

Polymer modified bitumen: Optimization and selection [☆]



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ABSTRACT

Polymer modification of bitumen has been commonly performed since the 1980s in order to decrease bitumen (and pavement) susceptibility to high and low temperatures, allowing reduction in common failure mechanisms as rutting and cracking. Bitumen modification has been commonly performed by addition of thermoplastic or elastomeric polymers. However, there are just a few studies on bitumen blends using multiple materials, seeking for specific advantages provided by addition of these modifiers. This work describes the results obtained after the preparation of multicomponent polymer-bitumen blends (MC) based on an 80/100 penetration grade bitumen with varying amounts of (i) Polyethylene wax (PW); (ii) Styrene–Butadiene–Styrene copolymer (SBS); and (iii) crumb rubber (CR). Ideal blends depending on the amount of polymer modifiers added were found by using an experimental design procedure. It was possible to propose charts allowing optimizing and selecting appropriate polymer modified bitumens (PMB) depending on target properties for a given application by following Ashby's materials selection methodology.

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1. Introduction

Optimize and select appropriate materials for specific applications have been the main goals of materials processing and design engineers worldwide for a long time. Polymer modified bitumens (PMB) are not an exception. Several studies have shown that addition of polymers to bitumen aiming at obtaining binary [1–3] or multicomponent blends (MC) [4,5] can improve the final properties of PMB, achieving materials with enhanced properties for applications where pure bitumen by itself would not perform appropriately. Despite of these improvements in properties, it is not common to find studies aiming at optimize the different blends in order to improve properties and reduce common failure mechanisms occurring in asphalt mixtures, while reducing production costs.

The two main failure mechanisms suffered by asphalt mixtures are cracking and rutting (permanent deformation). Cracking is usually associated to brittle fracture of bitumen at low temperatures, while rutting is associated to plastic or viscous behavior of

bitumen at high temperatures. In order to reduce cracking, bitumen has been typically modified by addition of elastomeric polymers; usually requiring mixing and shearing at high temperatures in order to uniformly disperse the polymers into the blends [6–8]. In general, the elastomeric polymers added to bitumen build a physical network assisted by bitumen's phases but do not create a chemical reaction, leaving bitumen as a continuous phase. On the other hand, thermoplastic polymers have been also added to bitumen pursuing improvements on rutting resistance at high temperatures, making use of the hardening effect of these polymers at high temperatures [4,9,10].

Regardless of the method used to characterize the modification levels achieved by different polymer modifiers on PMB, they all generally fall into a certain classification depending on the degree of change in defined property values. These property values will be finally dependent on parameters like amount and kind of each polymer added to the blend. In other words, the diversity of data obtained for each bitumen blend makes necessary to find effective ways to help improving and selecting the most appropriate bitumen blend (in terms of kind and amount of polymer) to obtain specific properties for a given application.

Blend optimization can be achieved by using an experimental mixture design process. These kind of procedures have been proved to be useful on establishing relationships between input (e.g., polymer modifiers and bitumen type) and output variables (e.g., expected properties) [11]. For instance, experimental mixture

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design was used to obtain a complete range of possible morphological states for ternary polymer blends [12] or to develop empirical models to correlate liquefied petroleum gas (LPG) composition to its octane (RON) and motor octane numbers (MON) [13]. Despite of the widespread use of experimental mixture design on different fields of engineering, for the best of the authors' knowledge, there are no studies using this technique to analyze PMBs.

The selection of a PMB for a specific application has been usually done based on engineering experience or on documentation given by manufacturers. Engineering materials selection has been performed during the last years mainly by means of the so called *Ashby plots*, which are graphical representations of material properties on coordinate axes that help on the selection procedure of technical attributes [14,15]. The advantages of these plots are summarized by three main points [16]: (i) help in giving an overview of the physical, mechanical and functional properties of materials, presenting the information in a compact way; (ii) reveal aspects of the physical origins of properties, helpful in understanding the underlying science; and, (iii) become a tool for optimized selection of materials to meet design requirements. These plots have been extensively used in different kinds of materials like natural, polymeric, metallic, composites, etc. However, no attempt has been reported of the use of this selection technique for bitumen or polymer modified bitumens.

The aim of this paper is to study the effect of the addition of three different polymer modifiers to bitumen, and by using an experimental design analysis, be able to find optimal blends for specific properties like penetration (*pen*), softening point (*SP*) and rheological parameters. Using the results obtained for those multi-component polymer-bitumen blends along with results reported by other authors, Ashby plots will be constructed in order to develop selection procedures for PMB, which will be helpful on the selection of materials for specific applications and will even help on deciding which PMB is most cost effective.

2. Experimental procedure

2.1. Materials

An 80/100 penetration grade pure bitumen obtained from Ecopetrol Barrancabermeja's (Colombia) refinery was used in this study for the preparation of all the blends analyzed. This bitumen had the properties and constituents listed in Table 1. The following polymer modifiers were used on the preparation of the different polymer-bitumen blends:

- Styrene–Butadiene–Styrene (SBS) Solprene R, commercial Reference 411, which was polymerized in solution to have a radial structure.

Table 1
Properties and characteristics of base bitumen and polymer modifiers.

<i>Bitumen</i>	
Penetration 25 °C (dmm)	83
Softening point (°C)	46
<i>SARA analysis (%)</i>	
Asphaltenes	12
Resins	36
Aromatics	38
Saturates	14
<i>SBS</i>	
Total styrene (over polymer)	20
Hardness shore A	60
<i>PW</i>	
Viscosity 140 °C (cps)	80
Penetration 25 °C (dmm)	1
Softening point (°C)	130
Melting temperature (°C)	122

- Polyethylene wax (PW), highly crystalline homopolymer, reference THP provided by Alphamin S.A.
- Tire crumb rubber (CR) post-consumer with size particle over mesh 25.

Previous studies have concluded the positive influence of adding these types of polymers on PMB properties. For instance, SBS have been added to bitumen in order to reduce cracking and rutting [6,8,17]; PW have been added to reduce rutting [4,5,9,10]; while CR have been proven to reduce cracking and rutting but at reduced levels to what can be reached with SBS or PW [7,18–20]. Some of the main characteristics of these polymer modifiers are also shown in Table 1.

2.2. Preparation of polymer-bitumen blends

Binary PMB were prepared using a conventional mechanical mixer Heidolph model RZR 2020. The blends were stirred at a speed of 2000 ± 10 rpm during two hours for SBS and CR blends, allowing for molecular swelling of the elastomeric components; while 45 min were necessary to prepare the PW blends. The blending temperature was kept between 180 and 190 °C in all the cases. This temperature ensured that both, polymers and bitumen were always above their softening point temperature (see Table 1). Temperature, mixing speed and blending time were chosen according to conditions reported by previous researchers [5,6]. However, mixing speed and blending time have not been standardized and different researchers have used various mixing conditions to obtain similar results [17,21].

A defined amount of bitumen (approximately 200 g) was heated up to 135 °C in a blending vessel in order to obtain a bitumen fluid enough to be easily stirred. The blending vessel was then heated up to 180 °C and while stirring still in place, the polymer modifier was added in the specified amount and was stirred for the required time period. Four different blends were prepared for each polymer, with weight percentages of 3%, 6%, 9% and 15%, respectively. It is worth mentioning that commercial PMB modified by SBS and PW are usually prepared with amounts up to 6%, while PMB modified with CR use higher amounts (up to 20% in some cases) of polymer modifier. Amounts up to 15% for all the polymer modifiers were used in this study in order to compare the effect of addition of the different materials to the blends. Special attention was kept on avoiding phase segregation of the polymeric components on blends containing high amounts of SBS and PW.

MC, which are blends containing two or three polymers added to the base bitumen, were prepared following a procedure similar to the one previously described to prepare binary blends. The amount of each polymer added into the MC was established following a so-called augmented (3, 2) simplex lattice experimental design [22]. The experimental design contained a total of ten design points for all the blends. The amount of bitumen was kept constant at 85% w/w for all the blends, while the fractions of polymers varied until reaching 15% w/w in total. The graphical representation of this design is shown in Fig. 1. The triangle vertices corresponding to polymer bitumen blends with only one polymer as a modifier (binary blends). The midpoint sides representing ternary MC polymer bitumen blends, while internal points represent quaternary MC polymer bitumen blends. Fig. 1 also shows the fractions of polymer modifiers used for each blend. A total of 10 blends were prepared in this study, and will be subsequently referred to as: multicomponent 1 (MC1), multicomponent 2 (MC2), and so on until reaching MC10. It is worth mentioning here that each of the blends was prepared in triplicate in order to establish repeatability of the results.

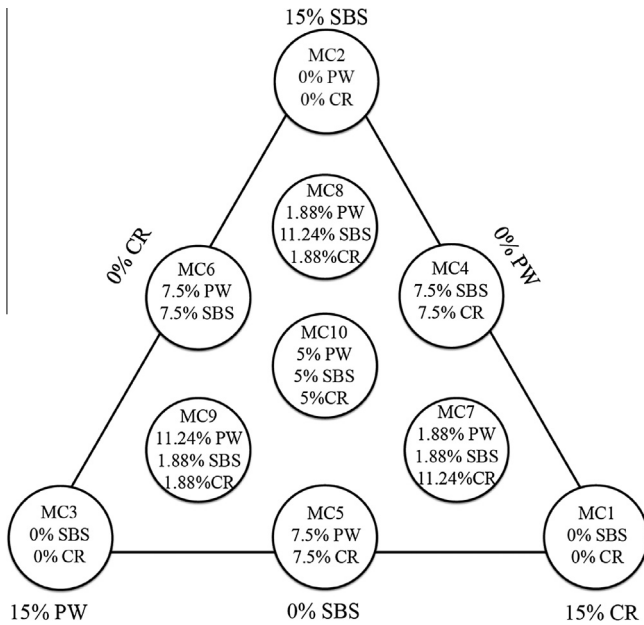


Fig. 1. Graphical representation of the experimental design used in the development of MC polymer-bitumen blends.

2.3. Penetration and softening point

Base bitumen and PMB were analyzed using Penetration and Ring and Ball Softening Point tests. The penetration test uses a standardized needle with a load of 100 g to measure its penetration in the material in a time period of 5 s. The depth of penetration of the needle expressed in units of 1/10 mm indicates the penetration for the analyzed material. The measurement at 25 °C defines the penetration grade of the tested bitumen according to ASTM: D5–13.

SP is defined as the temperature at which a steel ball sitting over a bitumen film produces a deformation of 25.4 mm on bitumen, following the ASTM: D36–12. The SP value can be understood considering its relation to viscosity; it has been established that bitumen's viscosity in its softening point is of about 5000 pa s [23].

The results obtained from penetration and softening point tests can also be used to find the so-called *penetration index (PI)*. This is an important parameter for pavement engineers and can be related to the well-known Van Der Poel [24] nomograph, useful in finding the stiffness of bitumen. A classical approach to calculate the PI is given by [25]:

$$PI = \frac{1952 - 500 \log(\text{pen}_{25}) - 20SP}{50 \log(\text{pen}_{25}) - SP - 120} \quad (1)$$

where pen_{25} is the penetration at 25 °C and SP is the softening point temperature for the material under consideration. Despite of the fact that PI is mainly used to classify pure bitumen, in this work PI was used as a mean of comparison of the effect of polymeric modification on the different blends. Further, these results will show to be useful in the construction and interpretation of material selection charts.

2.4. Rheological measurements

Rheological measurements were performed on base bitumen and PMB by means of a strain controlled Kinexus rheometer. The frequencies used were in the range between $1 \times 10^{-2} < f < 10^2$ rad/s. Tests were carried out within the linear viscoelastic regime using a parallel plates configuration with 25 mm in

diameter, at a constant temperature of 40 °C. This temperature was chosen as it is considered to be representative of medium-high working temperatures for bitumen. Complex shear modulus (G^*) curves were used to obtain the rheological parameters R_p and ω_p for each of the blends using a power law of the form:

$$G^* = \frac{G_g}{\omega_p} (\omega)^{R_p} \quad (2)$$

where G_g is the glassy modulus, commonly taken as 1 GPa for most bitumens [26]; ω_p is a material constant related to the crossover frequency, which is the frequency where bitumen switches from viscous to elastic behavior; and R_p can be understood as the rheological index. Furthermore, both parameters can be related to Christensen–Anderson's model parameters, which is a model widely used to describe the rheological behavior of bitumen and PMB [27,28].

3. Results and discussion

SP temperatures measured for each of the blends studied are shown in Fig. 2. Binary blends SP values are found in the vertices of the triangle, with MC3 (15% PW) having the highest SP temperature measured for these blends, reaching up to 120 °C. It was expected a considerable increase in the SP with the use of this modifier as it has a melting temperature of around 125 °C (see Table 1). Furthermore, binary blends MC2 (15%SBS) and MC3 (15% PW) increased the SP of base bitumen in excess of 140%, while for blend MC1 (15% CR) the increase in SP was just of around 30%. The works of Da Silva et al. [2] and Navarro et al. [7] found that for PBM blends with additions of up to 9% of polymer modifier the SP values were in the range between 70 and 80 °C, which are values lower than what were achieved with the MC blends of this study. However, blends MC1, MC4 and MC7 showed to have lower values of SP due to their high content of CR. The small level of modification achieved with CR is due to the interaction of this modifier with bitumen, which basically act as a filler, and only a small fraction of the three dimensional network of the vulcanized rubber ended-up dissolved in the bituminous binder [7,20]. Despite of this, CR is considered as an environmentally friendly bitumen modifier and this is why its use is widespread [7,19,20].

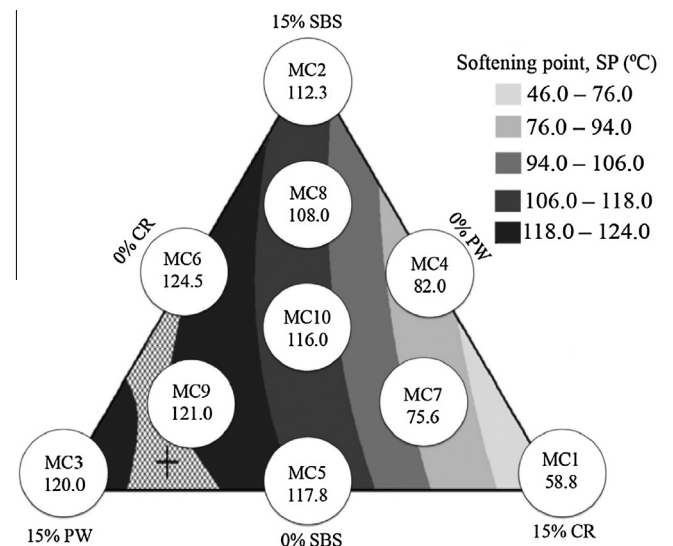


Fig. 2. Contours of SP temperatures measured for the different polymer-bitumen blends. The cross indicates an optimal blend with 1% SBS + 2.7% CR + 11.3% PW + 85% bitumen, to get an estimated SP = 124.5 °C.

The SP results for ternary and quaternary blends shown in Fig. 2 make evident the small effect of increased amounts of CR on SP, while increasing amounts of PW and SBS considerably increased the SP values of the blends. Increase in SP due to SBS additions has been related to the effective interaction between this material and the oily components of bitumen. By using Transmission Electron Microscopy (TEM), it has been observed that SBS suffers a swelling inside bitumen. Further, at contents higher than 5% it has been found to form a tridimensional continuum SBS rich matrix, behaving at this point as an elastic material [6]. Similarly, other researchers have related these interactions with thermal properties changes as shifting Tg [17] or even with introduction of new Tg's attributed to SBS rich phases [8].

A quadratic model was used to fit the experimentally measured values of SP for all the blends in order to establish the variation of this property depending on the amount of modifiers added; using a fixed amount of bitumen of 85%, getting (see Table 2):

$$SP[^\circ C] = 7.4(SBS) + 7.9(PW) + 3.7(CR) + 0.16(SBS)(PW) + 0.52(PW)(CR), \quad (3)$$

with SBS, PW and CR representing the amount of polymer modifiers in weight percentage, reaching a total of 15% combined. According to this equation, the effect of addition of SBS and PW on SP is similar. Furthermore, this equation was used to find the blend that can maximize this property, which is indicated in Fig. 2 by a cross. Therefore, Eq. (3) allows for the development of MC blends depending on a required SP, enabling even for an increased use of CR, in order to reduce costs.

Fig. 3 shows the variation of the measured values of penetration for the different blends. It can be seen that increased amounts of PW reduce the measured values of penetration, reaching values as low as 14 dmm for blend MC9 (11.2%PW + 1.9%SBS + 1.9% CR). A linear model was used to fit the experimental values of penetration for the multicomponent blends as (see Table 2):

$$pen_{at 25^\circ C [dmm]} = 0.75(PW) + 1.9(SBS) + 3.9(CR). \quad (4)$$

According to this, the effect of PW on the reduction of penetration is significantly higher in comparison to what can be achieved by using SBS and CR.

These results are in agreement to results reported by other researchers who obtained *pen* values of around 30 and 20 dmm with addition of PW in excess of 3% [2,28]. These results were attributed to the hardness of these materials based on Polyethylene at room temperature. Eq. (4) can also be used in the development of MC blends depending on the required penetration level.

Fig. 4 shows the evolution of complex moduli (G^*) with frequency at a temperature of 40 °C for base bitumen and the different blends. In general, the polymer modifiers increased the values of G^* in comparison to base bitumen, while the slope of the curves was reducing. Multicomponent blend MC9 (11.2% PW + 1.9%SBS + 1.9% CR) showed to be the one with the most considerable change in G^* and slope, indicating increased elastic and stiffness behavior in comparison to the remaining blends. It has been suggested in previous studies that PW molecules improve bitumen flow at temperatures over 122 °C, which is PW melting temperature, causing a reduction in viscosity. This is used as an

Table 2
Statistical information of the fitting parameters for the MC polymer-bitumen blends.

Parameter	Model	p-value	R ²
SP	Quadratic	0.017	98.4
pen	Linear	0.001	88.4
R _p	Quadratic	0.036	93.9
w _p	Linear	0.001	84.7

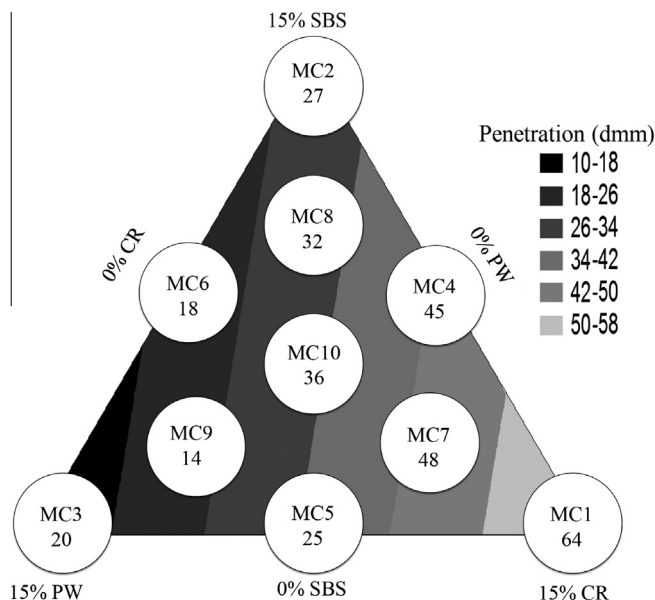


Fig. 3. Contours of penetration measured for the different polymer-bitumen blends.

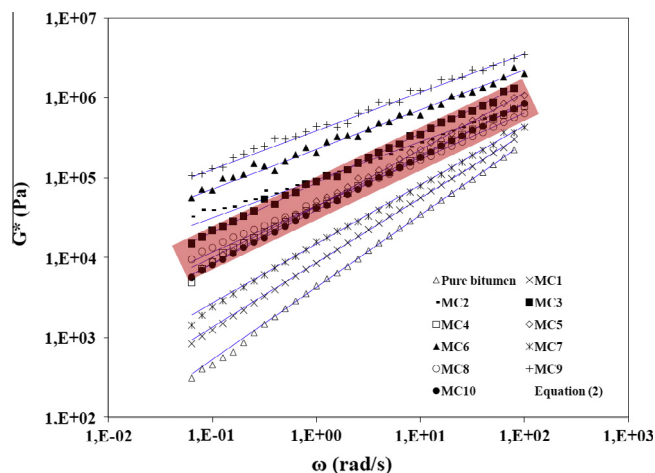


Fig. 4. Evolution of complex modulus depending on frequency at a temperature of 40 °C, for base bitumen and polymer-bitumen blends.

advantage, allowing reduction of temperature and energy consumption during asphalt mixture production. Nonetheless, at temperatures as 40 °C, PW is extremely hard in comparison to base bitumen. Additionally, it has been reported that addition of PW significantly increases the failure temperature of rubberized binders, pointing towards better resistance to permanent deformation at high temperatures [5].

The shaded region in Fig. 4 corresponds to blends with high amounts of SBS. In this region G^* does not vary considerably. This behavior had been identified by Chen et al. [6], who argued that SBS-bitumen blends with SBS in excess of 6% form a SBS rich phase, making that SBS increases by small amounts the measured values of G^* . However, MC6 shows that addition of PW increases the values of G^* .

Fig. 5 shows the results obtained for the rheological index R_p (see Eq. (2)) of the blends. This index varies between $0 < R_p < 1$, with low values (low slope in Fig. 4) indicating materials with elastic behavior, while high values (high slope in Fig. 4) indicate materials with viscous behavior. It can be seen that blends with high

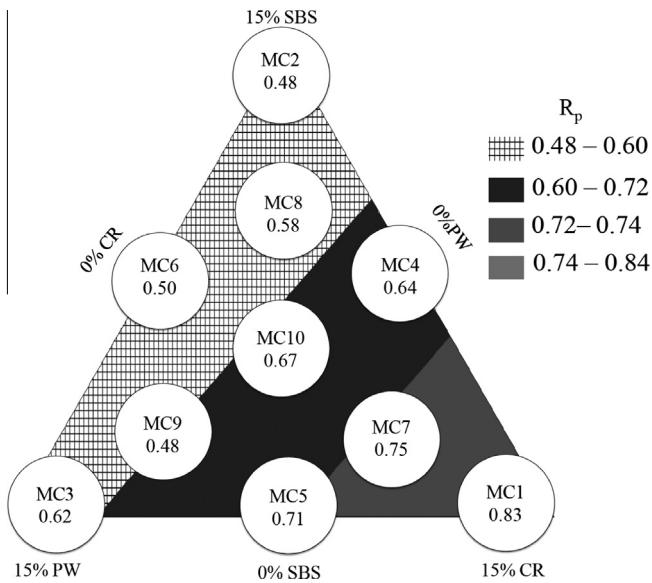


Fig. 5. Contour plot of the rheological index R_p measured for the different polymer-bitumen blends.

content of CR (i.e. MC1 and MC7) tend to increase the values of R_p , while blends with high content of PW and SBS (i.e. MC2 and MC6) tend to give reduced values. A linear model was used to fit the experimental results of the rheological index for the different blends as (see Table 2):

$$R_p = 0.033(\text{SBS}) + 0.038(\text{PW}) + 0.055(\text{CR}). \quad (5)$$

Note that increasing amounts of SBS will reduce the final value of R_p (increase elastic behavior) in comparison to the effect of the other two modifiers.

Fig. 6 on the other hand, shows the results obtained for the cross over frequency ω_p of the blends (see Eq. (2)). This parameter also indicates if the behavior of the material tends to be elastic (low values) or viscous (high values). It can be seen, as for R_p , that blends with high content of CR (i.e. MC1 and MC7) tend to increase the values of ω_p , while blends with high content of PW and SBS (i.e. MC6 and MC9) tend to give reduced values of this parameter.

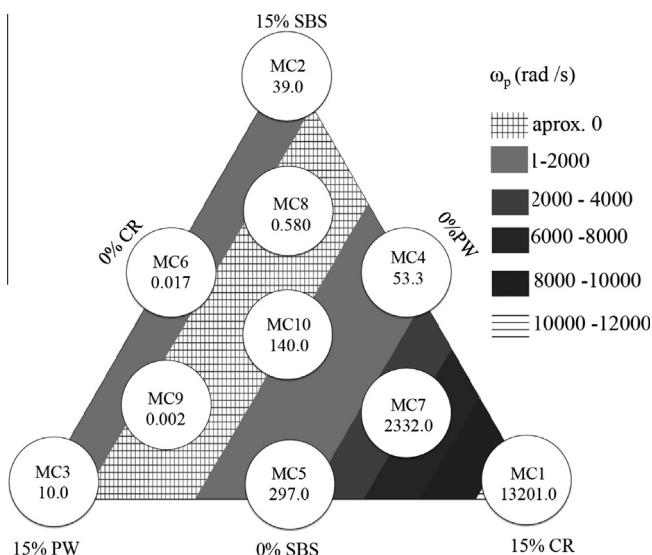


Fig. 6. Contour plot of the measured crossover frequency ω_p for the different polymer-bitumen blends.

A quadratic model was used to fit the experimental results of the multi component blends as (see Table 2):

$$\omega_p = 32.9(\text{PW}) + 34.4(\text{SBS}) + 805.7(\text{CR}) - 106.5(\text{CR})(\text{PW}) - 110.7(\text{CR})(\text{SBS}). \quad (6)$$

4. Selection procedure

Previous sections described the experimental procedures and results obtained for binary and MC, finding equations that allow prediction of material properties depending on the amount of polymer modifiers added to pure bitumen. However, in materials selection processes it is customary to optimize materials depending on different properties or characteristics that the end user require to maximize (e.g.: SP , PI , etc.) or minimize (e.g.: pen or $cost$). This kind of selection process is well established by using the so-called Ashby methodology (see for instance: [14–16,29]), where one or two properties are presented on a coordinate system with the ranges of variation of these properties plotted. In this way it is possible to get a graphical aid to select the best material for an intended application. Plots for selecting PMB, based on the results previously shown will be presented in the following sections, with the corresponding explanations on how to select the materials based on these plots.

4.1. Softening point – penetration chart

Fig. 7 shows the material property chart relating penetration and softening point for the materials studied, along with results found in other studies for binary polymer-bitumen blends, also using an 80–100 pen bitumen [6,29–32]. To the right and bottom of the plot can be found materials soft and temperature susceptible (e.g. Pure bitumen), while at the left and top of the plot can be found materials harder and less temperature susceptible (e.g. MC blends). It can be seen that the main effect of CR on the blends is to harden the material (reduce penetration), while the change in softening point is not significant in comparison to other modifiers. Additions of SBS increase hardness and softening point of the blends in a significant way, as is the case for additions of PW. MC showed to modify these properties, in some cases, better than what can be achieved with single additions of PW, CR or SBS.

Fig. 7 also includes guidelines representing various penetration indexes, PI (Eq. (1)). PI is commonly used for classifying the rheological behavior of bitumen based on two basic properties (pen

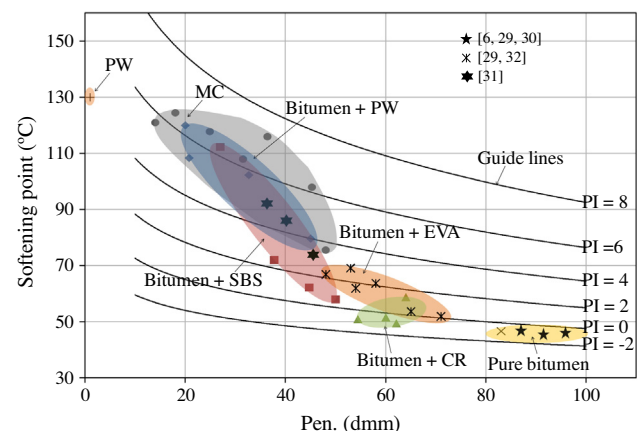


Fig. 7. Material property chart for penetration (pen) and softening point (SP) for the materials studied, along with results from other researchers [6,29–32], penetration index (PI) guidelines are also shown.

and SP). PI varies between approximately -2.0 and 8.0 for all the bitumen blends studied, but typically lies between -2 and $+2$ for paving grade bitumens. However, PMB with higher values of PI can be used in roofing applications, which represents approximately a 10% of the bitumen usage in Europe [33]. Furthermore, It has been recognized that bitumens with the same PI have similar rheological master curves [17]. Therefore, it is possible from this plot to select different modifiers in order to obtain materials with similar rheological behavior. For instance, it is possible to obtain blends with $PI = +6$ using PW or SBS binary blends or with multi-component blends. This last option can have reduced prices in comparison to binary blends, as will be discussed later.

4.2. Rheological properties chart

Fig. 8 shows the property chart for the rheological index (R_p) and cross over frequency (ω_p) for the blends studied. To the left of this chart can be found materials with increased elastic characteristics, while on top of it can be found materials increasingly viscous. Further, the slope of this plot can give a guide of the complex moduli (G^*) of the materials, as shown in the figure, noting that the lower the slope, the higher the modulus. The different modifiers reduce the viscous behavior, while increase the elastic response. Additions of SBS tend to have a higher reduction on the viscous behavior than the other modifiers. However, with multi component blends it is possible to reach better elastic responses than with binary blends.

4.3. Penetration index – cost chart

Fig. 9 shows the chart combining penetration index (PI) and relative cost of material per tonne for the different polymer – bitumen blends studied. This chart is important in the selection process as it gives a clear indication of the cost involved in obtaining a desired PI depending on the polymer modifier to be used in the production process.

Note that by using CR it is possible to keep the cost as low as possible, but the level of modification on PI is not as important as for the other two modifiers studied (SBS and PW). This chart also shows that it is possible to obtain blends with high PI at reduced costs by means of MC.

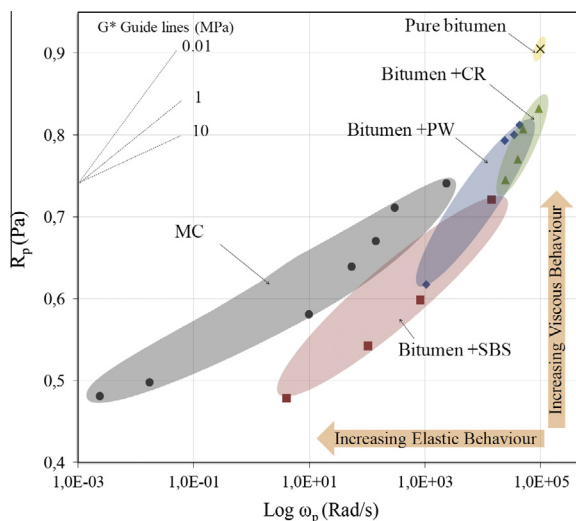


Fig. 8. Material property chart for rheological parameters R_p and ω_p . Guidelines for complex modulus are also shown.

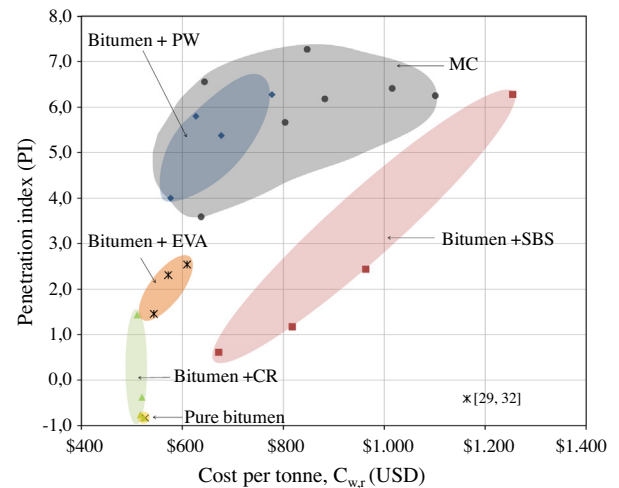


Fig. 9. Material property chart for penetration index (PI) and relative cost per tonne ($C_{w,r}$). Results from other researchers are also included [29,32].

Using Eqs. (3) and (4) to calculate PI (Eq. (1)), it is possible to plot a chart showing penetration index versus price for the different polymer-bitumen blends described in previous sections, as shown in Fig. 10. This chart presents a surface of possible combinations of polymer modifiers to obtain target PI 's and prices for blends with 85% of bitumen with a total of 15% of the remaining polymers, going from binary to multicomponent blends. The corners of this surface represent binary blends, with the black triangle representing a blend with 15% CR + 85% bitumen; the black star representing a blend with 15% PW + 85% bitumen; and the black square representing a blend with 15% SBS + 85% bitumen. The edges of the surface represent blends with two polymer modifiers, with the solid thick blue line representing the line where the blends have no PW; the thick black line represents blends with no SBS added; and the thick red line represents blends with no CR. Inside the surface can be found quaternary blends, which are defined by the guidelines showing amounts of each of the polymer modifiers. It is worth noting that this surface also allows obtaining two different blends to get the same PI and price in the region shown in Fig. 10b. For instance, the dashed lines in Fig. 10 indicate a target $PI = +6$ with a price of approximately USD 910

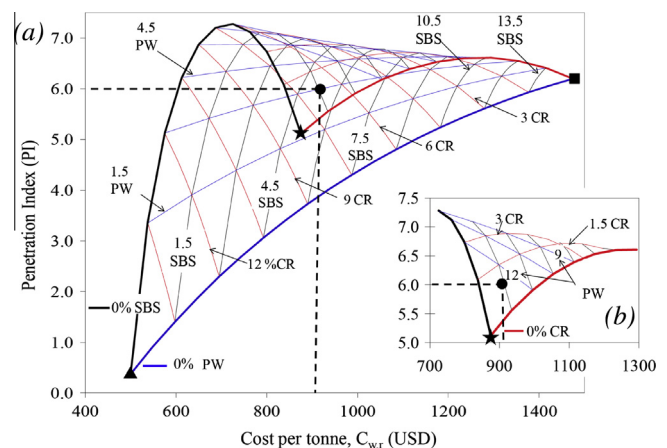


Fig. 10. Material property surface for penetration index (PI) and relative cost per tonne ($C_{w,r}$) for the different blends studied. The surface allows selecting specific blends depending on target PI and Cost. The black triangle represents a blend with 15% CR + 85% bitumen; black star representing a blend with 15% PW + 85% bitumen; and black square representing a blend with 15% SBS + 85% bitumen.

per tonne. This can be obtained by using: (i) 6.8% CR + 3.0% PW + 5.2% SBS + 85% bitumen; or (ii) 1.0% CR + 1.5% PW + 12.5% SBS + 85% bitumen, by using the top portion of the surface shown in Fig. 10b. This chart can be then used to select and optimize polymer modified bitumen blends depending on specific restrictions imposed by the user.

5. Conclusions

Multicomponent bitumen blends (MC) were studied and characterized by penetration, softening point and rheological tests. The results were used to develop response surfaces of each property as ternary plots, depending on the amount of polymer modifiers employed. These plots showed the ability to optimize multicomponent polymer-bitumen blends. This procedure served to obtain the necessary amounts of each polymer modifier into the multicomponent blends to reach precise values of specific properties. This procedure can also be useful for the modified asphalts industry to obtain the best blends with the higher possible amount of CR, in order to reduce costs. Moreover, materials selection charts were presented in this work to help in the selection process of polymer modified bitumens depending on the intended application and material properties. These charts served to present an overview of traditional (softening point and penetration) and rheological bitumen properties. In addition, the penetration index – relative cost chart was useful to identify cheap materials with improved properties. Furthermore, these diagrams enable to obtain proper multicomponent blends for several requirements like penetration, softening point or rheological characteristics.

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