

EFFECT OF PROCESSING TEMPERATURE VARIATION ON STORAGE STABILITY OF TIRE RUBBER MODIFIED BINDERS

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ABSTRACT

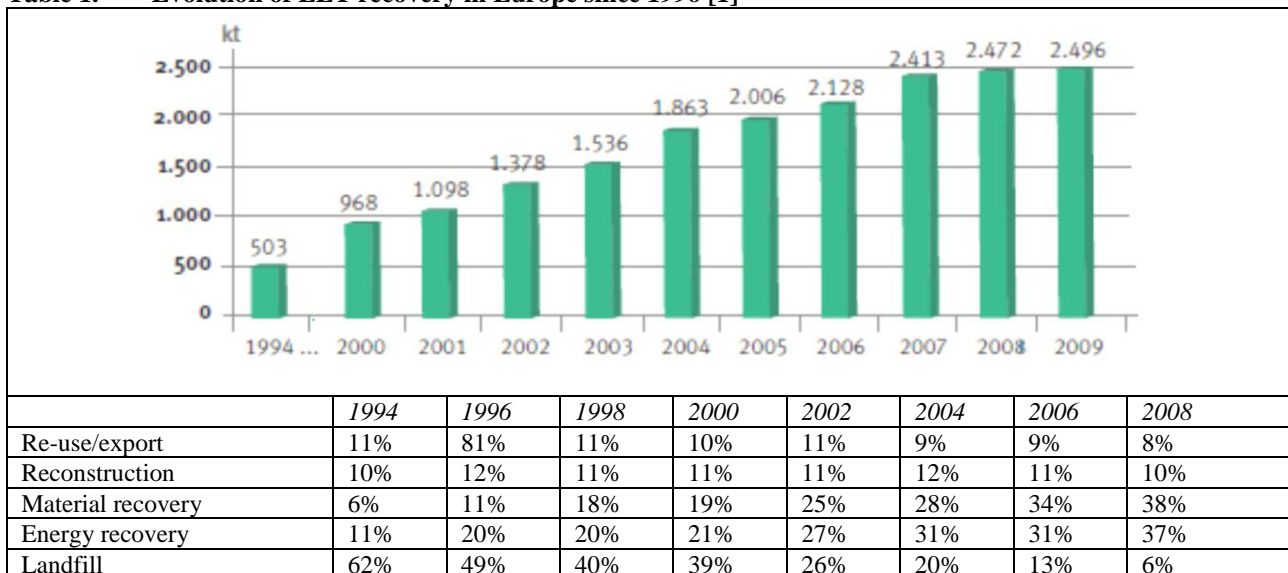
Tire Rubber Modified Binders (TR-MBs), produced through wet process and used worldwide (e.g. asphalt rubber), have been demonstrated to provide various benefits to pavements and, moreover, represent a good opportunity for recycling tire rubber. However, this technology is still struggling to be fully adopted in Europe, mainly because of the poor stability of TR-MBs during high temperature storage, which leads to high initial costs in modifying existing asphalt plants. No-agitation TR-MBs (also known as terminal blends) are proving to be a great option and their development could also be the key to spreading the use of tire rubber in paving applications in Europe. This paper aims to enrich this field of research by presenting the results of a study focused on the compatibility of tire rubber-bitumen blends as well as on the effect of processing temperature variation on storage stability of TR-MBs. The experimental program was carried out by a preliminary selection of materials, followed by the production of the TR-MBs, via practical laboratory protocols. A continual comparison between two commercially used SBS-MBs, with high and medium levels of modification, and the TR-MBs produced, helped to understand the effect of varying the selected processing conditions on binder properties. The results showed that an accurate binder design is fundamental in order to obtain high performance binders and to improve compatibility of TR-MBs. Moreover, the study highlights that dynamic mechanical analysis (DMA) is a suitable technique to assess the storage stability of modified binders.

Keywords: Tire rubber, modified bitumen, terminal blend, storage stability, DMA

1. INTRODUCTION

The increasing number of vehicles on the roads of industrialised and developing nations generates millions of used tires every year. About 1.4 billion tires are sold worldwide each year and subsequently as many eventually fall into the category of end-of-life tires (ELTs) (Tab.1). Moreover, the amount of ELTs in Europe, US and Japan is about to increase because of the projected growing number of vehicles and increasing traffic worldwide. These tires are among the largest and most problematic sources of waste, due to the large volume produced and their durability. Most countries, in Europe and worldwide, have relied on land filling to dispose of used tires but the limited space and their potential for reuse has led to many countries imposing a ban on this practice. The current estimate for these historic stockpiles throughout the EU stands at 5.5 million tonnes (1.73 times the 2009 annual used tires) and the estimated annual cost for the management of ELTs is estimated at €600 million [1]. In order to face this problem, in 1989, a Used Tires Group, composed of experts from the main tire manufacturers producing in Europe, was set up under the strategic guidance of the European Tire and Rubber Manufacturers Association (ETRMA). This group was dedicated to the management of end-of-life tires (ELTs). Also thanks to this group, since 1996, the collection rate has increased steadily while there has been a continual decline in the land filling of used tires (Tab.2). In 2009 the European Union was faced with the challenge of managing, in an environmentally sound manner, more than 3.2 million tonnes of used tires, of which 95% were recovered. This confirms Europe as one of the most active areas in the world in the recovery of ELTs.

Table 1. Evolution of ELT recovery in Europe since 1996 [1]



Recovery rates demonstrate that ELT management in Europe is allowing the progressive elimination of land filling and raises the availability of tire rubber to be recycled for other purposes. In fact, the same characteristics that make waste tires such a problem also make them one of the most re-used waste materials, as tire rubber is very resilient and can be reutilised in other products. These efforts should for example help to further develop the use of ELTs in rubberised asphalt in road construction, which has high growth potential in Europe and it is still relatively underutilised: only a few hundred kilometers of roads in total (ETRMA, 2010).

1.1 Tire Rubber in pavement applications

Recycling of Tire Rubber (TR) in asphalt mixes has been in use in the USA since at least the early 1960s by the Arizona Department of Transport. Since then the material has been used more widely in four states in the US and elsewhere, including South Africa, Europe and Australasia. There are two main processes of using rubber in asphalt, i.e. the dry and the wet process. The dry process consists in the production of an asphalt mixture with the addition of a small quantity of ground rubber from discarded tires as a substitute for a part of the mineral aggregate, while the wet process aims to obtain a new binder which captures beneficial engineering characteristics of both base ingredients. Extensive literature films [2-8] clearly show the numerous successes obtained when using Tire Rubber bituminous mixtures produced with the wet process. There are drawbacks to it, chiefly in terms of its workability [9], its recyclability [10], binder storage stability [11] and the fumes it emits during the paving process [12]. Bitumen-rubber pavements are also more expensive than conventional asphalt ones [13], and can be difficult for paving contractors not accustomed to working with the sticky product, which must be placed and compacted within a relatively narrow temperature window [14]. For these reasons, in the USA in the early 90s this technology was left as dead, but when in 1991 the United States federal law called "Intermodal Surface Transportation Efficiency Act" (ISTEA), mandated its widespread use,

the TR-MB technology concept started to make a “quiet comeback” [14]. Furthermore, in 1992 the patent of this specific process, expired and now the material is considered to be in the public domain. Since then, considerable research has been done worldwide to improve the properties of TR-MB in order to try to solve the issue related to its production and usage. In Europe, rubberised asphalt has been used since 1981 in Belgium, as well as in France, Austria, the Netherlands, Italy, Germany and Sweden, and more intensively since 1999 in Portugal and Spain [15].

1.2 Processing TR-MB

The method of modifying bitumen with TR is generally associable with the term “wet process.” As mentioned before, this technology has existed since the 1960s, but the nature of the mechanism by which the interaction between bitumen and TR takes place has not been fully characterized. The reaction itself is made up of two simultaneous processes: partial digestion of the rubber in the bitumen on one hand and, on the other, adsorption of the aromatic oils of the bitumen, within the polymeric chains of the rubber, both natural and synthetic, contained in the TR. The adsorption of aromatic oils causes the rubber to swell and soften [16,17]. TR particles are swollen by the absorption of the bitumen oily phase at high temperatures (160–220°C) into the polymer chains, which are the key components of TR-MB to form a gel-like material. Rubber reacts in a time-temperature dependent manner and during this process there is a simultaneous reduction in the oily fraction of the bitumen and an increase in the TR particle sizes with a consequent reduction in the inter-particle distance. If the temperature is too high or the time period is too long, the process will continue to the point where swelling is replaced, due to long exposure to high temperatures, by depolymerisation/devulcanisation [18]. This phenomenon implies a releasing of rubber components back to the liquid phase causing a decrease in the stiffness (G^*) while the elastic properties (δ) continue to change. If the temperature is high or the time period is long enough, depolymerisation will continue, causing more destruction of the binder networking [19]. The phenomenon described significantly affects the properties of the final blends and a proper selection of the processing conditions, mainly time and temperature, leads to a completely different type of TR-MBs. Caltrans [9], classified two different types of TR modification: the “wet process-high viscosity” and the “wet process-no agitation” (also known as “terminal blend”). Tire Rubber modified binders that maintain or exceed the minimum rotational viscosity threshold of 1,500 cPs at 177°C (or 190°C) over the interaction period should be described as “wet process-high viscosity” [9]. The idea with Tire Rubber binders produced with the NO agitation-wet process is to produce a material in which the tire rubber is fully digested, or better dissolved, in the bitumen without leaving visibly discrete particles. It means that these binders do not require constant agitation to keep discrete rubber particles uniformly distributed in the asphalt binders. Therefore this technology makes it possible to use standard asphalt plants for the production of rubberised asphalt mixtures without any modifications.

1.3 Aim of the study

As seen above, TR-MBs are extremely dependent on the selected processing conditions. In particular, the temperature is the factor that most seems to affect the whole modification process and only using high processing temperature leads to a TR-MB which does not require agitation. Therefore, based on these findings and also considering the lack of results in the literature, in this study the authors developed their own experience with TR-MB by using local materials and focused their research on understanding the effect of raising the processing temperature, from 180°C to 210°C, on the storage stability of the blends.

2. EXPERIMENTAL PROGRAM

Two TR-MBs were produced using the same materials and blending protocol but changing the processing temperature from 180°C and 210°C. The modified binders, as well as samples taken during the modification process, were characterized and compared through empirical tests such as penetration (EN 1426), softening point (EN 1427), rotational viscosity at 135 °C and 160°C (EN 13302) and apparent viscosity at 177.5°C as prescribed by the Asphalt Rubber standard (ASTM D6114). Furthermore a polymer dispersion analysis, by means of optical microscope, and a rheological characterization were also performed. The latter was carried out with a Dynamic Mechanical Analysis (DMA) by means of DSR tests (details in section 2.3). Furthermore, a comparison with two Styrene-Butadiene-Styrene modified bitumens (SBS-MBs with a medium and a high level of modification), supplied by an Italian oil company, was also performed in order to understand the level of modification achieved. Lastly, all the binders were subjected to a storage stability test in accordance with the EN 13399 Standard but modified due to the fast separation of the Tire Rubber. After storage, binders were analyzed in terms of empirical and rheological properties.

2.1 Materials

A 50/70 pen bitumen and a 30# ambient car and truck tire rubber (max 0.6 mm), processed at ambient temperature and free of fibers and steel, were selected as base materials for the production of two TR-MBs. The base bitumen was chosen among six available bitumens provided by various Italian plants and identified as bases for modification. In order to choose the best base, all the bitumens were chemically characterized through a SARA analysis, based on method B of the ASTM D4124 standard. According to what was found in the literature about the production of PMBs [20], higher aromatic content and lower colloidal index/higher dispersion factor enhance compatibility between the

bitumen and its modifier. Based on these findings, the bitumen shown in Table 2 was used for the production of the TR-MBs.

Table 2. Physical and chemical properties of the base bitumen selected

Penetration 25°C (mm/10) - EN 1426	50 dmm
Softening Point (°C) - EN 1427	51°C
Penetration Index	-1.04
Rotational Viscosity @ 100°C (ASTM T316-04)	3.458 Pa.s
Rotational Viscosity @ 135°C (ASTM T316-04)	0.429 Pa.s
Rotational Viscosity @ 160°C (ASTM T316-04)	0.139 Pa.s
Saturates [%]	11.9
Aromatics [%]	61.4
Resins [%]	17.1
Asphaltenes [%]	9.6

Summarizing, the following binders were incorporated in this study:

neat bitumen: pen 50/70 bitumen

TR-MB HS180: Tire Rubber-Modified Bitumen (TR-MB) produced mixing base bitumen with 18% in weight of 30# tire rubber in High Shear (HS)

TR-MB HS210: Tire Rubber-Modified Bitumen (TR-MB) produced mixing base bitumen with 18% in weight of 30# tire rubber in High Shear (HS)

SBS-MB H: modified bitumen (SBS-MB) with a high content of SBS (Hard - H)

SBS-MB M: modified bitumen (SBS-MB) with a medium content of SBS (Medium - M).

2.2 TR-MB blending protocol

Two TR-MBs were blended using a Silverson high shear laboratory mill and adapting a thermostatic bath as processing vessel. The proportions between the bitumen and the tire rubber (TR) were maintained constant (18% of weight of bitumen), while two different blending temperatures were used: 180°C and 210°C.

The blending of rubber and bitumen was carried out using the following protocol:

- The required amount of bitumen was heated at 180°C in the oven and then transferred into the processing vessel pre-heated at 180°C or 210°C.
- Time was allowed for the temperature to settle at the selected processing temperature.
- After the equilibration time, TR was fed into the bitumen within the first 5 minutes and high shear mixing up to 3500 rpm was undertaken at 3500 rpm for 2 hours.
- Sampling was undertaken every 20 minutes to check viscosity and morphological and rheological changes.

Table 3 summarizes the mixing parameters used in the blending procedure.

Table 3. Blending protocol

mass of bitumen	Mass of TR (18%)	rubber size	total weight	mixing time	mixing speed	mixing temp.
g	g	mm	g	min	rpm	°C
3000	540	0-0.6	3540	120	3500	180 or 210

2.3 Rheological analysis

Optimizations of TR-MB production and performance comparisons were mainly based on the evaluation of the rheological properties. Therefore, in order to obtain a complete rheological characterization, dynamic mechanical analyses (DMA) were performed by making frequency sweep tests over a wide range of temperatures with an Antoon Paar Physica MCR 101 dynamic shear rheometer (DSR) since it has been proved that SUPERPAVE DSR protocol can also be applied to rubber modified binders [21]. The tests were performed under the following conditions:

- Loading Mode: controlled-strain
- Temperatures: 0°C to 80 °C with 5°C intervals (neat bitumen)
30°C to 80 °C with 5 or 10 °C intervals (TR-MB)
- Frequencies: 0.10, 0.16, 0.25, 0.40, 0.63, 1, 1.6, 2.5, 4, 6.3 and 10 Hz
- Plate geometries: 8 mm ϕ and 2 mm gap (0-50°C) (only neat bitumen)
25 mm ϕ and 1 and 1.5 mm gap (30-80°C)
- Strain amplitude: 0.5% with 8 mm plates (within LVE response dependent on G^*)
0.5% and 2%-12% @80°C with 25 mm plates (within LVE response dependent on G^*)

For each test, samples were prepared by means of a hot pour method, based on Alternative 1 of the AASHTO TP5 Standard (AASHTO, 1998). The gap between the upper and lower plates of the DSR was chosen such that the

rheological properties taken at wider gap widths (1.5-2 mm for 25mm) were independent of the gap and sample geometry was also maintained at high temperatures. The rheological properties of the binders were measured in terms of their complex (shear) modulus, G^* ; and phase angle, δ . Once the raw data were obtained, the Time Temperature Superposition Principle was applied in order to produce master curves at 30°C, isochronal plots and shift factor curves. Black diagrams, Isochronal plots, master curves and binder rutting parameters (SHRP and Shenoy parameters) were used as the basis of all the rheological analyses in this paper.

3. RESULTS AND DISCUSSIONS

3.1 Monitoring of the modification process

Based on a preliminary optimization study, the selected bitumen and TR were blended using high shear mixing for 120 minutes. Every twenty minutes 100 g of materials were sampled and apparent viscosity at 177.5° C was monitored in order to find out the optimum processing time at both temperatures (ASTM D6164). The results reported in figure 1 show that the viscosity peaks, which provide the optimum conditions, were achieved between 40-80 minutes at 180°C and between 20-40 minutes at 210°C.

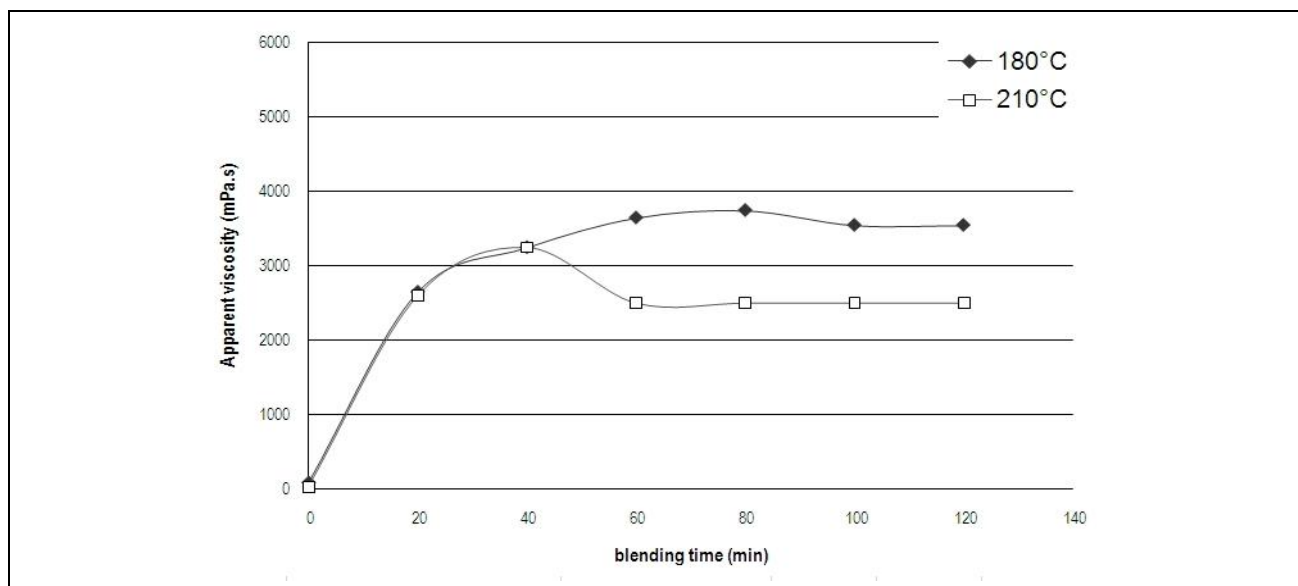


Figure 1. Apparent viscosity @ 177.5°C and morphology of the TR-MBs at different processing time

3.2 Assessing the achieved level of modification of the TR-MBs produced

Finally, in order to assess the achieved level of modification of the processed binders at both temperatures, the TR-MBs were compared with the neat bitumen and the two SBS polymer modified bitumens. Comparisons were made in terms of conventional and rheological properties. From the conventional tests (Tab. 4), it can be concluded that both TR-MBs are harder than their base bitumen (neat) and have a higher SP and Penetration Index. Furthermore, both TR-MBs have the characteristics (only in terms of Pen, SP and Apparent Viscosity) to be classified as Asphalt Rubber type I or II (ASTM D 6114, 2002). Nevertheless, considering PMBs as the target, the TR-MB HS180 shows the best improvements, but it is even stiffer than the SBS-MB Hard and too viscous for the requirements of the ASTM Standard specifications for modified bitumen (ASTM D5892, 2000). In fact, according to this standard, the viscosity at 135°C can be a maximum of 3000 Pa.s. TR-MB HS 210 has a much lower viscosity but it is still to be considered a high-viscosity TR-MB. It is also stiffer than PMBs, but has poor improvement in terms of its softening point.

Table 4 : Results of the empirical tests and viscosity measurements

Conventional properties	neat	TR-MB HS180	TR-MB HS210	SBS-MB M	SBS-MB H
Penetration 25°C (mm/10) (EN 1426)	50	32	37	51	47
Softening Point (°C) (EN 1427)	51	62	55	64	85
Penetration Index	-0.96	0.38	-0.70	1.84	4.81
Apparent Viscosity at 177°C (Pa s) (ASTM D6114)	-	3.500	2.600	-	-
Viscosity at 135°C, (Pa s) (EN 13302)	0.429	5.950	3550	1.290	2.450
Viscosity at 160°C (Pa s) (EN 13302)	0.139	2.300	1.120	0.472	0.742

Furthermore, due to their combination of Penetration and Softening points, both TR-MBs cannot be classified as PMBs within the European Framework Specification (EN 14023, 2005). Supplementary information is given by the rheological tests. The frequency dependence of complex modulus and phase angle for the modified binders was

assessed by producing Black diagrams (Fig. 2) and master curves at a reference temperature of 30°C (Fig. 3a,b). Isochronal plots at 10 rad/s were also produced to show the temperature dependency of the binders (Fig. 4a,b), while the Shenoy and SHRP rutting parameters were calculated to be able to estimate the binder performance at high service temperatures. A comparison regarding the complex modulus of both the SBS-MBs and both the TR-MBs indicates that the behavior of the binders is pretty similar over the whole range of frequencies considered. Making a comparison with the original base bitumen, all the modified binders show clear improvements, having a lower frequency (temperature) susceptibility and being stiffer at low frequencies (high temperatures), but softer at high frequencies (low temperatures) (Fig. 3a and 4b). The latter result is due to the presence of the polymer network which affects the rheological properties of the modified binders, with a slight plateau on the complex modulus curves at low frequencies, which leads to lower rigidity in the material at high frequencies (low temperatures). The plateau is even more evident on the phase angle master curves, where it is noticeable that the modification process significantly reduces the phase angle, at medium/low frequencies (temperatures), thus making it possible to significantly increase the elastic properties (Fig. 3b and 4a). From the estimation of the binder performance at high service temperature, through the SHRP and Shenoy parameters, it is possible to notice that TR-MB 180HS performs almost like a PMB with high level of modification, while by using high processing temperature, TR-MB HS 210, the performance are only comparable with the SBS-MB M. It is important to underline that Shenoy's parameter better highlights how SBS-MB H is overall the best binder against rutting, emphasizing the differences clearly also present in the phase angle master curve. An overall analysis of the results (Figs. 2, 3,4 and 5), demonstrates that between the two TR-MBs processed for two hours in high shear, TR-MB HS180 is stiffer and more elastic, at high temperatures and that it has rheological behavior almost comparable with the SBS-MB H. The TR-MB HS210 is softer and less elastic than the TR-MB HS180 and thus it has rheological properties comparable only with a PMB with a medium level of modification. This confirms that the reaction process between TR and bitumen is sped up by the higher processing temperature and it highlights the importance of preliminary investigations to optimize the mixing time relative to the selected processing temperature.

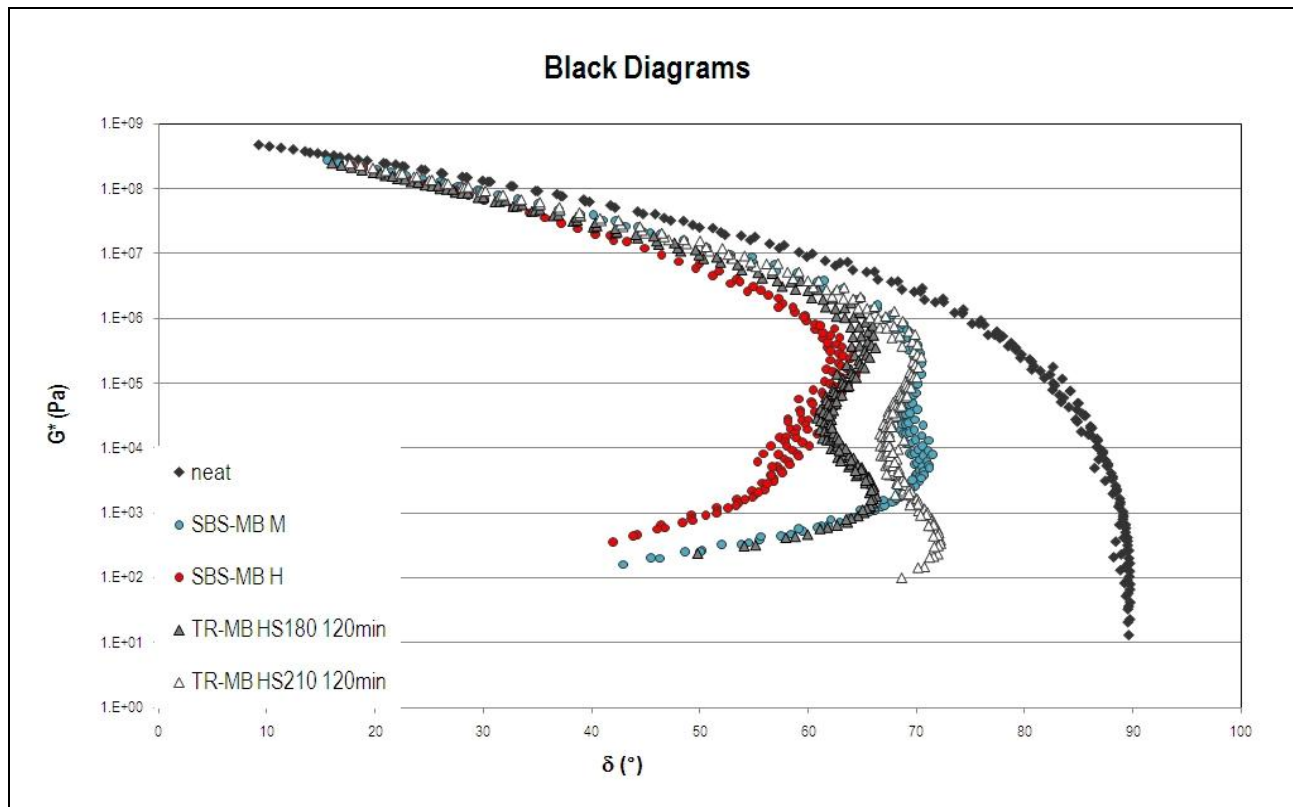


Figure 2. Black diagrams of neat bitumen, PMBs and TR-MBs HS produced at 180°C and 210°C

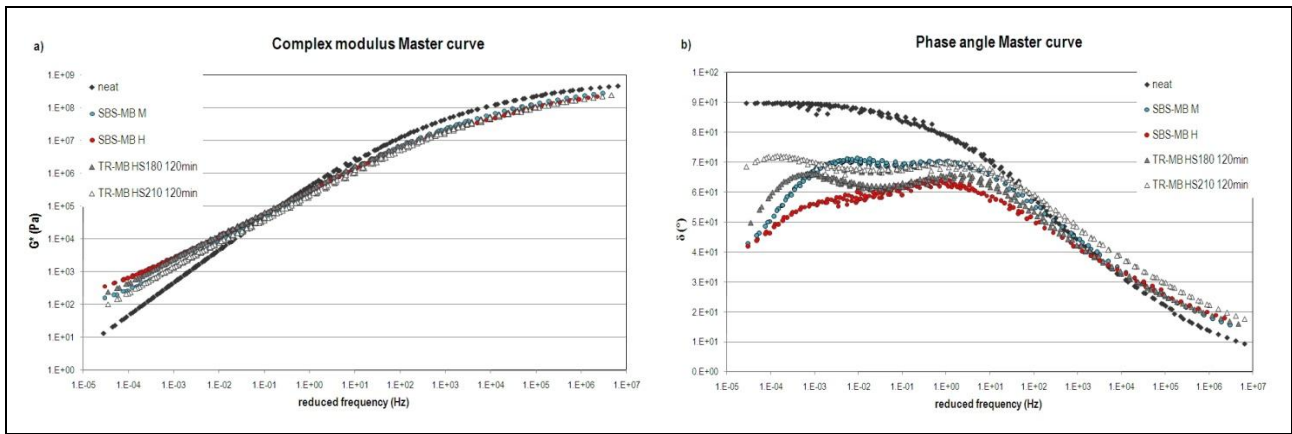


Figure 3. Master curves of $|G^*|$ (a) and δ (b) at 30°C of neat bitumen, PMBs and TR-MBs

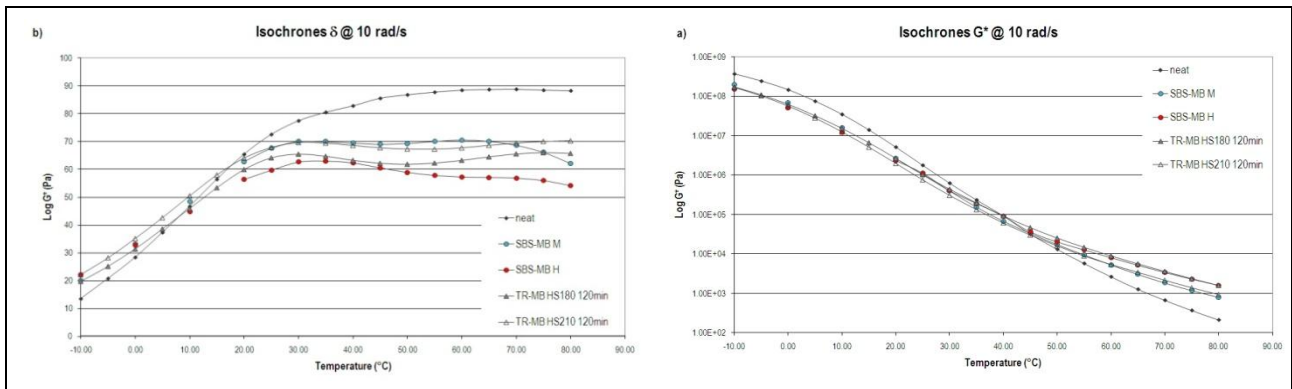


Figure 4 Isochronal plots of $|G^*|$ (a) and δ (b) at 10 rad/s of neat bitumen, PMBs and TR-MBs

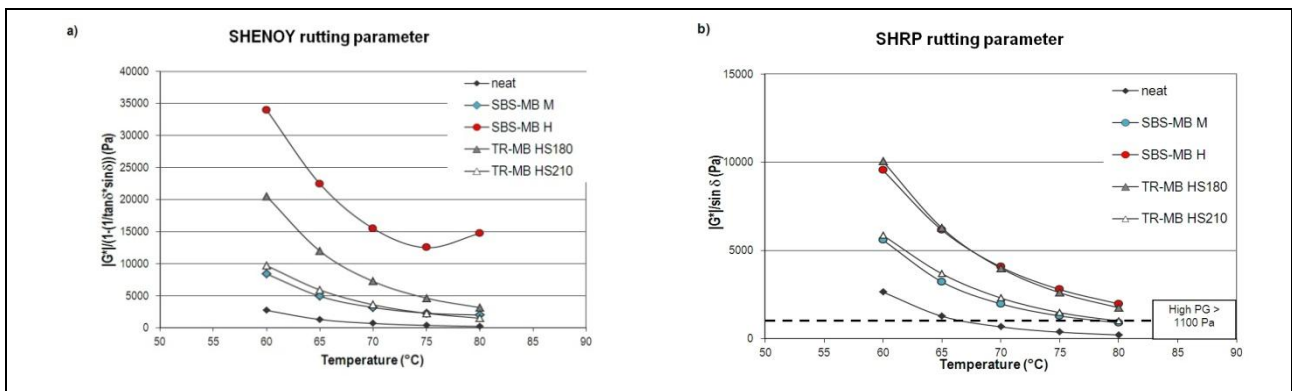


Figure 5. Performance comparison of TR-MBs HS, PMBs and neat by Shenoy and SHRP parameters

3.3 Storage stability analysis

This section was focused on assessing the effect of the processing temperature on the stability of the blends. TR-MBs produced after 120 minutes of high shear were subjected to storage stability analysis, as well as the two PMBs (SBS-MB M and SBS-MB H), which were used as storage-stable binder references. All the modified bitumens were subjected to hot storage tests based on the relative European Standard UNI EN 13399, but modified due to the fast phase separation of the rubber. Softening point results (Tab.5) show that rubber phase separation starts to be relevant after a few hours. The results do not show a clear trend, since TR-MB produced at 210°C seems to have a higher separation rate after 4 hours, while after 24 hours of hot storage it is the TR-MB HS180 which shows the higher difference between the top and bottom sections. It also has to be noted that performing the ring and ball test with such heterogeneous material is almost never problem-free, compared to testing neat bitumen. Again, when it is necessary to classify modified binders, it is really difficult to make comparisons only based on conventional classification, also because probably they are not suitable for this kind of binders.

For this reason, all the binders were also characterized through rheological tests. DMA was performed by frequency sweep tests at high service temperatures (30-80°C) with conditions shown in section 2.3. The results of these analyses, here shown in terms of Black diagrams, clearly highlight the differences between the two TR-MBs. After 1 hour (Fig. 6) both of them show almost no separation, but already after four hours (Fig. 7) TR-MB HS 210 demonstrates a very high difference in performance between the top and bottom sections. The majority of the rubber tends to settle towards

the bottom and the Black diagram of the top part tends to have the properties of the base bitumen (neat). After 24 hours, (Fig. 8) TR-MB HS180 also shows a big gap between the upper and lower sections, but it is not comparable with the loss in properties of TR-MB HS210, which shows rheology of the top section really close to neat bitumen.

Table 5: Softening points of TR-MBs HS before and after hot storage

Softening point (UNI EN 1427)	TR-MB HS 180		TR-MB HS 210			
	°C					
	top	bottom	top	bottom	ΔSP	
before hot storage	62.0		55.0			
after 1h of hot storage	61.0	63.0	54.0	55.0	2.0	1.0
after 4h of hot storage	59.0	61.5	55.0	59.0	2.5	4.0
after 24h of hot storage	50.0	63.0	50.0	59.0	13.0	9.0

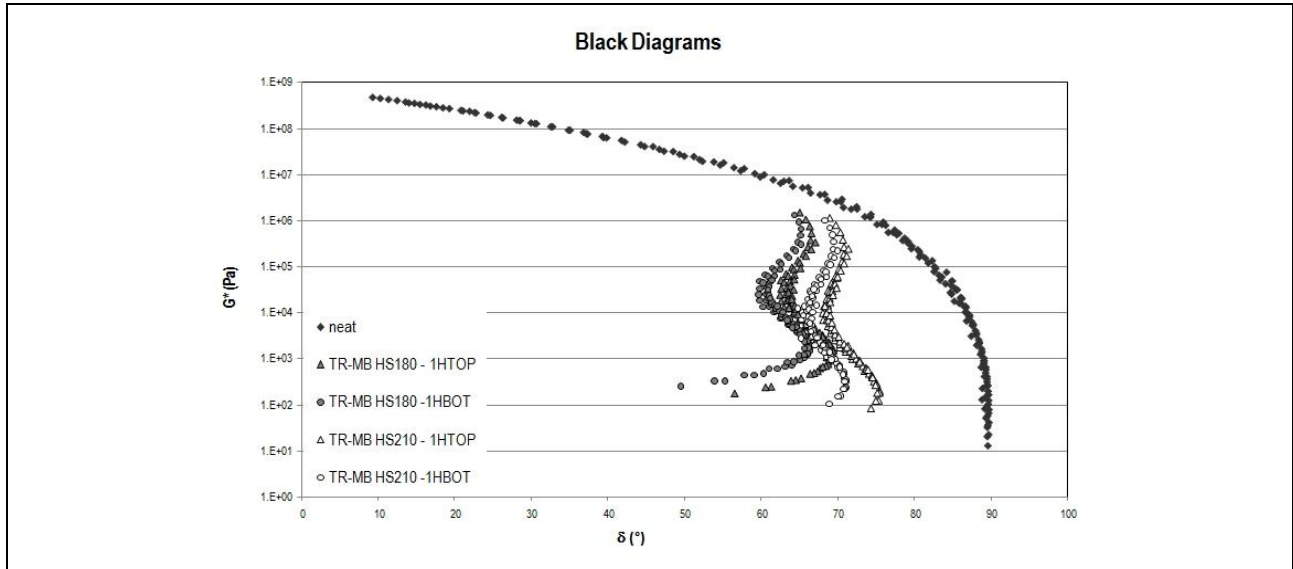


Figure 6. Black diagrams of top and bottom parts of the TR-MBs after 1h of hot storage @ 180°C

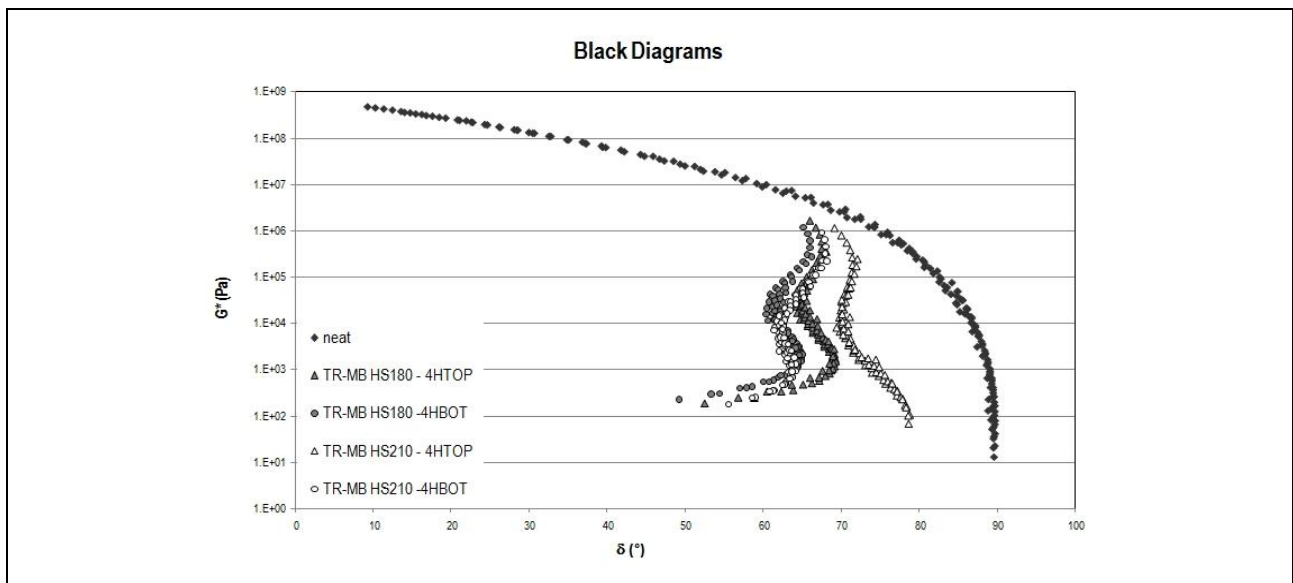


Figure 7. Black diagrams of top and bottom parts of the TR-MBs after 4h of hot storage @ 180°C

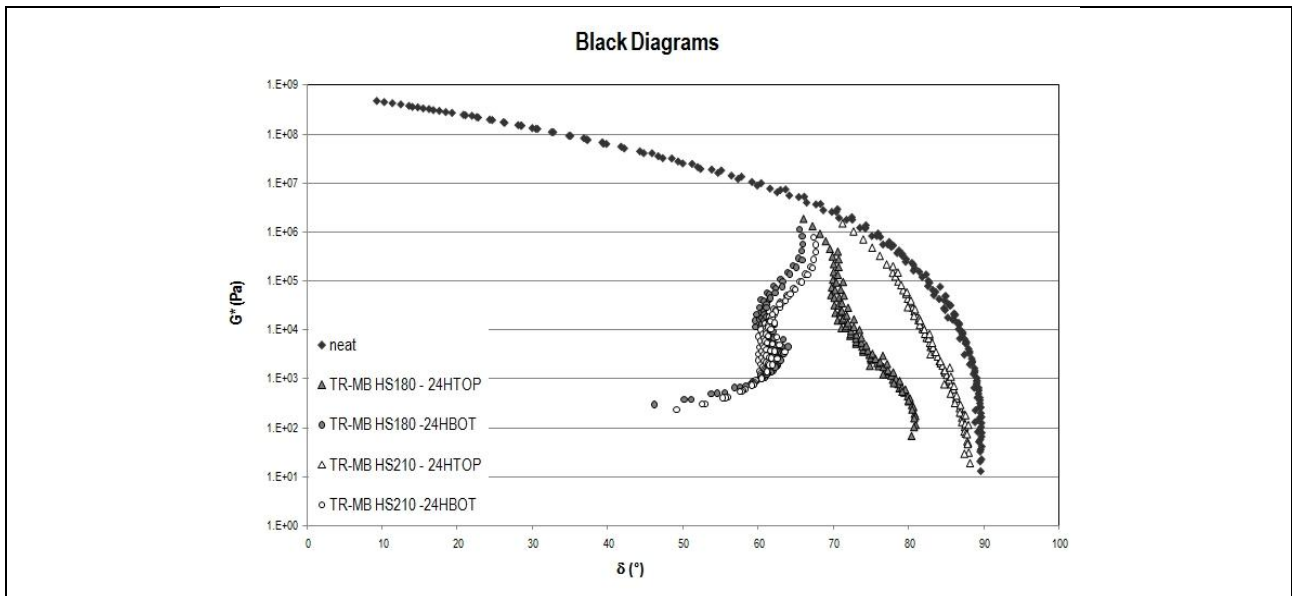


Figure 8. Black diagrams of top and bottom parts of TR-MBs after 24h of hot storage @ 180°C

Table 6. SPs of TR-MBs HS and SBS-MSs after 24h of hot storage

Softening point (EN 1427)	Before hot storage	After 24 hours of hot storage @ 180°C		
		top	bottom	ΔSP
	°C			
TR-MB HS 180	62.0	50.0	63.0	13.0
TR-MB HS 210	55.0	50.0	59.0	9.0
SBS-MB Medium	64.0	66.5	66.0	0.5
SBS-MB Hard	85.0	86.0	84.0	2.0

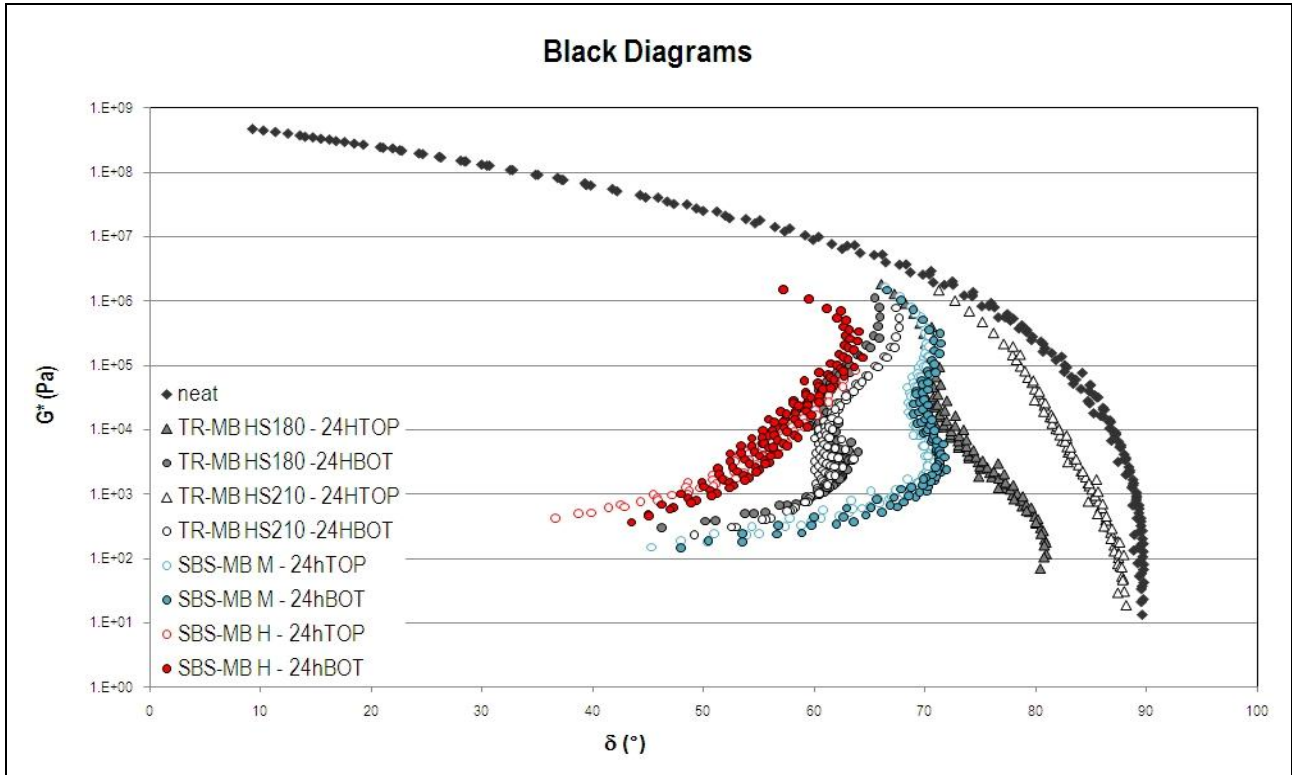


Figure 9. Black diagrams of top and bottom parts of TR-MBs and PBMs after 24h of hot storage @ 180°C

Also in the storage stability analysis the PMBs were taken as a reference. A comparison in terms of conventional and rheological properties, was carried out before and after hot storage. The results of these analyses confirm, as expected, that also after 24 hours the SBS-MBs have got a much better stability than the TR-MBs, in terms of both softening points (UNI EN 13399 prescribes a maximum ΔSP of 5°C) (Tab. 6) and rheology (Fig. 9). Instead, the analysis of the

hot storage stability of the TR-MBs, when based on softening points, did not show a coherent trend. In fact, table 6 shows that TR-MB HS180 seemed to be the less stable. On the contrary, by performing a DMA (Fig. 9) it has been clarified that TR-MB produced at 210°C has a higher separation rate than the one produced at 180°C. Indeed, the difference in terms of Black curves between top and bottom sections of the TR-MB HS210 is much higher than the one measured for the TR-MB HS180.

4. CONCLUSIONS

From these results, it is possible to conclude that raising the processing temperature from 180°C to 210°C significantly speeds up the reaction between TR and bitumen and reduces the peak apparent viscosity. Nevertheless, by choosing the correct TR content and the appropriate mixing time, also at a high processing temperature, 210°C, it is possible to produce TR-MBs with a high level of modification. Furthermore, TR-MBs processed at 210°C proved to be easier to handle and less shear susceptible than TR-MB processed at 180°C. Therefore in terms of viscosity, high processing temperatures and high shear mixing are mandatory for going toward the production of no-agitation TR-MB with the selected materials. However, processing at such high temperature and without using any additive seems to negatively influence the storage stability of the TR-MBs. Another important result is represented by the use of DMA as a tool to establish the storage stability of modified binders. In fact, phase separation that can occur during the storage of modified binders at high temperature is a complex phenomenon that has not been yet fully characterized. Furthermore, empirical tests such as Softening points has not been designed for such complex binders. For these reasons, the authors believe that assessing the storage stability of modified binders through rheological characterization gives a better understanding of the separation process.

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