike the asphalt rubber with cryogenic rubber, the complex modulus tends to decrease after the initial digestion time. After, the shear complex modulus is constant. In terms of phase angle, that is constant after 8 hours of digestion time, it has two different behaviours depending of the testing temperature: for low temperatures the phase angle increased with the digestion time whereas for high temperatures it decreases.

Table 5. Shear complex modulus and phase angle for asphalt binder A50/70 modified with ambient crumb rubber.

| Time (h) | Shear complex modulus (MPa) | | | | | | | Phase angle (degree) | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Temperature (ºC) | | | | | | | Temperature (ºC) | | | | | | |
| | 46 | 52 | 58 | 64 | 70 | 76 | 82 | 46 | 52 | 58 | 64 | 70 | 76 | 82 |
| 0.5 | 99 | 57 | 32 | 18 | 10 | 5 | 3 | 49 | 51 | 54 | 58 | 65 | 71 | 75 |
| 8 | 66 | 36 | 20 | 12 | 2 | 4 | 3 | 53 | 53 | 53 | 55 | 67 | 62 | 65 |
| 24 | 60 | 32 | 18 | 10 | 6 | 4 | 2 | 55 | 55 | 54 | 56 | 58 | 60 | 64 |
| 30 | 64 | 34 | 19 | 11 | 6 | 4 | 2 | 55 | 54 | 55 | 57 | 58 | 61 | 63 |
| 48 | 65 | 35 | 19 | 11 | 7 | 4 | 3 | 55 | 56 | 56 | 57 | 58 | 60 | 63 |

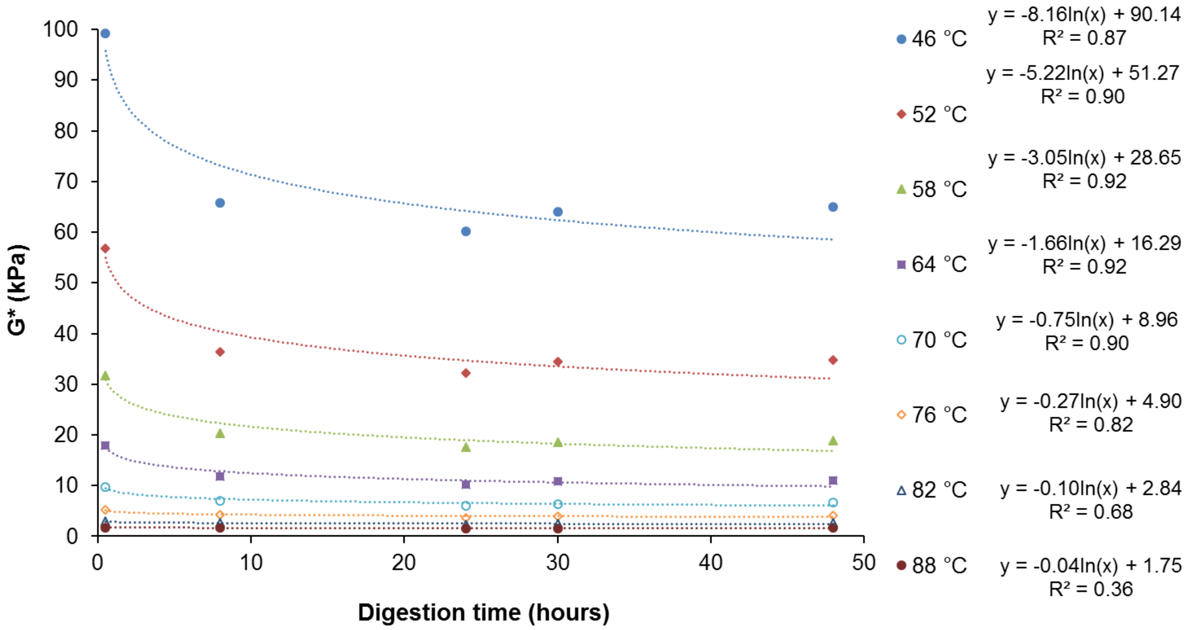


Figure 15. Evolution of shear complex modulus with temperature and digestion time of asphalt binder A50/70 modified with ambient crumb rubber.

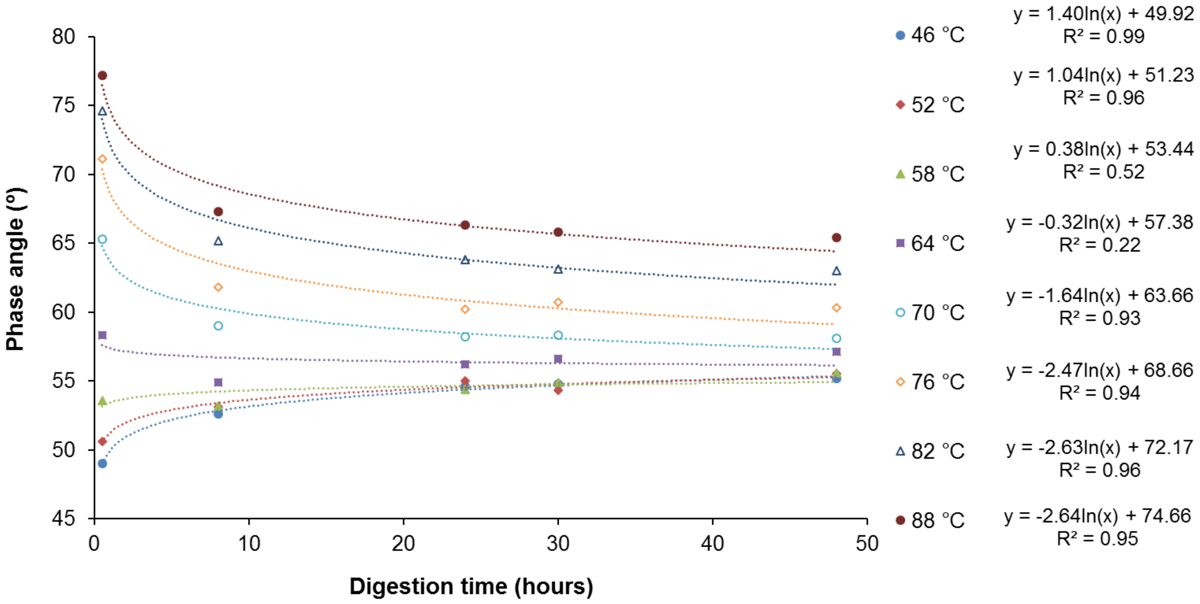


Figure 16. Evolution of phase angle with temperature and digestion time of asphalt binder A50/70 modified with ambient crumb rubber.

After comparing the results of shear complex modulus and phase angle from the asphalt binders A35/50 and A50/70 modified with cryogenic and ambient crumb rubber, it is possible to verify that the type of added crumb rubber doesn't have a great difference in the final results. Also, in both scenarios the shear complex modulus is almost constant during the digestion time while the phase angle depends on the test temperature, being almost constant for low temperatures and decreasing with the digestion time for high temperatures.

The type of asphalt binder used in this work, namely the A35/50 and A50/70, influences the asphalt rubber binder function of the base properties of the asphalt. The hard asphalt binder (A35/50) increases the shear complex modulus and decreases the phase angle for all configurations studied, i.e., the digestion time and testing temperature. This expected behaviour has been observed in many studies on asphalt rubber behaviour.

### 4.3. Influence of digestion time on Conventional Properties

Despite the asphalt technologist is moving towards rheological characterisation of complex binders, the typical conventional tests such as penetration, softening point, resilience and viscosity still play an important role in the evaluation of the asphalt rubber properties within stakeholders. In fact, these properties are still prescribed in specifications which indicate the target values that must be followed for a proper asphalt rubber design.

Table 6 presents all results for penetration, softening point and resilience obtained for all asphalt rubbers produced in this work, function of the digestion time.

Table 6. Conventional properties of the asphalt rubbers.

| Time (h) | Penetration (mm/10) | | | | Softening point (ºC) | | | | Resilience (%) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | A35/50 RC | A35/50 RA | A50/70 RC | A50/70 RA | A35/50 RC | A35/50 RA | A50/70 RC | A50/70 RA | A35/50 RC | A35/50 RA | A50/70 RC | A50/70 RA |
| 0.5 | 24 | 23 | 30 | 28 | 72 | 80 | 66 | 72 | 49 | 49 | 46 | 39 |
| 8 | 24 | 26 | 32 | 34 | 76 | 81 | 70 | 70 | 52 | 49 | 49 | 41 |
| 24 | 25 | 26 | 29 | 38 | 77 | 82 | 72 | 69 | 50 | 42 | 45 | 37 |
| 30 | 24 | 24 | 30 | 38 | 79 | 82 | 74 | 70 | 48 | 44 | 47 | 35 |
| 48 | 24 | 26 | 29 | 37 | 82 | 83 | 76 | 70 | 44 | 39 | 47 | 36 |

The needle penetration test results are condensed in Figure 17. A clear decrease on the penetration was observed from 42 mm/10 and 61 mm/10, respectively for A35/50 and A50/70 asphalts, for values in the range of 23-26 and 28-38 mm/10.

For A35/50 asphalt, despite some variations in the asphalt rubber consistency, the influence of digestion time is not clear since the penetration is almost constant. Beyond that, the maximum variation of the penetration on the 48 hours is 3 mm/10 for the asphalt rubber produced with ambient crumb rubber which evidences the small effect of digestion time on the penetration parameter. For the cryogenic crumb rubber, the variation was only 2 mm/10.

For A50/70 asphalt it is evident that the addition of crumb rubber increases the asphalt rubber stiffness at 25ºC, since the penetration for conventional asphalt is 61 mm/10 and for the asphalt rubber is at least 38 mm/10.

During the digestion time, the penetration varies differently depending on the crumb rubber type. For asphalt rubber with cryogenic crumb rubber the penetration tends to be almost constant during the mixing time with a variation of 4 mm/10. For the asphalt rubber with ambient crumb rubber, its penetration is higher than the penetration of the asphalt rubber with cryogenic crumb rubber and its tendency is to increase with the digestion time reaching a steady state after 24 hours.

The results obtained from the penetration test allows to conclude that the asphalt rubber consistency at 25ºC depends on the type of asphalt and type of crumb rubber. The digestion time influences the consistency only of the asphalt rubber with the softest asphalt and with the ambient rubber.

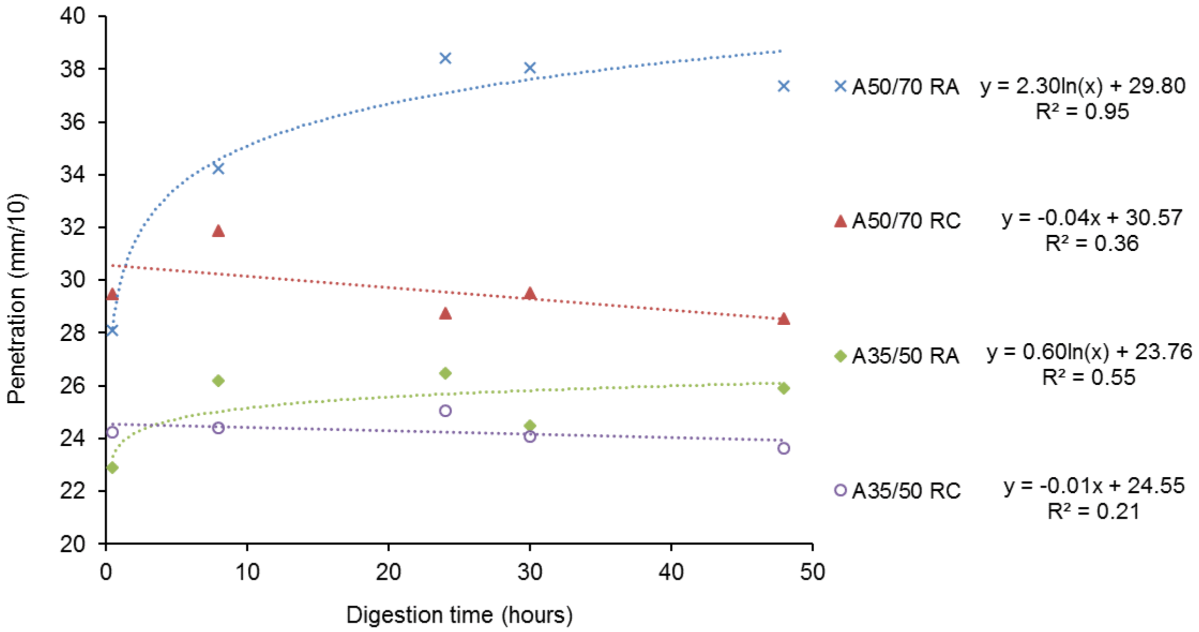


Figure 17. Evolution of the penetration with digestion time.

The softening point results are represented in Figure 18. A clear increase of the softening point was observed from 54ºC and 51ºC, respectively for A35/50 and A50/70 asphalts, for values in the range of 72-82ºC and 66-75ºC.

Observing the evolution of the softening point with the digestion time it is possible to see that the asphalt rubbers with cryogenic rubber have an increase in the softening point during digestion time. The evolution of the softening point of the asphalt rubbers with the ambient rubber depends on the type of conventional asphalt. For the hardest asphalt binder (A35/50) the softening point is almost constant during the time, whereas for the softest asphalt binder (A50/70) there is a small decrease of the softening point up to 8 hours and a constant behaviour after that digestion time.

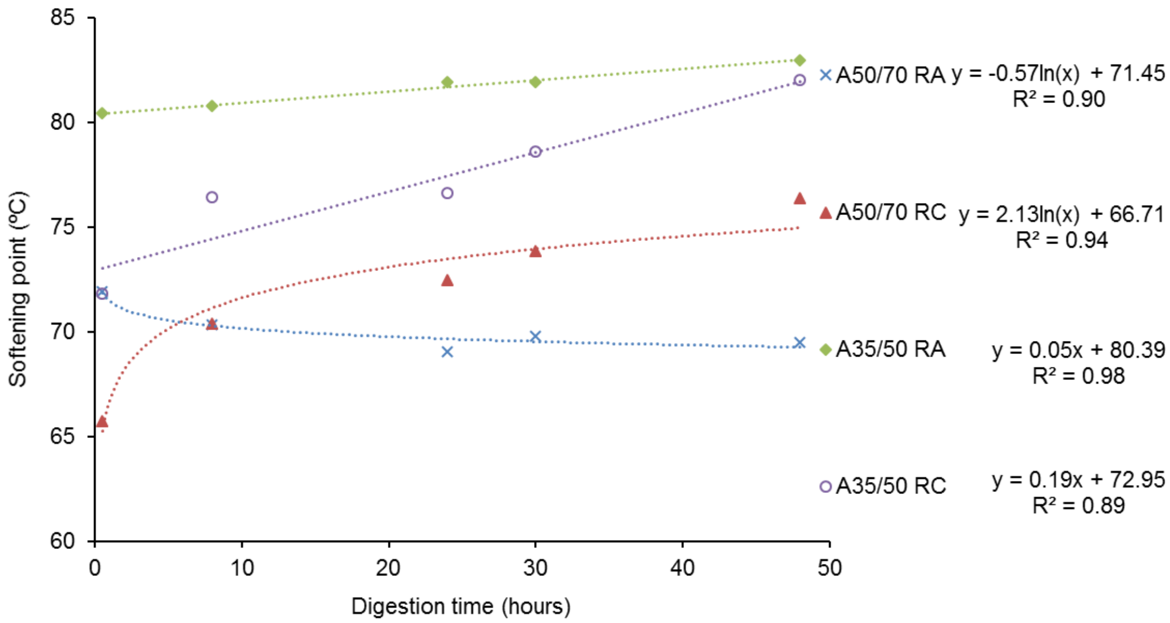


Figure 18. Evolution of the softening point with digestion time.

Resilience results are presented in Figure 19. Resilience of conventional asphalt binders usually varies between 0 and 5%. Modification of the asphalt binder with crumb rubber increases and makes the asphalt rubber elastic and more resistant to fatigue. Here there is not a comparison with the conventional asphalt binder because this property is related to the modified asphalt binders.

For A35/50 asphalt binder, it is possible to identify a tendency towards the decrease of resilience with digestion time, meaning a reduction of the elastic component of the asphalt rubber for both crumb rubbers used in this work. For A50/70 asphalt binder, the obtained results show that the asphalt rubber with cryogenic crumb rubber maintains the resilience during the digestion time with small variations during time.

Hence, from the results it seems difficult to draw out conclusions on trends and differences amongst the different asphalt binders and crumb rubber grinding process. It is worth highlighting that only binder with a stable and even upward trend is the A50/70 RC and the changes in values of resilience are always limited within a 10% change.

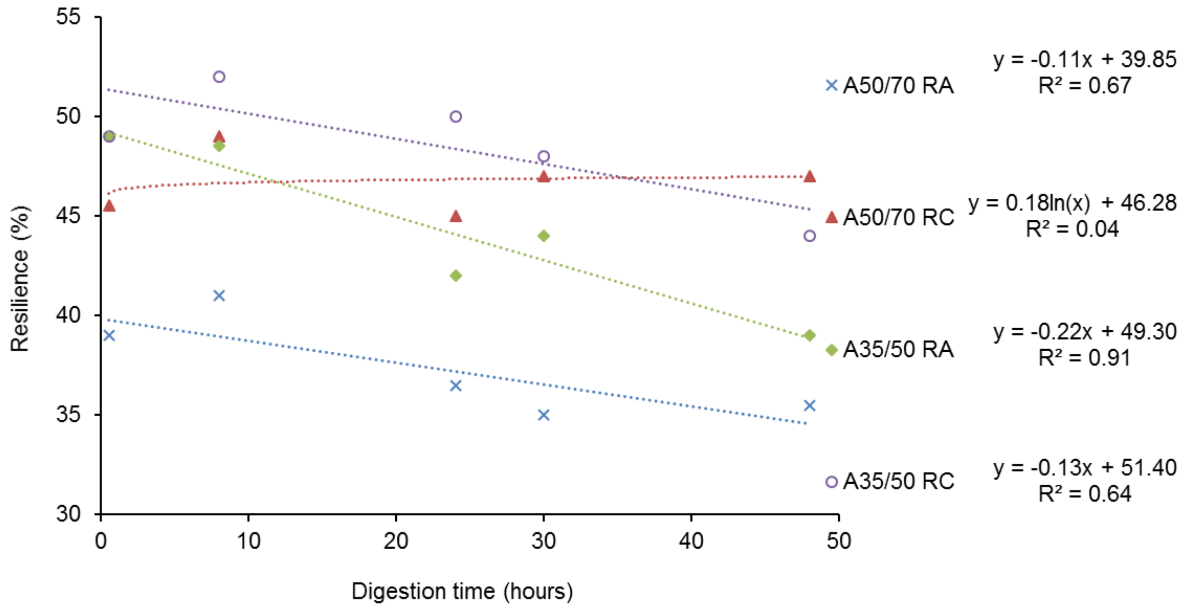


Figure 19. Evolution of the resilience with digestion time.

The results of the assessment of the viscosity during the digestion process are indicated in Table 7 and represented in Figure 20 through Figure 23 for the four asphalt rubbers studied in this work. The viscosity was evaluated with a Brookfield viscometer for test temperatures from 100ºC through 180ºC with a standard coaxial cylinder configuration. A fast analysis of the viscosity for all asphalt rubbers allows to conclude that the digestion time has smaller influence on the viscosity when compared with the conventional and rheological properties. However, all the blends show a similar trend which is related to the type of crumbs. In fact, with both asphalt bases, using ambient crumbs implies a decrease of apparent viscosity with increasing the digestion time. Instead the opposite trend is registered when cryogenic crumbs are used.

The analysis of these results permits to conclude that the viscosity values of the binders is influenced by the crumb rubber type, i.e., for asphalt rubbers produced with ambient crumb rubber the viscosity decreases with the digestion time while for cryogenic crumb rubber the viscosity increases with the digestion time.

Table 7. Viscosity (Pa.s) of the asphalt rubbers.

| Time (h) | Binder | Temperature (ºC) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 100 | 110 | 120 | 130 | 140 | 150 | 160 | 170 | 180 |
| 0.5 | A35/50RA | 642 | 345 | 201 | 72 | 42 | 29 | 19 | 9 | 7 |
| | A35/50RC | 145 | 70 | 26 | 14 | 7 | 4 | 2 | 2 | 1 |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | A50/70RA | 240 | 105 | 58 | 37 | 16 | 11 | 9 | 6 | 5 |
| | A50/70RC | 71 | 32 | 13 | 7 | 4 | 2 | 1 | 1 | 1 |
| 8 | A35/50RA | 711 | 490 | 295 | 187 | 128 | 51 | 20 | 13 | 10 |
| | A35/50RC | 371 | 173 | 78 | 35 | 15 | 8 | 5 | 3 | 1 |
| | A50/70RA | 205 | 123 | 72 | 44 | 18 | 10 | 7 | 4 | 3 |
| | A50/70RC | 167 | 82 | 38 | 16 | 8 | 4 | 3 | 2 | 1 |
| 24 | A35/50RA | 589 | 302 | 172 | 89 | 43 | 27 | 11 | 8 | 2 |
| | A35/50RC | 487 | 227 | 99 | 41 | 17 | 9 | 5 | 3 | 1 |
| | A50/70RA | 207 | 133 | 55 | 36 | 14 | 10 | 7 | 4 | 3 |
| | A50/70RC | 342 | 180 | 98 | 37 | 16 | 10 | 6 | 3 | 3 |
| 30 | A35/50RA | 699 | 290 | 152 | 65 | 39 | 25 | 16 | 7 | 4 |
| | A35/50RC | 430 | 211 | 117 | 32 | 18 | 11 | 6 | 5 | 1 |
| | A50/70RA | 211 | 133 | 55 | 35 | 14 | 9 | 6 | 4 | 2 |
| | A50/70RC | 233 | 116 | 62 | 25 | 15 | 8 | 5 | 3 | 2 |
| 48 | A35/50RA | 661 | 381 | 179 | 75 | 42 | 26 | 17 | 8 | 4 |
| | A35/50RC | 482 | 254 | 154 | 59 | 18 | 14 | 9 | 3 | 1 |
| | A50/70RA | 184 | 107 | 44 | 18 | 11 | 8 | 4 | 3 | 2 |
| | A50/70RC | 282 | 113 | 62 | 24 | 14 | 9 | 5 | 3 | 3 |

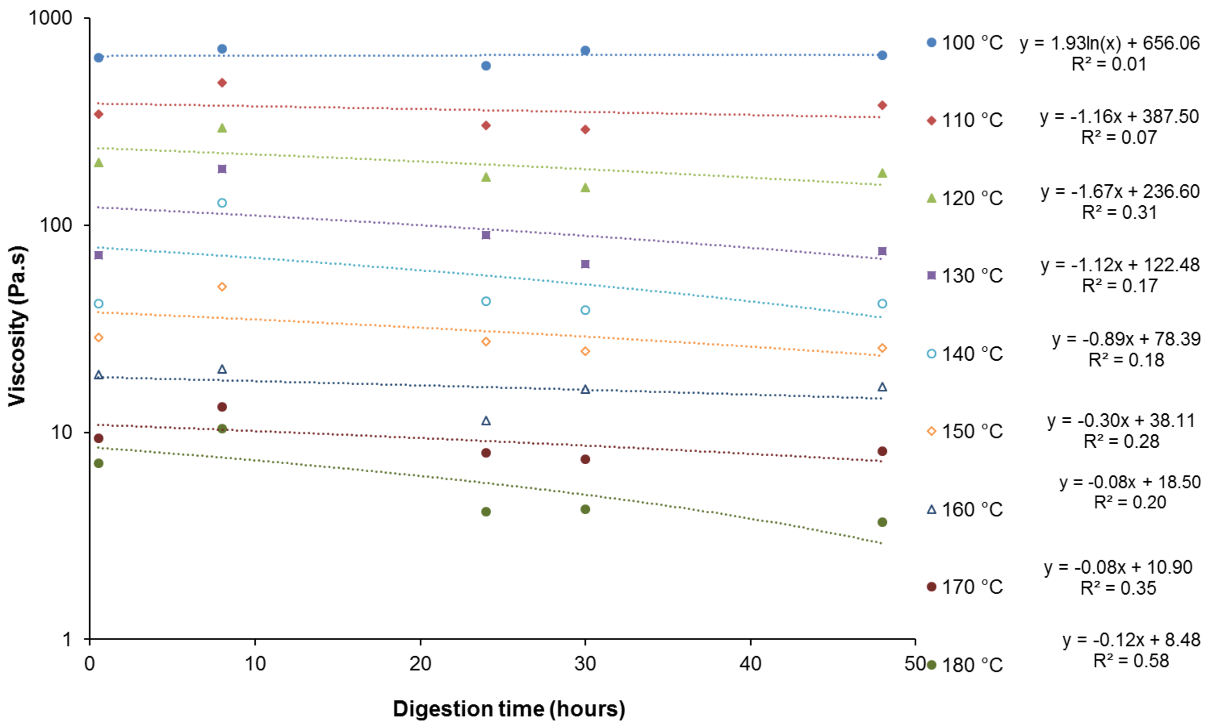

Figure 20. Evolution of the viscosity with digestion time for asphalt rubber A35/50RA.

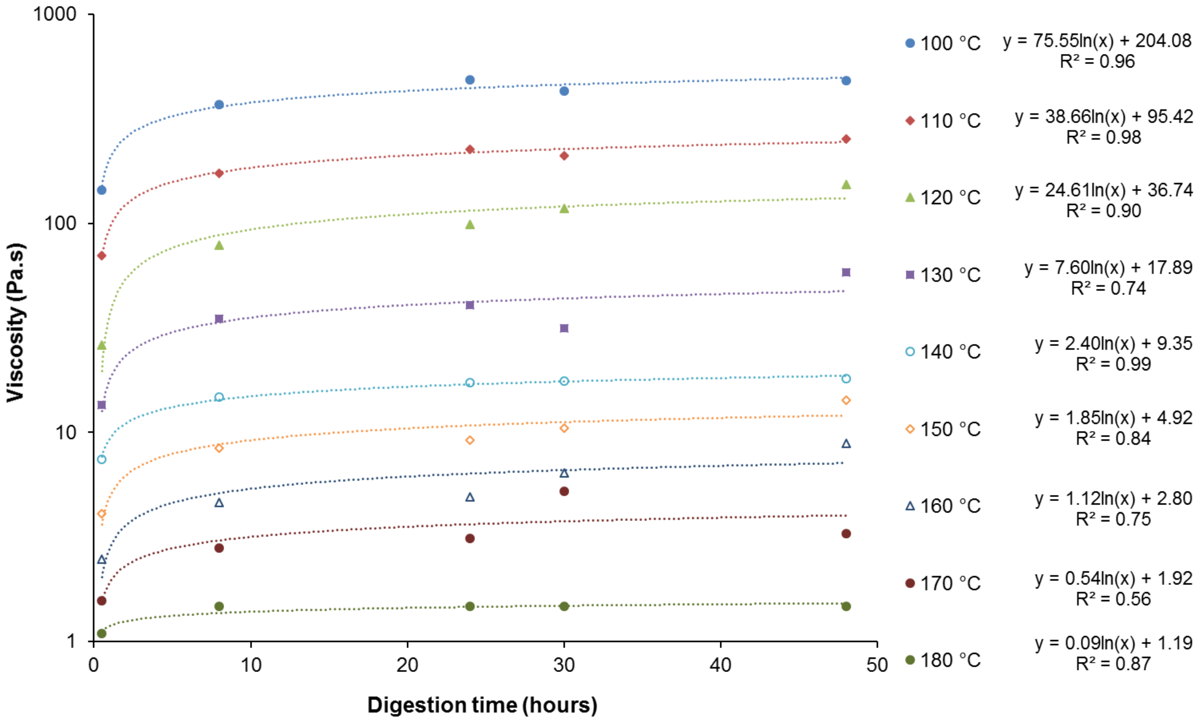


Figure 21. Evolution of the viscosity with digestion time for asphalt rubber A35/50RC.

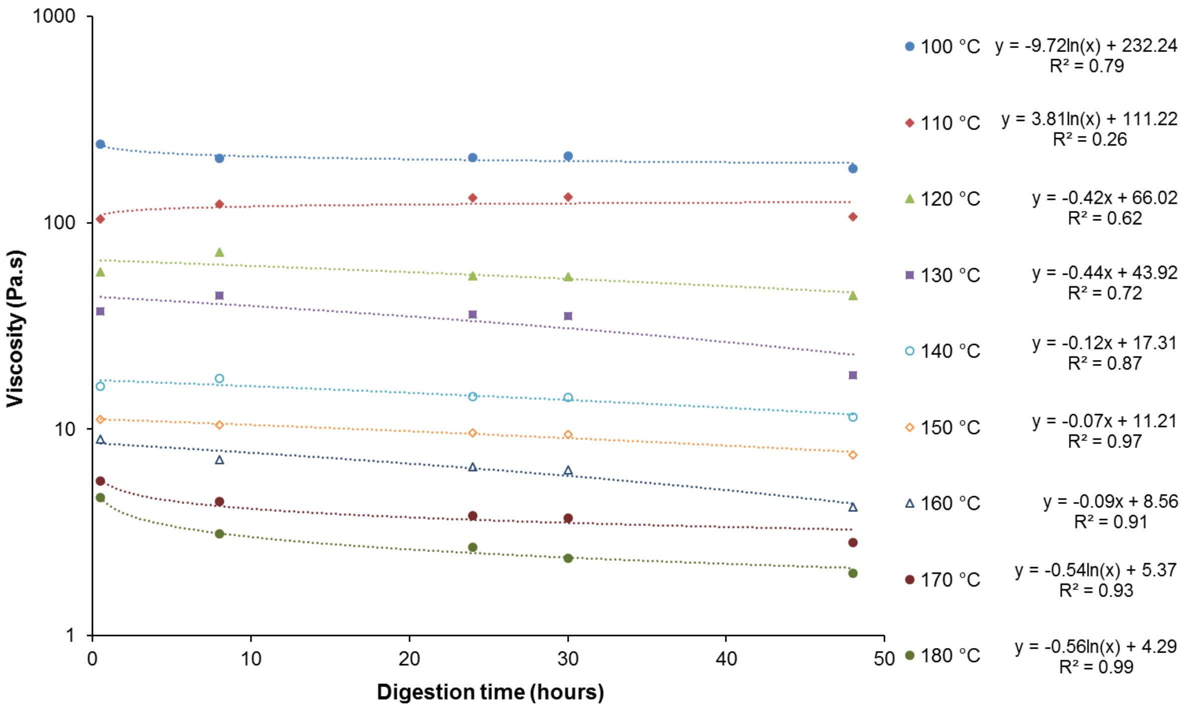


Figure 22. Evolution of the viscosity with digestion time for asphalt rubber A50/70RA.

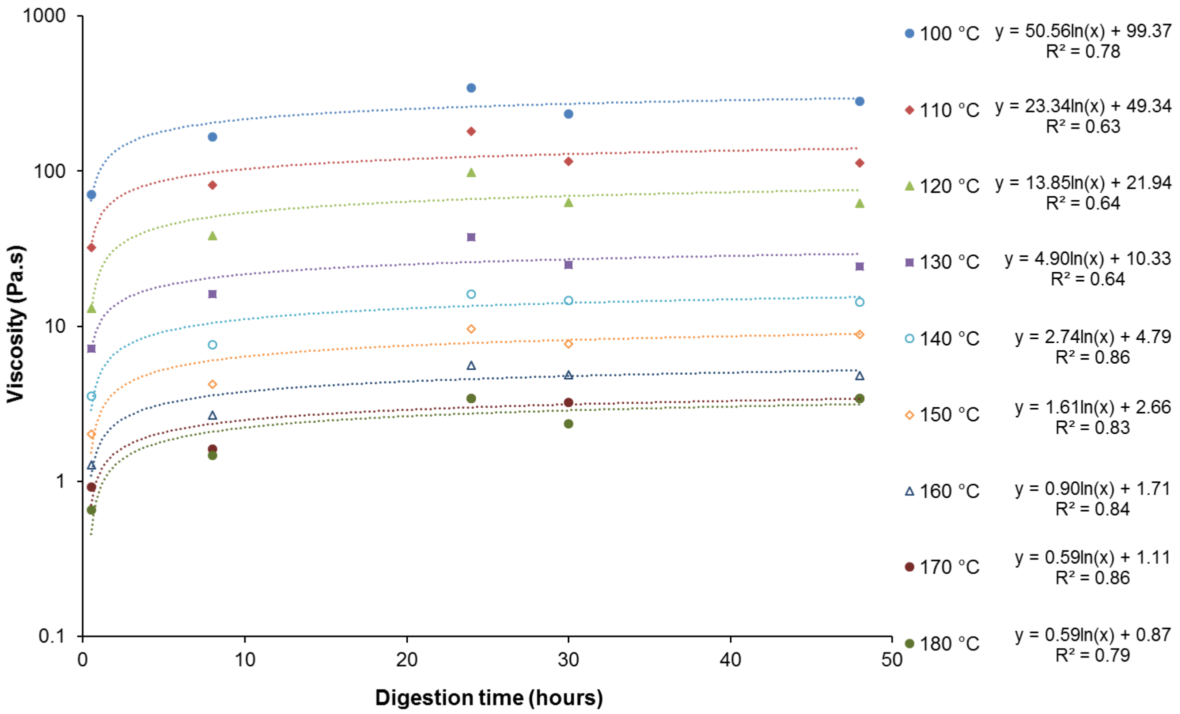


Figure 23. Evolution of the viscosity with digestion time for asphalt rubber A50/70RC.

## 5. Summary of results

As a result, the following was observed:

- Looking at the morphology of the asphalt rubber, by means of SEM, it is confirmed that when the digestion time increases, the dispersion of the crumb rubber into the asphalt rubber increases. Beyond that, the changes in the morphology of the rubber particles are evident and cryogenic rubber particles seems to maintain better their solid shape while ambient crumb rubber looks well dispersed and hardly recognisable after 48h of mixing in the asphalt binder.
- In support of this, the analysis of the rheological properties highlighted that, only for the asphalt rubber with ambient crumb rubber, the shear complex modulus is decreasing with increasing the digestion time. On the other hand, the phase angle, varies within the first hours but then stabilizes for long digestion times. These changes seem to be overall beneficial for the asphalt binder.
- In terms of conventional tests: the influence of digestion time on penetration of asphalt rubber at 25ºC is dependent on the type of asphalt binder and type of crumb rubber. Increase in digestion time seems detrimental only with ambient rubber and pen 50/70. Observing the evolution of the softening point with the digestion time it is possible to

see that the asphalt rubber binders with cryogenic rubber do not decrease their softening point during digestion time. Also, the evolution of the softening point of the asphalt rubber binders with the ambient rubber depends on the type of conventional asphalt binder. In terms of resilience, tests showed that a long digestion time has no remarkable disadvantages in the elastic characteristics of the asphalt rubber, while the results of the high service temperature viscosity confirm that crumb rubber grinding process plays an important role: for ambient crumb rubber the viscosity decreases with the digestion time whereas for the cryogenic crumb rubber increases with the digestion time.

## 6. Conclusions

Asphalt rubber is a complex material which holds new challenges for asphalt technologists as well as contractors. In fact, in order to handle and manufacture asphalt rubber through the wet process, asphalt plants need to be equipped with tanks with special augers able to maintain the asphalt rubber stable during hot storage, hence avoiding phase separation. Typically, digestion time needed to achieve the desired peak performance are up to one hour, however due to practical issues related to operations at the asphalt plant, the “holding” time of the binder might increase significantly. Hence, it is of paramount importance for asphalt technologists to realize the effect of long digestion time on the properties of the asphalt rubber binder, so to avoid contractors to discard a large quantity of asphalt rubber or asphalt mix. Hence, the main objective of this work was to analyse the effect of long digestion time, up to 48 hours, on the morphology, rheology as well as on the conventional properties of the asphalt rubbers kept agitated in low shear at fixed temperature of 180 ºC, and also highlighting eventual differences when different materials are used, both asphalt binders and rubber crumbs. As a results, it is possible to conclude that the over-digested asphalt rubber seems having properties comparable with those typically produced/store within 30 minutes, allowing to be used even after prolonged digestion times.

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