LINEAR VISCOELASTIC PROPERTIES OF HIGH RECLAIMED ASPHALT CONTENT MIXES WITH BIOBINDERS

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The use of high Reclaimed Asphalt (RA) content mixtures together with binders produced from renewable resources (biobinders) is one of the current challenges in pavement engineering research. On one hand, RA has been used for decades but there are still some concerns about its performance, especially when high contents are used (>30%). On the other hand, biobinders are relatively new materials which have to be deeply characterised and studied in order to develop good-practices for their use. In this paper, linear viscoelastic properties of biobinders and bio-mixtures manufactured with high-RA content and biobinders are analysed and discussed. High-modulus mixtures with 50% RA were selected for the mix design. Binders and mixtures were tested over a wide range of asphalt service temperatures and frequencies by means of DSR and two-point bending tests respectively. Results show that biobinders have an important effect on mixtures behaviour. However, no direct links between their linear viscoelastic properties were found. Bio-asphalt mixtures still need further development for commercial exploitation; however the take-away fact of this investigation is that it is possible to manufacture asphalt-like mixtures with acceptable viscoelastic properties while being composed only of RA and non-petroleum based binders.

Keywords: reclaimed asphalt; high content; biobinders; mixture; viscoelasticity

1. Introduction

The use of high Reclaimed Asphalt (RA) content in new asphalt mixtures is currently considered as one of the main alternatives for the reduction of raw materials consumption in pavements. However, the introduction of high RA percentages in mixtures implies some concerns specially related to the aged binder that it contains. Binder in RA has been exposed to climate changes and traffic loads during its service life in a road. Due to this fact, RA binder suffers changes of its initial properties becoming stiffer and more brittle (Hajj et al., 2009; Mogawer et al., 2012). Therefore, some considerations have to be taken into account to use it in high amounts while avoiding compromising the new mixture performance.

For this purpose, when high RA contents (>30%) are used, the fresh binder to be added should be soft (high penetration grade), or rejuvenating agents or fluxes should be used in order to be able to restore some of the properties of the RA binder (Lo Presti et al., 2016; Silva et al., 2012; Zaumanis et al., 2014). Nevertheless, these techniques still imply the use of petroleum-based resources that are non-renewable. Therefore, a relatively new approach for this issue is to use alternative materials as fresh binders in high-RA content mixtures, which can take advantage of the bituminous binder already present in RA while avoiding the use of petroleum-based resources.

Bio-based materials are one of these alternatives and they have already shown good potential to be introduced in asphalt mixtures (Aflaki et al., 2014; Fini et al., 2012; Mohammad et al., 2013). Binders made from renewable resources can be termed biobinders and they can have very different origins. In this sense, every biobinder should be fully characterised before being used in the field in order to provide confidence in its use. This characterisation should show that biobinders' behaviour is suitable for being used in asphalt mixtures and to some extent, similar to that of bitumen or bituminous binders (Pouget & Loup, 2013).

Within this framework, the objective of this paper is to show the linear viscoelastic properties of biobinders used in high-RA content mixtures in order to provide a further step for their characterisation and use. For this purpose, two biobinders and a reference one were rheologically tested at binder and mixture level including 50% RA. Data was fitted with mechanistic models to obtain and compare their linear viscoelastic properties.

2. Materials

2.1. Binders

RA binder was recovered from a RA source in France according to EN 12697-4:2005 Fractionating Column by Distillation. Two bio-materials were considered as binders, namely: BioBinder (BB) and Biophalt® (BP). BB is a binder produced from the blend of pine resin (80% in mass) and linseed oil (20% in mass). These resin/oil proportions were adjusted in order to obtain a final blend with the RA binder equivalent to a 50/70 penetration grade binder (Jimenez del Barco Carrion et al., 2016). Biophalt® is a vegetal binder containing polymers and patented by Eiffage Company. The control binder for reference was selected as a high-performance binder in order to compare the bio-materials with high-quality binders. Therefore, the reference binder (Ref) is a cross-linked polymer modified binder which is used in practice in France to recycle bituminous pavement, especially for large motorway projects.

Biobinders as well as the reference binder were blended in the laboratory with the binder extracted from the selected RA (according to EN 12697-4:2005), which has a binder content of 3.6% (according to EN 12697-1:2012). The blends' compositions were determined by calculating the percentage that would be present in 50% RA mixtures with a 5% total binder content and assuming full blending (all RA binder in the mixture would activate and blend with the fresh binders). This blending assumption was made taking into account previous studies which showed that when high RA amounts are added to a new mix, high blending between fresh and RA binders may occur (McDaniel et al., 2012; Shirodkar et al., 2011; Soleymani et al., 2000). Nevertheless, the actual blending percentage remained unknown at this point of the investigation and the blends produced could be the different than the final binder present in the mixture. In this regard, the percentage of fresh binder that is replaced by RA binder in the mixture is known as

Replaced Virgin Binder (RVB) (Jiménez del Barco Carrión et al., 2015) and is calculated as shown in Eq. (1).

$$\mathrm{RVB}\ (\%) = 100 \cdot \frac{\mathrm{RA} \cdot \mathrm{DOB} \cdot \mathrm{RABC}}{\mathrm{BC}} = 100 \cdot \frac{0.5 \cdot 1 \cdot 0.036}{0.05} = 36\% \qquad (1)$$

Where, RA is the total RA percentage in the mixture by weight, DOB is the assumed degree of blending between RA and virgin binders (100%), RABC is the RA binder content determined in the laboratory and BC is the designed final binder content in the mixture, with all the parameters expressed in decimals. Thereby, blends of the different binders and RA binder were composed of 36% RA binder and 64% of BB, BP and Ref respectively in mass. Conventional characterisation of the materials is shown in Table 1.

Table 1. Conventional characterisation of binder and blends.

BINDER	**NAME**	**PENETRATION @ 25° (dmm)** (EN 1426, 2007)	**SOFTENING POINT (°C)** (EN 1427, 2007)
BLENDS' COMPONENTS			
Reclaimed asphalt binder	RA	9	75.8
BioBinder (80% pine resin + 20% linseed oil)	BB	235	40
Biophalt®	BP	147	73.5
Crosslinked polymer modified binder	Ref	85	67
FINAL BLENDS: 36% RA + 64% Fresh binders			
Reclaimed asphalt binder plus BioBinder	RA+BB	69	45
Reclaimed asphalt binder plus Biophalt®	RA+BP	63	62
Reclaimed asphalt binder plus crosslinked polymer modified binder	RA+Ref	37	70

2.2. Mixtures

50% RA mixtures were manufactured and studied in the laboratory. Mix design was selected as GB5®, which stands for "Graves Bitumes 5". These types of mixtures were recently developed to optimise aggregates' packing by maximising their interlock and allowing for high complex modulus and improved fatigue and rutting resistance (Olard & Pouget, 2015). This mix design was chosen in order to provide a strong mineral skeleton to produce high-performance mixtures with high-RA content and biobinders.

Three mixtures were manufactured with 50% RA content by mass and BB, BP and Ref as the only fresh binder respectively. Mixtures were termed "Mix BB", "Mix BP" and "Mix Ref" depending on the fresh binder used. Total binder content in the three mixtures was 5%. Figure 1 shows GB5® mixtures' gradation.

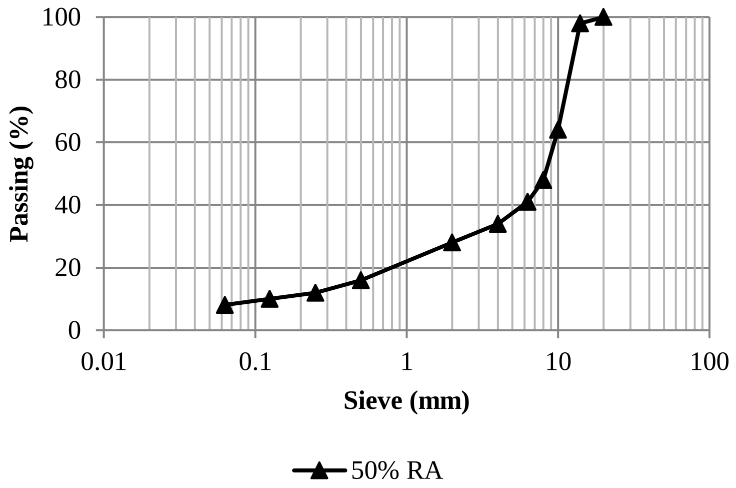

Figure 1. GB5® 50% RA mixtures' gradation.

3. Tests and modelling

3.1.Rheological testing

Blends' components and final blends described in Table 1 were tested in the Dynamic Shear Rheometer (DSR) to obtain their shear complex modulus (G*) and phase angle (δ). Temperature and frequency sweeps were carried out from -10 to 60°C and from 1 to 25Hz. Due to the wide range of temperatures used, two parallel-plates geometries were used in the DSR: 8mm (from -10 to 30°C) and 25mm (from 20 to 60°C). The correct data for each binder at 20°C and 30°C were then selected using their Black diagrams (Airey, 2002).

After manufacture and compaction, mixtures were cut to trapezoidal specimens and tested in two-point bending mode to find their complex modulus (E*) and phase angle (δ). Temperature and frequency sweeps were carried out from -10 to 40ºC and from 1 to 40Hz (EN 12697-26:2012 Annex A).

3.2. Blends and mixtures modelling

RA's blends and mixtures were modelled using the 2S2P1D (2 Springs, 2 Parabolic elements, 1 Dashpot) (Di Benedetto et al., 2004; Olard & Di Benedetto, 2003). This model is able to reproduce binders' viscoelastic behaviour through a combination of one spring (E_∞), two parabolic creep elements (0<h<k<1) with one coefficient (δ) that regulates the balance between them, a dashpot (β) placed in series with the other elements, and finally a spring in parallel to the previous system (E_0) (Figure 2). Model parameters for the blends were fitted from the rheological tests performed in the DSR, while data from two-point bending tests and relative master curves at T_{ref}=0°C were used to obtain the model's parameters for the mixtures (Chailleux et al., 2006). Furthermore, in the case of blends, G* data from the DSR were converted to E* using Poisson ratio of 0.5 (Whiteoak & Read, 2003), which in turns allowed obtaining E_∞. This conversion was carried out in order to be able to further relate the binder's parameters with those derived from the mixtures modelling.

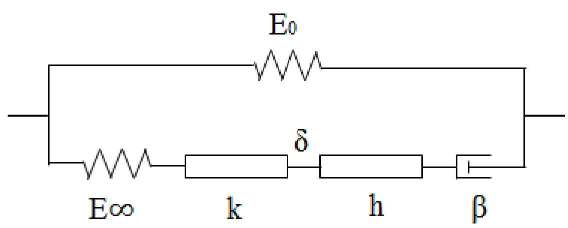

Figure 2. 2S2P1D model.

4. Test results and discussion

4.1.Binders

Figure 3 shows the results from frequency and temperature sweeps of the binders in the Black space. This representation allows all binders, even BP and Ref containing polymers to be seen as thermo-rheologically simple due to the overlap of the data at the different temperatures (-10 to 60ºC) and frequencies (1 to 25Hz). This fact allows master curves and modelling to be undertaken. In addition, in Figure 3 the effect of polymers is noticeable due to the increased elastic behaviour at high temperatures in the case of BP (decrease of phase angle after peak), and the plateau region observed for the Ref. On the other hand, BB is a non-elastic material that reaches high phase angles for most of the test temperatures. This could be expected due to BB's composition (resin+oil). Figure 4 displays binders' Cole-Cole diagram which highlights the high viscous component (E'') of BB in comparison to RA, BP and Ref which show a more similar balance between their elastic (E') and viscous (E'') components.

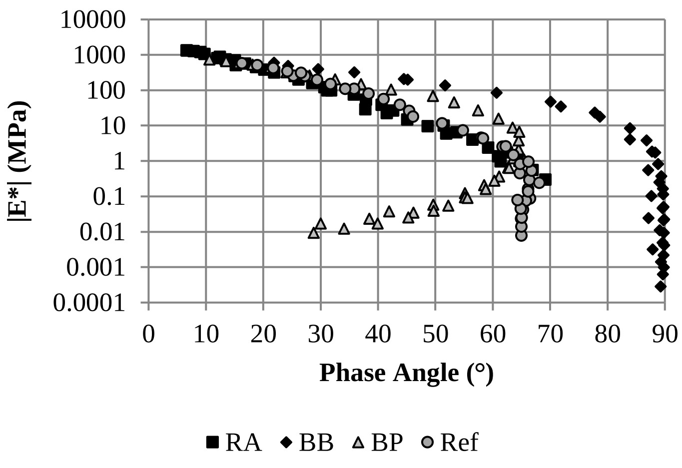

Figure 3. Binders' Black diagram.

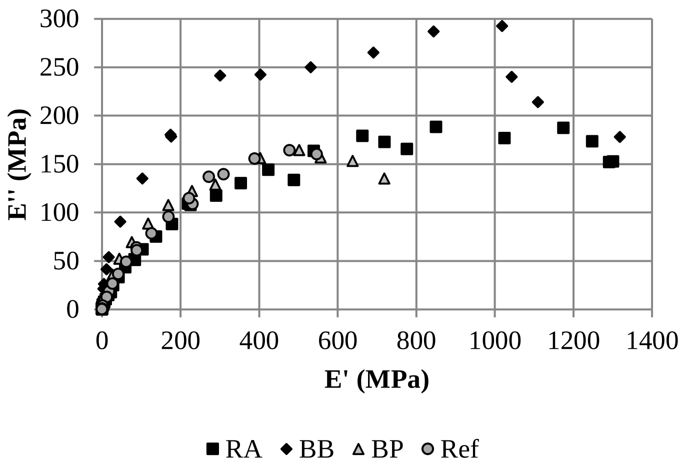

Figure 4. Binders' Cole-Cole diagram.

4.2.Blends

Figures 5 and 6 display the results of frequency and temperature sweeps of the blends of RA and the different binders in the Black and Cole-Cole diagrams respectively. Black diagrams show that the blend of BP and Ref with RA reduced the effect of polymers shown in Figure 3 as a consequence of the reduction of the polymers' content in the blends. Therefore, the increased elastic response of the phase angle and the plateau value of the original BP and Ref at high temperatures are less pronounced. In the case of BB, its blend with RA increases its elasticity reducing the phase angle shown in Figure 3 for the same level of complex modulus. Cole-Cole diagrams reveal that RA+BB maintains a high viscous component as BB showed in Figure 4 even after blending with RA, while RA+BP and RA+Ref exhibit comparable diagrams. In summary, Figures 5 and 6 provide two main ideas: firstly, the blend of RA with the three binders produces an increase in RA viscous behaviour, which could be considered as viscoelastic properties' rejuvenation; secondly, under binders' full blending assumption, mixtures with BP and Ref could have similar rheology while the mixture with BB would be different.

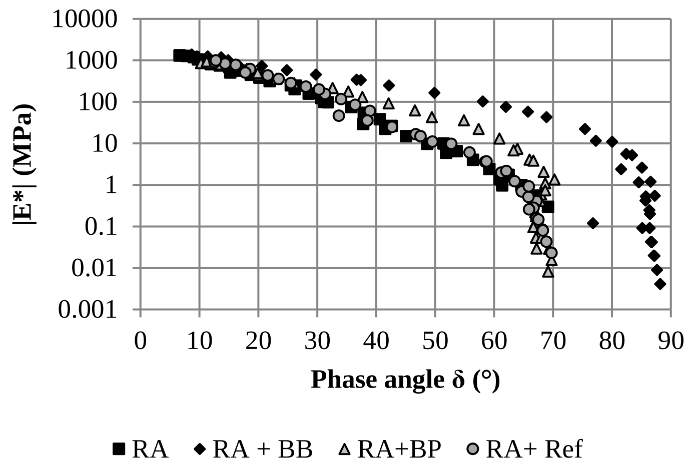

Figure 5. RA and blends' Black diagram.

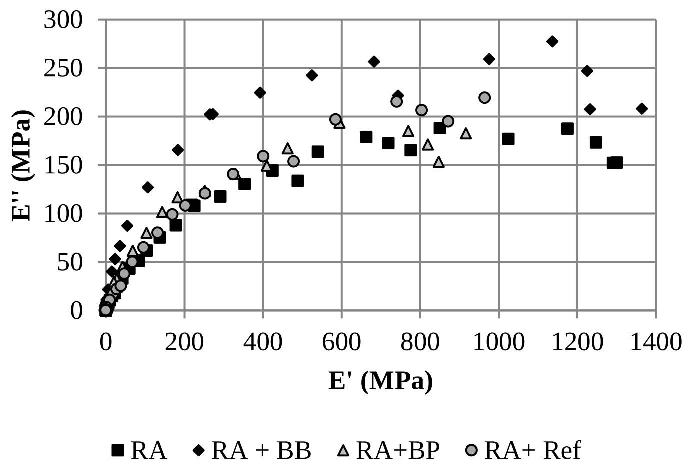

Figure 6. RA and blends' Cole-Cole diagram.

4.3.Mixtures

Figures 7 and 8 display Black and Cole-Cole diagrams of the three 50% RA content mixtures after two-point bending tests. These diagrams provide different results to what could be expected from the blends' results. Black diagram in Figure 7 shows that Mix BB and Mix BP have a comparable behaviour at high temperature. Furthermore, Cole-Cole diagrams of the three mixtures are different, with Mix BB and Mix BP being closer to each other than to Mix Ref. These facts are not in accordance to those showed for blends and could be due to partial blending between RA and binders in the mixture or to changes that the biobinders could have suffered during the manufacturing process. In this regard, it has been shown by different authors (Fini et al., 2015; Yang et al., 2015) that biobinders could age faster than conventional binders depending on their composition and therefore they could exhibit noticeable modifications on their properties during manufacturing.

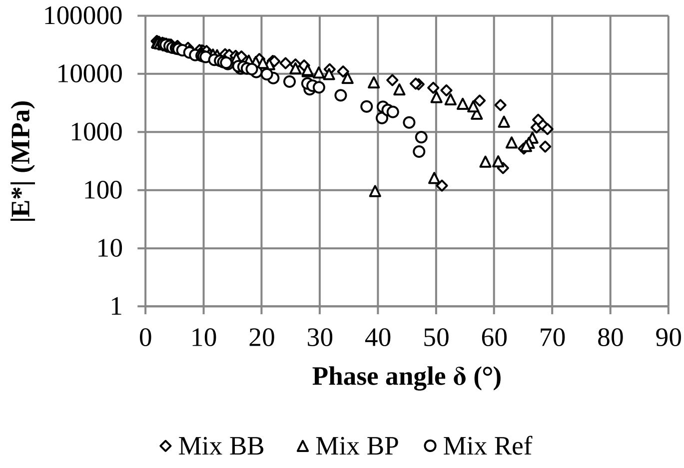

Figure 7. Mixtures' Black diagram.

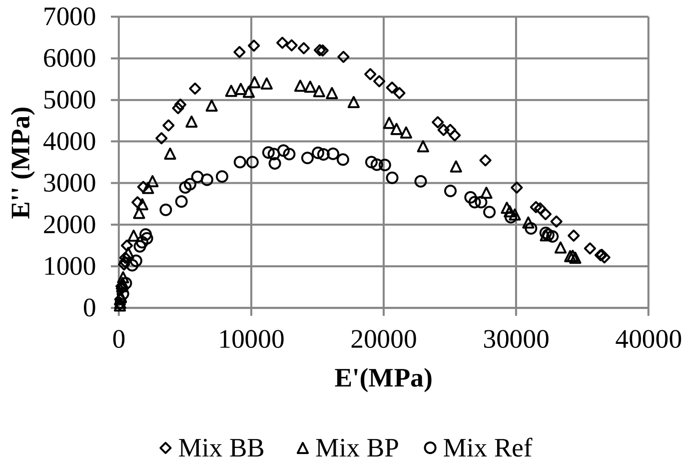

Figure 8. Mixtures' Cole-Cole diagram.

Table 2 shows the norm of the complex modulus and phase angle of the three mixtures at 15^{o}C and 10Hz as a reference parameter for fatigue. It can be seen that bio-mixtures have an acceptable complex modulus to be considered as high-modulus mixtures, which was the aim of the design using only recycled materials and biobinders. It is also worth noting that Mix BB has the highest norm of the complex modulus at 15^{o}C and 10Hz, which was not expected since BB is the softest fresh binder used. This fact reinforces the idea that biobinders, especially BB, could harden during the manufacturing of the mixtures more than conventional binders due to their composition.

Table 2. 50% RA mixtures' complex modulus and phase angle at 15°C and 10Hz.

Mixture	|E*| @ 15°C, 10Hz (MPa)	δ(°)
Mix BB	16587	21.9
Mix BP	12416	25.8
Mix Ref	13381	16

5. Modelling and discussion

Master curves at T_{ref}=0°C were produced for each blend and mixture and the 2S2P1D model fitted to the data. However, Mix BB, BP and Mix BP reached infinite values of the β parameter during the minimization process. This fact means that the linear dashpot in 2S2P1D model could be removed without influencing the results. The 2S2P1D model without linear dashpot is known as the "Huet-Sayegh" model (Sayegh, 1965). This peculiarity was particularly true for BP, which seems to have a very different rheological behaviour to conventional bitumen.

Master curves of the norm of the complex modulus and phase angle are displayed in Figures 9 and 10 respectively together with the model results. The model parameters are shown in Table 3. Previous research showed that binders and mixtures parameters should be related having the same value for δ, k, h and β (Di Benedetto et al., 2004; Olard & Di Benedetto, 2003). However, the materials studied in this paper do not show such a relationship and therefore the step of predicting mixtures behaviour from binders seems less direct. This fact could be due to the high RA content in mixtures which could hinder the full blending between RA and fresh binders, which was already highlighted by Mangiafico et al. (2013). If this is the case, the binder present in mixtures is not the same as the blends studied, and therefore the relationship between them is not possible. On the other hand, the lack of relationship can also be affected by changes in binders during the manufacturing of the mixtures. In order to further assess this issue, the normalised Cole-Cole diagrams of blends and mixtures were produced. To this purpose, E* is normalised following Eq. (2).

$$E^*_{norm} = \frac{E^* - E_0}{E_\infty - E_0} \quad (2)$$

Where E_0 and E_∞ are respectively the minimum and the maximum asymptotic values of the norm of the complex modulus obtained with the models (Table 3). The normalised Cole-Cole diagrams display the real part of E^*_{norm} in the x-axis and the imaginary part in the y-axis and are shown in Figure 11. It can be observed that blends and mixtures with the same binder do not completely overlap, especially for BP and low temperatures, which confirms that there is no direct link between blends and mixtures rheological properties (Mangiafico et al., 2013).

Nevertheless, the influence of the binders in mixtures can be noticed in Figures 9 and 10 where at low frequencies blends and mixtures provide the same ranking for modulus and phase angle.

Table 3. 2S2P1D and Huet-Sayegh parameters for blends and mixtures at T=0°C.

Binder/Mixture	**E_0**	**E_∞**	**δ**	**k**	**h**	**β**	**τ**	**C1**	**C2**
RA+BB	0	1344	4.7	0.34	0.83	2.7	0.12	17.7	122.0
Mix BB	42	38033	1.3	0.26	0.80	-	4.87	30.3	211.7
RA+BP	0	1828	3.6	0.24	0.73	-	0.03	20.2	122.4
Mix BP	40	36395	1.2	0.22	0.71	-	3.33	28.2	172.1
RA+Ref	0	1682	5.9	0.31	0.69	361.0	0.14	22.1	134.0
Mix Ref	88	39935	2.0	0.18	0.52	100.7	15.31	30.6	180.4

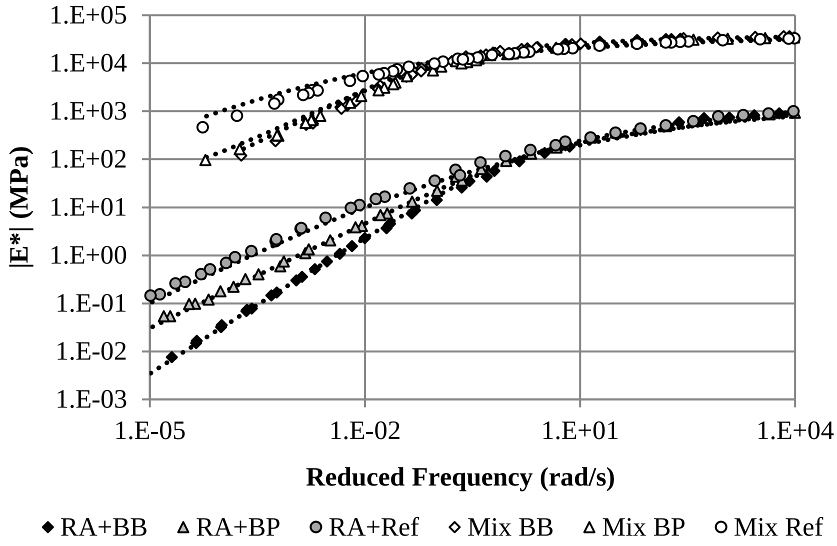

Figure 9. Master curves of the complex modulus and 2S2P1D model (dotted lines) at T_{ref}=0°C for blends and mixtures.

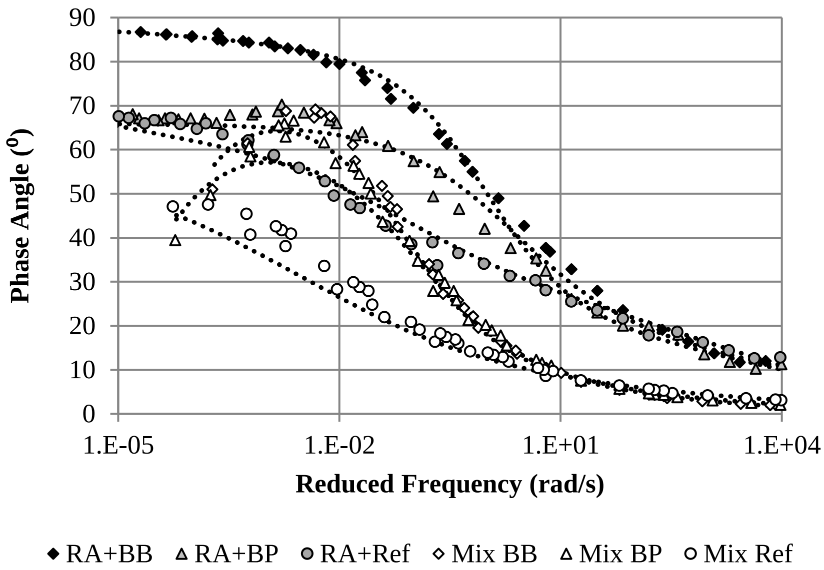

Figure 10. Master curves of the phase angle and 2S2P1D model (dotted lines) at T_{ref}=0°C for blends and mixtures.

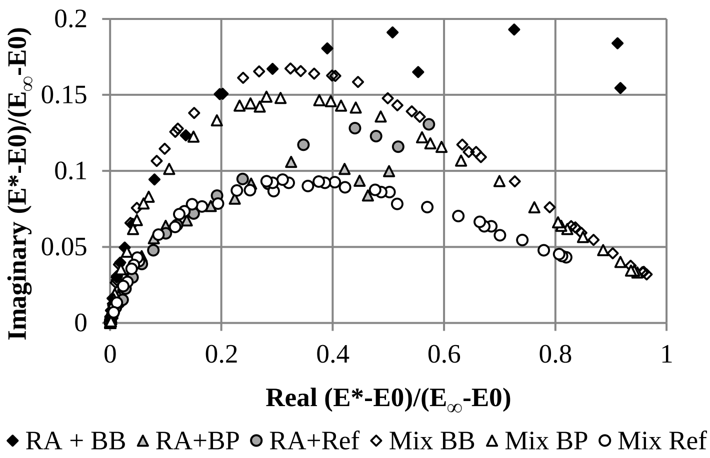

Figure 11. Normalised Cole-Cole diagram for blends and mixtures.

6. Conclusions

This paper shows the linear viscoelastic properties of binders and mixtures with 50% RA and biobinders. Binders were studied by means of DSR while mixtures were tested in two-point bending mode. Rheological data from both tests was analysed and modelled using 2S2P1D and Huet-Sayegh models. The following conclusions can be drawn:

- Biobinders studied are thermo-rheologically simple materials which allow their rheological characterisation by means of the same techniques used for conventional binders.
- Biobinders used in this investigation showed to act as rejuvenating agents over the RA binder (decreasing its complex shear modulus and increasing its phase angle) as well as allowing obtaining high-modulus asphalt mixtures with high RA content.
- Biobinders have an important effect on mixtures behaviour. However, no direct links between their linear viscoelastic properties were found. This fact could possibly be due to the hypothesis of full blending between RA and biobinders that

was made for blends and mixtures manufacturing; or to changes suffered in biobinders during the mixtures' manufacture.

High RA content mixtures with biobinders seem promising in order to evolve toward a more sustainable pavement engineering reducing the use of virgin materials and non-renewable resources such as those derived from oil. However, further work to fully understand their behaviour is still needed.

Acknowledgements

The research presented in this paper was carried out as part of the Marie Curie Initial Training Network (ITN) action, FP7-PEOPLE-2013-ITN and BioRePavation. These projects have received funding from the European Union's Seventh Framework Programme for research, technological development and demonstration under grant agreement number 607524 and 31109806.0002 respectively. BioRePavation is co-funded by Funding Partners of The ERA-NET Plus Infravation and the European Commission.

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