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A practical approach to estimate the degree of binder activity of reclaimed asphalt materials

Using Reclaimed Asphalt (RA) in new asphalt mixtures can reduce the amount of new material required thereby saving money and natural resources. In addition, asphalt mixtures with RA have shown comparable properties and performance to that generally associated with asphalt mixtures made with 100% virgin material. However, RA content in pavement surface layers is still limited due to specification and technical limitations. For higher contents, the aged RA binder must be analysed to accurately determine the requirements for virgin binders and additives while the degree of blending (DOB) between the RA binder and the virgin binder also needs to be quantified. This is not a simple process and generally designers assume one of two opposing theories associated with 100% (full blending) or 0% ("Black Rock") DOB. This paper proposes a new approach to estimate a unique property of the RA known as the Degree of Binder Activity (DoA) as a function of the processing temperatures of the RA. The study showed positive results and indicated that this DoA approach can be used as a tool to better understand RA in order to improve the binder/blend design for recycled asphalt mixtures.

Keywords: Degree of binder activity; reclaimed asphalt; indirect tensile test; ageing binders.

Introduction

Using high contents (>50%) of Reclaimed Asphalt (RA) in new asphalt mixtures potentially saves money and natural resources. However, the incorporation of high contents of RA in asphalt surface layers is still generally constrained by highway agencies throughout the world with a maximum RA content of approximately 40% normally specified (Mollenhauer, 2010; Copeland, 2011; Austroads, 2015; EN 13108-1, 2016). This maximum allowance is specified based on concerns over the performance of surfacing layers consisting of asphalt mixtures containing high proportions of aged RA but also due to technical issues associated with production or manufacturing limitations of existing asphalt plant. This second concern can tend to be more restrictive in terms of limiting RA content as research studies have shown that with an appropriate mix design, high content RA asphalt mixtures (including surfacing materials) can in most cases achieve satisfactory performance-related properties compared to conventional asphalt mixtures made with virgin materials (Shen *et al.*, 2007; Al-Qadi *et al.*, 2012; Kalman, 2013; Oliveira *et al.*, 2013; Zaumanis *et al.*, 2014b; Mohajeri, 2015; Lo Presti *et al.*, 2017). In addition to these asphalt mixture performance studies, research has also been undertaken to develop and propose innovative methods for characterising RA and adapting asphalt mixture design methodologies to take advantage of this recycled component (McDaniel and Anderson, 2001; West, 2010; Zhou *et al.*, 2010; Nicholls and James, 2011; Tebaldi *et al.*, 2013; Zaumanis *et al.*, 2014a). However, despite the considerable amount of research undertaken in this area, questions still remain related to how much binder is actually reactivated from RA during the asphalt mixing process.

The amount of RA binder that is possibly activated during the manufacture of new asphalt mixtures containing RA has been described in the literature using various terms and definitions - the degree of blending (DOB), the degree of (re)activation, mobilised binder, mobilisation rate, blending efficiency, binder blending, etc. (Huang *et al.*, 2005; Al-Qadi *et al.*, 2009; Shirodkar *et al.*, 2011, 2013; Navaro *et al.*, 2012; Coffey *et al.*, 2013; Bowers, Huang, *et al.*, 2014; Bowers, Moore, *et al.*, 2014). All these concepts are associated with two opposite scenarios related to the behaviour of the RA in this context, namely “full blending” (100% DOB) and “Black Rock” (0% DOB). Full blending assumes that 100% of the RA binder is reactivated and becomes part of the new mixture whereas “Black Rock” states that 0% of the RA binder will be activated and that the recycled asphalt material will simply be part of the aggregate components.

However, the term “Degree of Blending (DOB)” is one that often creates confusion with DOB been used to either identify the proportion of RA binder that blends with the virgin binder in a recycled asphalt mixture or the proportion of RA binder that is activated and therefore available to potentially blend with virgin binder. To remove some of this confusion, this study uses a new term known as the “Degree of Binder Activity (DoA)”, fully explained by Lo Presti *et al.* (2019), which can be defined as the proportion of RA binder that is activated in the RA and can be considered a unique intrinsic property of the RA. DoA is only influenced by the composition of the RA and the processing conditions associated with the use of the RA (conditioning and mixing temperatures and times associated with asphalt production). DOB, on the other hand, can be considered a parameter of the final asphalt mixture and it is defined only in the presence of additional materials, usually virgin binder or rejuvenators. It is important to appreciate that DoA can be used to predict a level of DOB but not vice versa, because the DoA is a property of the RA itself and cannot be estimated with accuracy when recycling agents are added. Furthermore, the schematic representation of the blending phenomenon presented by Lo Presti et al. (2019), including DoA and DOB, was the foundation of the present work undertaken to investigate the RA bitumen activity.

One of the characteristics of RA binder is that it is usually much stiffer than typical virgin binders used for asphalt mixtures due to the oxidative ageing of the RA binder that occurs during asphalt mixture production (high mixing and compaction temperatures) and the service life of the asphalt mixture (lower rate of oxidation but for longer periods of time). It goes without saying that in the case of high-content RA asphalt mixtures, correctly predicting the DoA of the selected RA (and subsequently the DOB of the recycled asphalt mixture) is crucial to obtain asphalt mixtures which comply with specific design standards. If the RA binder does not blend with the virgin binder as predicted, pavement performance could be compromised.

Several studies have already provided an assessment of the DOB of a recycled mixture for specific asphalt mixture manufacturing processes and material combinations (Huang *et al.*, 2005; Shirodkar *et al.*, 2011, 2013; Gaitan, 2012; Booshehrian *et al.*, 2013;

Bowers, Huang, *et al.*, 2014; Bowers, Moore, *et al.*, 2014; Mohajeri, 2015). However, none of them have proposed a practical and validated methodology to characterise the DoA of RA. Without the ability to predict DoA and despite the appreciation of the importance of this parameter, most research studies, as well as field practices, assume that full blending takes place. Various studies have highlighted that this is an acceptable practice when RA contents are below 25% (McDaniel and Anderson, 2001; Mollenhauer, 2010; Denneman *et al.*, 2013) but moving towards higher content RA asphalt mixtures, it is necessary to define a methodology that provides at least a plausible range of DoAs for the selected RA and the specific manufacturing conditions. It is generally accepted that RA does not act in the mixture simply as "Black Rock" but likewise it is accepted that full blending usually does not occur. Instead it is plausible to believe that the DoA of any RA, considering both conditioning and processing temperature and time associated with the manufacture of the recycled asphalt mixture, is somewhere between these two extremes. For example, a partial degree of binder activity of 80% (DoA of 80%) means that 80% of the RA binder should be available in a new recycled asphalt mixture. This concept has already been successfully used at the design stage in previous research studies (Jiménez del Barco Carrión *et al.*, 2015; Lo Presti *et al.*, 2016). However, these studies assumed a DoA range of values without providing a practical methodology to determine precise values.

This paper aims to fill this gap by proposing an innovative and practical methodology to allow researchers as well as practitioners to determine the range of DoAs for selected RAs as a function of the specific material and the recycled asphalt mixture manufacturing processes. Various researchers have already shown the importance of not only the properties of the asphalt mixture components but also the specific processing conditions on the design of recycled asphalt mixtures (García-Morales *et al.*, 2007; Navarro *et al.*, 2007; Lo Presti *et al.*, 2012). This innovative, as well as practical approach, will provide a DoA value for a selected RA to optimise the design of high-content RA asphalt mixtures produced at different manufacturing conditions from traditional hot (170°C) to half-warm (70°C).

Previous binder blending and activity studies

Previous research highlighted the importance of the blending concept and proved that the DOB of a selected RA can be related to the ageing level of the material (RA binder) as well as the selected manufacturing conditions such as temperature and mixing times. The studies highlighted below have provided suggested ways to measure DOB while recognising the need for further investigation to better understand RA as a material with its own characteristics in the recycling process.

Huang et al. (2005) proposed a method to establish how much aged RA binder will be available to coat virgin aggregates under normal mixing conditions. In this coating study, the RA was blended with virgin coarse aggregates with the RA being limited in size to material smaller than the #4 sieve (approximately 20 mm) and the virgin coarse aggregates all being larger than the #4 sieve (sieves according EN 933-2:1996). This allowed the aggregates in the final recycled asphalt mixture to be distinguished between those originating from the RA and those added as virgin aggregates. Three RA proportions (10%, 20% and 30%) were used with blending (mixing) performed at 190°C for 3 minutes. In all three cases, the binder content of the RA reduced from 6.8% (before mixing with virgin aggregate) to 6.0% (after mixing and separation from the now coated coarse virgin aggregate). This represents an approximately 12% binder loss due to mechanical mixing at the conditions specified above, thereby indicating that the vast majority of the aged RA binder tended to “stick” to the RA aggregate with only a small portion (about 12%) being available to blend with virgin materials (aggregate). It can be concluded from this study that the DOB associated with this particular RA at all three proportions was approximately 12%.

Shirodkar et al. (2011) also developed a coating method to estimate the degree of partial blending of RA binder and virgin binder for recycled asphalt mixtures containing 25% and 35% RA. Using a similar procedure to that used by Huang et al. (2005), the RA binder transferred to the virgin aggregate (determined as an increase in weight of the virgin aggregates) was found to be 24% (DOB of 24%) for the 25% RA content mixture but only 15% (DOB of 15%) for the 35% RA content mixture. According to the authors, these values of binder transfer seem much lower than what is expected and the lower binder transfer for higher RA content may be result of a larger relative proportion of the RA binder "sticking" to the RA aggregates and therefore not being activated in the final recycled mixture. The increase in weight of the virgin aggregates is due to the coating by the RA binder after mixing. However, the reduction in weight of the RA aggregates may be due to four factors (a) loss of moisture content, (b) RA binder lost to mixing bucket, (c) loss of fine particles of RA during mixing, and (d) transfer of RA binder to virgin aggregates. The authors also determined the $G^*/\sin(\delta)$ values of the binders recovered from the virgin and RA aggregates after mixing and compared them to the original virgin and RA binders (coating study including a virgin binder). Using the rheological data from the recovered binders, the authors developed two equations to determine the DOB and concluded that the DOB for 25% RA recycled asphalt mixtures was 70% and for 35% RA mixtures was 96%. These DOB values were higher than that determined by undertaking the coating study without virgin binder and highlighted the influence of the presence of the virgin binder resulting in much higher DOBs.

The study by Shirodkar et al. (2011) illustrated the issue of terminology with two different processes (coating study with and without the addition of a virgin binder) both having DOB as an output. DOB can therefore not be considered a unique property of the RA with its definition and values being different in the case when only virgin aggregate is added (simple transfer of RA binder to the virgin aggregate) and when both virgin aggregate and binder are added (transfer and blending of materials). The suggested use of a RA binder activity parameter (DoA) would seem to be a more RA material specific parameter.

Bowers, Moore, et al. (2014) investigated the influence of mixing time, temperature and the inclusion of warm mix asphalt (WMA) additives to access the blending of virgin and RA binder. A coarse virgin aggregate and fine RA were blended with a virgin binder at defined mixing times from 30 seconds to 300 seconds and over a range of temperatures (130°C - 180°C). To analyse the results, the aggregates were separated and the binders recovered from each case and tested using a DSR in order to analyse the binders' complex shear modulus (G*). The authors found that there is a limit to which mixing time has an influence on binder blending (increasing the mixing time beyond 150 seconds showed little or no influence) although mixing temperature as well as the use of WMA additives had a significant effect. Mixing temperature presented a significant increase in blending from 130°C to 180°C and the use of WMA additives also increased blending.

The study by Bowers, Moore, et al. (2014) also highlighted the influence of variations in conditioning and processing times and temperatures on blending of recycled mixtures and virgin materials. Their study showed that it is essential to analyse how RA behaves for different conditioning and mixing processes in order to fully understand the blending behaviour and performance of the final recycled mixture containing different proportions of RA.

Finally, Bressi *et al.* (2016) proposed a methodology for investigating the clustering phenomenon (agglomerations of RA particles) of RA in recycled asphalt mixtures by studying combinations of mixtures, varying the amount of RA, the type of virgin (added) binder and the production temperature. One significant finding was that only limited amounts of blending occurred between the aged bitumen (from the RA) and the virgin bitumen (added to the mixture) for the coarse aggregate component of the RA. The authors also observed that increasing RA amount in the mixture reduced the clustering phenomenon.

Although these studies (and others) have attempted to determine the DOB of combinations of RA and virgin materials, known have specifically attempted to determine the DoA of RA. However, the methodologies followed by the papers and the identification of the need to consider production and mixing conditions have helped frame the proposed methodology used in this paper.

Proposed methodology for RA-DoA labelling

According to the research described above, it is evident that full blending (100%) between a virgin binder and RA binder is unlikely to occur with the degree of blending being a function of the properties of the virgin and RA binder and various mixing factors such as temperature and time. In this sense, to maximise and optimise the incorporation of recycled material into new asphalt mixtures, it becomes fundamental to have a plausible assumption of the DoA of a selected RA for a specific asphalt manufacturing process. By obtaining an estimate of the RA DoA, it would be possible to optimise the binder blend design with virgin binders and/or additives (rejuvenators), where the DoA could be used to improve the DOB with the RA binder and result in higher quality controlled mixtures. It is, therefore, necessary initially to quantify the amount of binder released (reactivated) from the selected RA during the manufacturing process of the final asphalt mixture.

The approach that is followed in this paper is based on the procedure known as the Cohesion Test (Campher, 2012) which uses the Indirect Tensile Strength (ITS) Test to classify 100% RA samples (RA material) as inactive, semi-active or active. The Cohesion Test procedure is performed on different RA sources by compacting Marshall specimens with 100% RA after mixing and conditioning the material for four hours at different temperatures

(20°C, 75°C and 100°C). The specimens are then conditioned at 25°C for testing by ITS (dry and wet conditioned) according to the appropriate standard. The hypothesis is that the active RA would produce Marshall specimens with higher ITS values than inactive RA. Furthermore, an increase in compaction temperature will soften the RA bitumen and thereby decrease the overall viscosity of the RA mixture, improve compaction and result in a stronger specimen. In general, the classification of RA was found to fit into the three categories based on the RA binder stiffness as follows: Inactive (hard), semi-active (moderate) and active (soft). The ITS results (and therefore the above classification) was found to be a function of both the mixing and conditioning temperature and type of RA. This procedure has also been used as the framework for a RILEM TC 237-SIB study on Cohesion Testing of Recycled Asphalt (Tebaldi *et al.*, 2018) which assessed the ability of this procedure to characterise RA with an easy-to-perform test that did not require binder recovery.

The Cohesion Test approach developed by Campher (2012) and applied by Tebaldi et al. (2018) has formed the basis of the research methodology used in this paper. The present investigation has coupled the potential of the cohesion test approach with the aim of having a ‘label’ for the DoA of RA at different processing temperatures and times. The philosophy used as part of the research methodology is that if a selected RA has latent binding properties (i.e. aggregates surrounded by “active” bitumen), the total energy, pre-peak energy, post-peak energy, flexibility and relative tensile strength of a sample of 100% of this RA mixture should be higher than those of a mixture of 100% of RA surrounded by “inactive” bitumen (Harvey, 2010; Jitsangiam *et al.*, 2012; Katman *et al.*, 2012; Jaya and Asif, 2015). This behaviour should also vary and be more evident if RA is processed at higher temperatures and/or longer times.

In order to prove this hypothesis, a detailed experimental programme using an artificially created control RA was undertaken to provide parameters and indexes to label RA based on its DoA. Figure 1 shows the experimental programme crafted around this idea which aims to compare the binding properties of a 100% RA mixture with a control mixture ("artificial" RA mixture) manufactured at the same temperature. The primary objective of this study is the creation of a RA-DoA labelling framework from ITS results derived from the two different materials: RA and the "artificial" RA mixture using the Cohesion Test. The results from the Cohesion Test for the two materials are compared to obtain an index and estimate the activity values for a specific RA material. The idea is to get comparable results by creating the artificial RA mixture with similar characteristics to that of the original RA (including the same conditions such as bitumen characteristics, grading curves, RA aggregate types and mixture binder content). The principle of this investigation is that if the artificial RA mixture is produced from 100% new materials (no recycling) then full activity in the mixture can be assumed. This requires bitumen with similar characteristics to that of the RA binder to be produced and mixed with virgin aggregates giving a mixture with 100% "blending" and therefore 100% "activity", independent of the mechanical test results. The ITS results of the artificial RA are then compared with the 100% RA mixture making it possible to create the DoA index for this investigation.

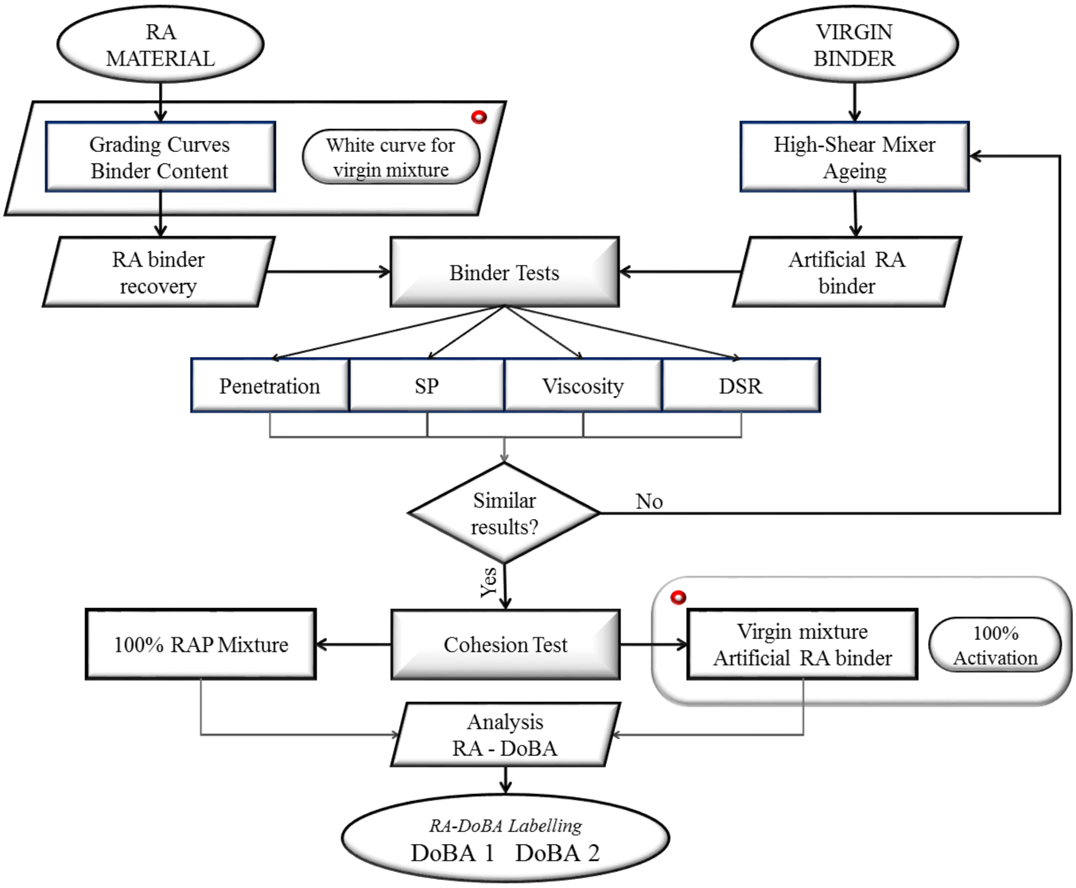

Figure 1. Flowchart – RA-DoA labelling framework.

Materials

Two sources of RA where used in the study together with two control asphalt mixtures consisting of virgin materials (aggregate and binder) but with the same aggregate gradation, binder content and binder consistency as the RAs.

In order to determine the properties of the RA and virgin materials, a range of tests were performed on the two RA sources and virgin materials:

RA:	Black/White grading curves (EN 12697-2, 2015);
	Binder content (EN 12697-1, 2012);
	Maximum Density (EN 12697-5, 2009);
RA aggregate:	Water absorption (EN 1097-6, 2013);
Binders:	Needle penetration at 25^{0}C (EN 1426, 2015);
	Ring and ball softening point (EN 1427, 2015);

Asphaltenes content (BS 2000-143, 2004);

Rotational viscosity - 100 to 200^{0}C (EN 13302, 2010);

DSR frequency/temperature sweeps - 0.1-10Hz / 0-80°C (EN 14770, 2012).

The RA binder was tested after binder recovery following the standard procedure (EN 12697-4, 2015) Bituminous mixtures - Test methods, Part 4: Bitumen recovery: Fractionating column.

RA gradation and compositional analysis

The grading curves of the two sources of RA were determined using the procedures detailed in the European testing standard (EN 12697-12, 2015) in terms of, firstly, the grading of the original material (known as a Black curve) and, secondly, on the recovered aggregate after binder extraction (known as a White curve). Figure 2 presents the grading curves of the two RA samples (Black and White curves) together with the grading curves produced with virgin aggregates to create the two control asphalt mixtures. The grading envelope for a standard Asphalt Concrete 20 mm asphalt mixture (AC 20) (BS 4987-1, 2005) is also included in the figure.

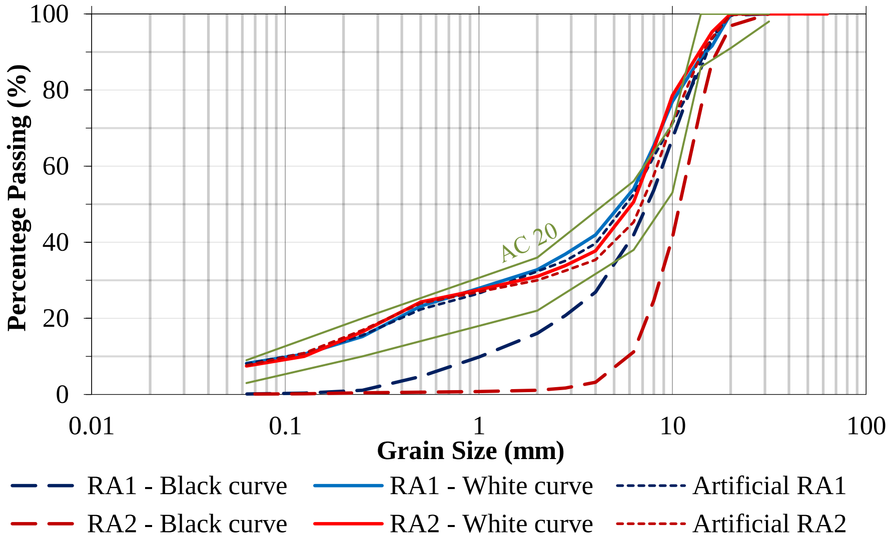

Figure 2. RA – Grading curves.

The particle size distribution confirms the most common finding associated with RA grading of a low amount of fine material. This characteristic occurs due to the presence in the Black curve of the finer particles of the material bound together with the bitumen and the subsequent formation of lumps (small conglomerates). However, after bitumen extraction, the white curve shows a significant amount of fines in both materials (approximately 30% of the particles equal or smaller than 2mm). Surprisingly, considering the different Black curves, both RA1 and RA2 presented very similar grading curves after binder extraction, meaning that the same original gradation curve may have been used for these two materials. The difference in the Black curves will probably be a result of differences associated with the milling process of the RA (e.g. processing, crushing and fractionating). The Artificial RA grading curve was produced from virgin material fractions using the RA White curve as a target. In addition to the grading curves, the grading limits for Asphalt Concrete 20 (AC 20) (BS 4987-1, 2005) are also shown with the White curve fitting almost entirely inside the grading envelop.

The binder content of the RA was also determined and found to be 4.4% for RA1 and 4.7% for RA2. These binder contents were then adopted for the artificial (control) RA mixtures. In addition, the absorption of the virgin aggregates should ideally be similar to that of the RA aggregate. In this sense, the water absorption was determined for both virgin and RA aggregates (after total binder extraction) for a range of 3 aggregate sizes (12.5, 8 and 4mm). The results showed values equal to 0.4% (12.5mm), 0.6% (8mm) and 0.8% for both materials, meaning that the chosen virgin aggregate can absorb the same amount of binder as the original RA aggregates. These values were expected to be similar due to the origin of both the virgin and RA aggregates being the same. The maximum density of the RA mixtures was also determined and found to be equal to 2,475 Mg/m^3 for RA1 and 2,417 Mg/m^3 for RA2.

Binder properties

A neat virgin 50/70 penetration grade bitumen was selected for the ageing procedure in order to create the aged binder used in the control mixtures. A procedure proposed by Wu (2009) was chosen to produce the aged binder. This procedure consists of ageing a 4 litres volume of the bitumen using high-shear mixing at a temperature of 163°C. The mixer is operated at a speed of approximately 3500 rpm with the top surface of binder being continuously exposed to air. During the high-shear ageing procedure, bitumen samples of the artificial binder (ArtRAb) were taken at fixed intervals (every 6 hours for up to 36 hours) in order to measure their conventional and rheological properties and compare these with the recovered bitumen from the two RA samples (RA1b and RA2b). The following binder characterisation was performed: Penetration - Needle penetration at 25^0C; Softening Point - Ring and Ball; Viscosity - Rotational viscosity - 100 to 200^0C; and DSR - Frequency sweeps 0.1-10 Hz; Temperature sweeps 0-80°C.

The initial analysis was based on the results of Penetration at 25°C and Softening Point with the dashed lines in Figure 3 representing the targeted values of the recovered RA binder results from RA1 and RA2. The ageing was stopped after 36 hours of high shear mixing of the virgin 50/70 penetration binder with the penetration of virgin binder decreasing after 36 hours from 68dmm to 19.8dmm which was slightly higher than RA1 (16.3dmm) and lower than RA2 (24.6dmm). The ring and ball softening point was determined as 61.8°C after 36 hours of ageing which was lower than RA1 (64.9°C) and almost identical to RA2 (61.1°C).

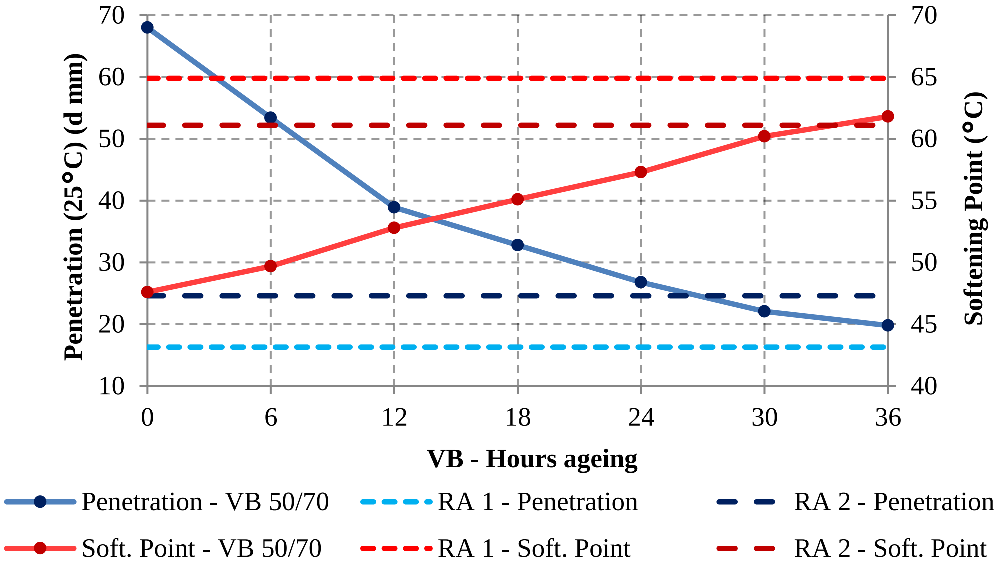

Figure 3. Ageing procedure – Penetration and Softening Point

The viscosity increased every 6 hours of ageing for each measured temperature with values at 135 and 170°C presented in Table 1. Finally, the rheological properties from the DSR frequency and temperature sweeps consisting of Log(G*) at 0.4Hz and 25°C (used to predict needle penetration using the correlation proposed by Gershkoff (1991)) were recorded.

Table 1. Binder's ageing characterisation

Binder / Test	VB 50/70	VB HSM - Ageing					
		6h	12h	18h	24h	30h	36h
Penetration 25°C (dmm)	64	53.4	38.9	32.8	26.8	22.1	19.8
Softening Point (°C)	47.6	49.7	52.8	55.1	57.3	60.2	61.8
Penetration Index (-)	-1.07	-1.13	-1.08	-0.92	-0.87	-0.67	-0.58
Fraass Breaking P. (°C)	-8	-	-	-	-	-	+2
Rot. Visc. 135°C (Pa.s)	0.275	0.300	0.373	0.486	0.596	0.735	0.868
Rot. Visc. 170°C (Pa.s)	0.066	0.071	0.083	0.100	0.117	0.137	0.154
Log(G*@0.4Hz-25°C)	5.492	5.612	5.875	6.032	6.177	6.310	6.388
Asphaltenes Content (%)	15.76	-	-	-	-	-	19.67

Table 2. Binder and RA ageing comparison

Binder Test	VB 50/70	VB HSM 36h	RA 1		RA 2	
				Relative Error		Relative Error
Penetration 25°C (dmm)	64	19.8	16.3	+21.5%	24.6	-19.5%
Softening Point (°C)	47.6	61.8	64.9	-4.8%	61.1	+1.1%
Penetration Index (-)	-1.07	-0.58	0.37	-	-0.31	-
Fraass Breaking P. (°C)	-8	+2	-3	+5°C	-7	+9°C
Rot. Visc. 135°C (Pa.s)	0.275	0.868	0.836	3.8%	0.984	-11.8%
Rot. Visc. 170°C (Pa.s)	0.066	0.154	0.157	-2%	0.177	-13%
Log(G*@0.4Hz-25°C)	5.492	6.388	6.320	+1.1%	6.152	+3.8%
Asphaltenes Content (%)	15.76	19.67	21.3	-7.7%	20.8	-5.4%

Table 2 shows the comparison of the conventional and rheological properties of the original virgin 50/70 penetration binder, the 36-hour high shear aged binder and the two recovered binders from RA1 and RA2. The table shows the relative error (as a percentage) between the aged "artificial" RA binder and the two recovered binders from RA1 and RA2. The results show that the majority of properties are similar when the high shear aged virgin binder is compared to the two recovered binders from RA1 and RA2. The range of variability for particular parameters are as follows:

- Penetration (dmm - 25^{0}C): ±20%
- Softening Point (°C): ±5%
- Viscosity (Pa.s - 135^{0}C): ±12%
- Complex shear modulus – Log(G*@0.4Hz-25°C) (Pa): ±4%

Experimental programme

The primary objective of this study is to determine a DoA Index for RA from the Indirect Tensile Strength measurements for two different materials: the RA and an "Artificial" RA Mixture. The different mixtures were therefore compacted into cylindrical 100mm diameter specimens and subjected to the Cohesion Test to obtain the following five parameters;

Indirect Tensile Strength, Total Energy, Pre Peak Energy, Post Peak Energy and Flexibility Index.

Cohesion Test: Indirect Tensile Test (ITT)

The Indirect Tensile Test (ITT) has proven to be an important test procedure for characterising materials such as Portland cement concrete and asphalt mixtures. The loading arrangement of this test is through the application of diametrically opposite concentrated compression forces on a cylinder specimen. The vertical loading produces both a vertical compressive stress and an indirect horizontal tensile stress on respective diameters of the specimen. The test is easy to perform and relatively quick with the load and vertical deformation being recorded and used to determine various parameters including the Indirect Tensile Strength (ITS) (Peak-load), Total Energy (whole test), Energy (pre and post-peak load) and Flexibility Index (whole test). These properties were chosen based on previous studies where researchers have found that the ITT results are affected by the optimum binder content (OBC) of the mixtures with results tending to be lower at binder contents above and below the OBC (Harvey, 2010; Jitsangiam *et al.*, 2012; Katman *et al.*, 2012). In this study, the DoA is synonymous with the effects related to OBC as the activation process of the binder from the RA produces portions of active as well as inactive bitumen. There is therefore a reduction in the amount of available activated binder in the recycled asphalt mixture similar to a reduction in binder content below the OBC for standard asphalt mixtures. Both effects (lower degree of binder activity and lower binder content relative to OBC) result in a reduction in the ITT parameters. To increase the amount of useful data from the ITT, parameters in addition to the standard Indirect Tensile Strength (ITS) have been included. These extra parameters include Total Energy and Flexibility Index, which are related to the whole test and not just the peak load.

Indirect Tensile Strength – ITS

The test follows the standard EN 12697-23 (2017). Cylindrical test specimens were used in this study with dimensions of 100mm diameter and a target height of 63.5mm. The tests were carried out at a constant temperature of 25°C following the procedure developed by Campher (2012). The peak load for each specimen was used to calculate the ITS according to Equation 1.

$$\mathrm{ITS} = \frac{2 \times P}{\pi \times D \times H} \tag{1}$$

Where ITS is the indirect tensile strength, expressed in megapascals (MPa); P is the peak load, expressed in Newtons (N); D is the diameter of the specimen, expressed in millimetres (mm); and H is the height of the specimen, expressed in millimetres (mm).

Test Energy

The use of an energy approach for characterisation, as opposed to a strength-based approach, provides a better characterisation of the cracking processes in asphalt mixtures. The test energy in the ITT can be defined as the energy required to create a unit surface area of a crack (AASHTO TP105, 2013). The energy (E) is determined by first calculating the work (W), defined as the area under the load-displacement curve (Putman and Amirkhanian, 2004; Vasconcelos *et al.*, 2012). This work can be divided into pre-peak ($W^{\text{Pre-peak}}$), associated with energy necessary to initiate a crack, and post-peak fracture work ($W^{\text{Post-peak}}$) associated with energy needed to propagate the crack, as shown in Figure 4. The total work can be estimated according to Equation 2:

$$\mathrm{W} = \mathrm{W}^{\mathrm{Pre-peak}} + \mathrm{W}^{\mathrm{Post-peak}} = \int_{0}^{\Delta_{\mathrm{Pmax}}} \mathrm{P.dx} + \int_{\Delta_{\mathrm{Pmax}}}^{\Delta_{\mathrm{Pfinal}}} \mathrm{P.dx} \tag{2}$$

Where P is the load, expressed in Newtons (kN); x is the horizontal displacement, expressed in millimetres (mm).

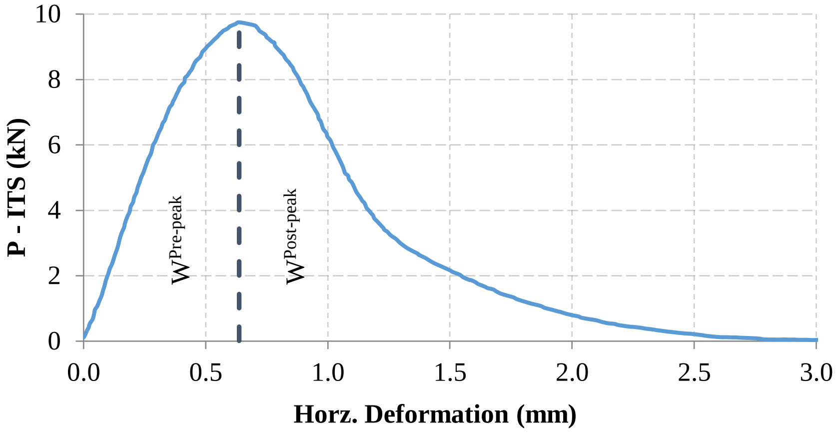

Figure 4. Fracture work definition

Using the results, the total energy can be calculated by normalising the total work by the area of fracture surface that is generated during the test. This area can be estimated as a product of the height of the specimen (H) and the length of a new crack formed during the test. This crack length is often referred to as the ligament length – the specimen diameter (D) in the ITT on cylindrical specimens. Total energy calculations are determined for the pre-peak and post-peak regions in order to distinguish the crack propagation process from the complete test using Equations 3 to 5:

$$E_{Total} = E^{Pre\text{-}peak} + E^{Post\text{-}peak} \tag{3}$$

$$E^{Pre\text{-}peak} = \frac{W^{Pre\text{-}peak}}{H \times D} \tag{4}$$

$$E^{Post\text{-}peak} = \frac{W^{Post\text{-}peak}}{H \times D} \tag{5}$$

Flexibility Index

The flexibility index (FI) is a parameter that can describe fundamental fracture processes and overall patterns of load-displacement curves and can determine the cracking potential of asphalt concrete mixtures (Al-Qadi *et al.*, 2015; Ozer *et al.*, 2016). The authors show that low-temperature fracture testing is variable between different mixtures and provides evidence that fracture energy alone cannot be used to differentiate between some mixtures. However, fracture testing at an intermediate temperature (25°C) provides the desired distinction according to the authors. Depending directly on the shape of the load-displacement curve, the energy is a function of both the strength (defined by peak load) and ductility (defined as the maximum displacement at the end of the test) of the material. If the material displays a high peak load, it may compensate its energy for the lack of ductility in the post-peak region of the load-displacement curve. This is a potential explanation for why brittle asphalt mixtures with high-RA amounts may display similar or sometimes higher total energy values than their reference mixtures used for comparison. FI can be calculated using Equation 6 (Al-Qadi et al., 2015; Ozaer et al., 2016):

$$\mathrm{FI}=\frac{E}{\mathrm{m}} \tag{6}$$

Where FI is the Flexibility Index, E is the energy expressed in $\mathrm{J/m^2}$ and m is the slope of the curve at an inflection point.

Cohesion Test: mixing and compaction

In order to analyse the RA variability, a range of mixing temperatures and times were selected. The mixing temperatures were selected from 70ºC (half-warm) to 170ºC (traditional hot mixture), whereas the mixing times were from 30s (simulating asphalt plants) to 180s (laboratory conditions). Table 3 shows the details of the testing associated with the

Reclaimed Asphalt and Virgin Reference (Artificial RA) mixtures that are analysed in the proposed methodology. All the combinations were tested with RA1 being tested first and a reduced number of combinations being used for RA2.

Table 3. Cohesion test variations and number of samples tested

CONDITIONING/MIXING		MATERIAL			
Temperature	Time	RA1	RA2	ART1	ART2
70°C	30s	3	-	-	-
	60s	3	3	5	5
	90s	3	-	-	-
	180s	3	3	-	-
100°C	30s	3	-	-	-
	60s	3	3	5	5
	90s	3	-	-	-
	180s	3	3	-	-
140°C	30s	3	-	-	-
	60s	3	3	5	5
	90s	3	-	-	-
	180s	3	3	-	-
170°C	30s	5	-	-	-
	60s	5	5	-	-
	90s	5	-	-	-
	180s	5	5	5	5
TOTAL		56	28	20	20
		124			

All the RA mixtures were manufactured using the same production protocol. First, the maximum density of the RA material was determined (EN 12697-5, 2009) in order to calculate the amount of material that should be placed in each mould to achieve the target height and air voids content. This was similar to the process used in the RILEM TC SIB 237 - TG-6 (Tebaldi *et al.*, 2018) study. The RA needed to produce the ITT specimens was then dried in an oven at 40°C for 48 hours before it was rifled, quartered and conditioned at the temperatures given in Table 3 for 4 hours prior mixing. Mechanical mixing (chosen due to high manufacturing requirements) for the times specified in Table 3 was undertaken and specimens compacted using a Marshall Compactor with 50 blows per side. These procedures were adopted in accordance with previous studies (Campher, 2012).

A slightly different procedure was used to manufacture the artificial RA mixtures. All the mixtures were pre-blended at high temperatures (170°C) as if they were virgin materials. They were then conditioned in the oven for 1 hour in order to reach the desired temperature listed in Table 3 (except for the 170°C mixtures where compaction was carried out directly after mixing). A mixing time of 180 seconds was used with the 170°C mixtures while 60 seconds mixing was used for the mixtures at temperatures lower than 170°C.

To control the 100% activity of the artificial RA, the artificially aged binder is heated up to 170°C for production and this temperature is never reduced to a lower value than the one required for production (cases of 70, 100 and 140ºC). This allows the assumption to be made that the maximum activity of the binder at that temperature is obtained whereas lower temperatures would decrease the activity.

Compaction was undertaken using the Marshall Compactor with the same procedure as used for the RA mixtures. The 60s mixing time prior to compaction was chosen to allow the results for the artificial RA mixtures to be compared to the RA mixtures.

Results

Influence of compaction temperature and time

The air voids content and ITS results as a function of compaction temperature and time for the two RA materials (RA1 and RA2) are shown in Figures 5 and 6. The air voids were calculated according to the European Standard EN 12697-6 (2012). The average air voids content (together with error bars showing the range in air voids associated with one standard deviation above and below the average) for the RA1 and RA2 compacted specimens are presented in Figure 5. The results show that the average air voids content decreases as a function of increasing mixing (and conditioning) temperature for each group of mixing times. However, the influence of mixing times (30 seconds to 180 seconds) appears to have less effect on the resulting average air voids content for RA1 with similar values being found for the different mixing times at each particular mixing temperature. Due to the results shown for the RA1 air voids, the mixing times were adjusted for the RA2 specimens by removing the 30s and 90s mixing times.

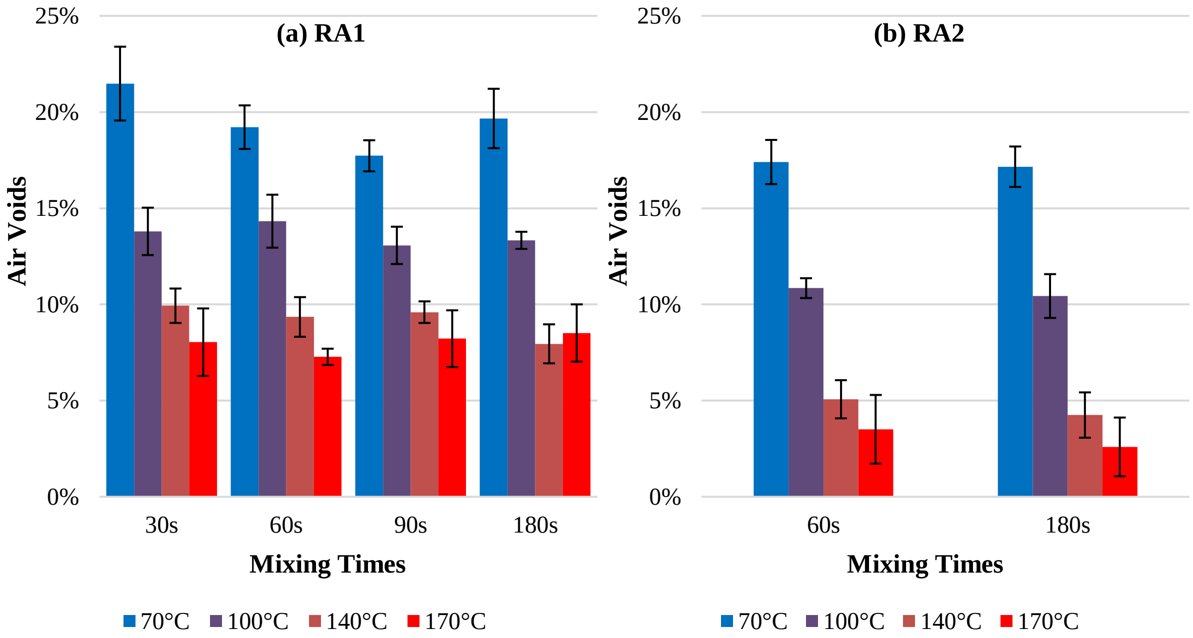

Figure 5. Cohesion test results for (a) RA1 and (b) RA2 – Air Voids

The influence of mixing temperature for the study can clearly be seen in Figure 5 with the higher temperatures resulting in a softening of the RA binder (reduction in binder viscosity) thereby improving the workability of the material and aiding in the compaction of the RA samples and the resultant lower air voids content. Volumetric proportions (air voids) of compacted RA samples could therefore also be used as a measure of binder mobilisation (or activity). The results also show that there are smaller differences in air voids content between 140 and 170°C compared to the differences found from 100 to 140°C. This would imply that the amount of binder activity in RA (at least for the RA materials used in this study) potentially reaches an optimum (or threshold) at high mixing temperatures.

The ITS results for RA1 and RA2 as a function of mixing temperature and time are shown in Figure 6. All the specimens used to determine the ITS results were conditioned for 24 hours at 25°C inside a temperature-controlled cabinet prior to subjecting the specimens to the ITT. These initial results were used to determine the mixing times selected to produce the control "artificial" RA mixtures for the DoA investigation.

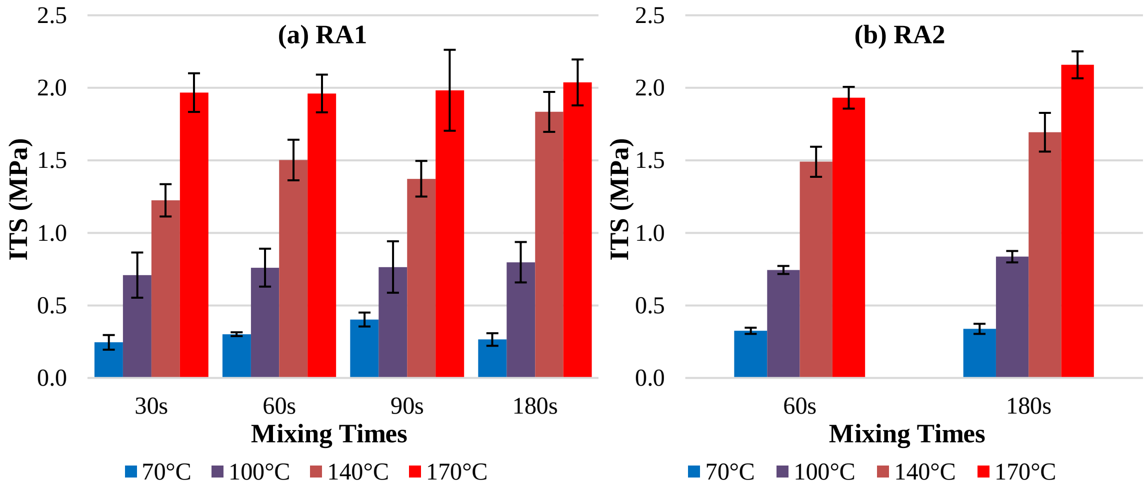

Figure 6. Cohesion test results for (a) RA1 and (b) RA2 – ITS by mixing temperatures and times

The results show that the ITS values increase with increasing conditioning and mixing temperature regardless of the mixing times. The highest tensile strength values were consistently found for the RA1 and RA2 specimens produced at 170°C, indicating the significant temperature dependent of the RA material regarding proposed binder activity (increased material cohesion) and increased density (higher degree of compaction). Same as seen for the air voids content, mixing times were found to be less important and influential compared to temperature.

Considering each of the specific mixing temperatures for RA1, the results at 70°C show some variation with average ITS values tending to increase with longer mixing times, although the differences cannot be considered significant. In terms of a visual assessment of the RA material during mixing and compaction, there is little evidence of significant binder activity with the material remaining opaque in colour with evidence of uncoated fine fractions on the surface of the compacted specimens.

Regarding the conditioning and mixing temperature at 100°C, the variation, in this case, was quite low with the ITS results not being affected by the mixing time. However, it is possible to affirm that at this temperature, the specimens began to show superior consistency with the apparent contribution of the bitumen. It can be concluded therefore that not only the improved compaction (compared to specimens at 70°C) but also the reactivation of the bitumen has contributed to improved ITS results.

Considering the results for those specimens produced at 140°C, the behaviour of the material seems to be significantly influenced by the bitumen. The appearance of the RA mixture after each of the four mixing times starts to become similar to a virgin asphalt mixture – although they are not present in this research. Regarding the mixing times, significant variation was found with the ITS increasing with the increase of mixing time, different from the previous two mixing temperatures.

Finally, specimens compacted at 170°C, as for the previous conditioning temperature, showed similar appearance to that of a virgin asphalt mixture with significant amounts of activated bitumen. However, the mixing times do not show any influence on the final ITS results.

Assessment of the degree of binder activity of the selected RAs

Due to the results presented in the previous section, just one mixing time (60 seconds for 70°C to 140°C and 180 seconds for 170°C) was chosen to manufacture the artificially aged RA mixtures and to analyse the DoA due to manufacturing limitations and comparison capability. These limitations are related to the production at 170ºC, where 180 seconds is necessary for mixing the aged bitumen and aggregates (artificial RA mixtures), thus, there is no possibility of selecting the mixing time of 60 seconds for the RA mixtures. Figure 7 shows the air voids of the produced samples with the values being controlled during the manufacturing process in order to have comparable cases regarding volumetric properties to remove the influence of air voids on the mechanical results.

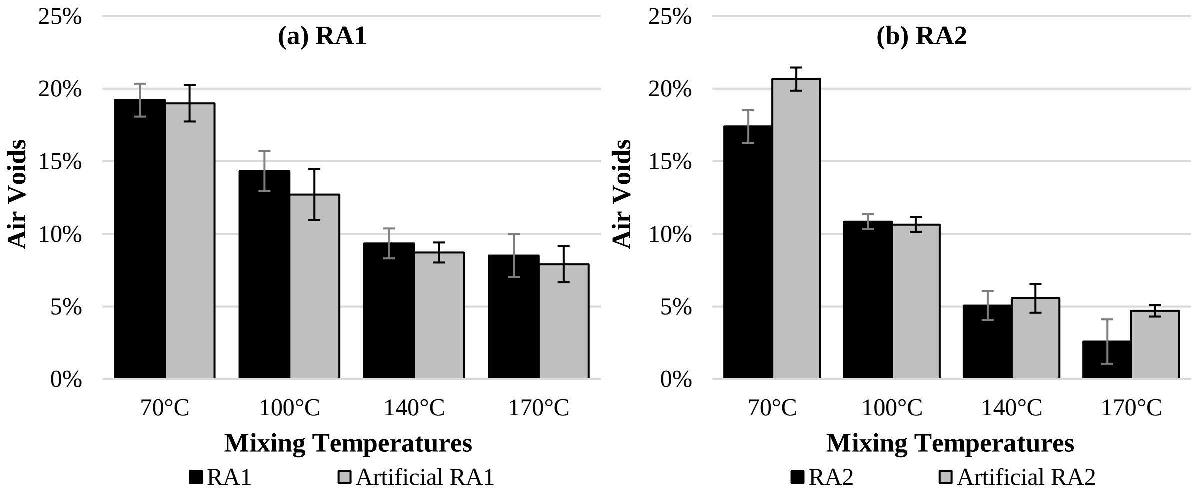

Figure 7. Cohesion test RAs and Artificial RA – Air Voids

Figures 8, 9, 10 and 11 present the results from the ITT in terms of ITS Peak Load, Total Energy, Pre and Post Peak Load Energy and Flexibility Index for each temperature and mixture studied.

In terms of the ITS results (Figure 8), a similar tendency for both cases can be seen with peak load increasing in accordance with the temperature increase probably due to the increased mobilisation of the binder raising the ITS. Furthermore, the ITS results confirm the importance of temperature conditioning where for the higher temperature there is a smaller difference between RA1-RA2 and the Artificial RA1-RA2 values. This means that the obtained maximum values for the Artificial RAs are close to the maximum possible values for these RAs (full binder activity). It is important to note the similar results for RA1 and

RA2 which can be explained by the similar properties of the two binders and mixture gradings.

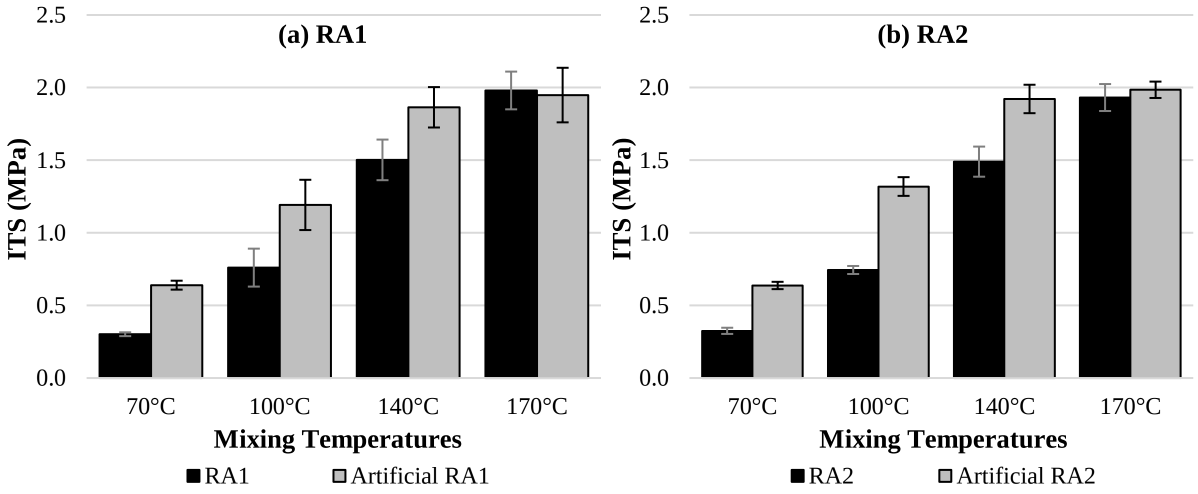

Figure 8. Cohesion test RA's and Artificial RA – ITS

Figure 9 shows the Total Energy (ETotal) results determined according to Equation 3. Similar to the ITS results, higher ETotal values were obtained for the Artificial RAs with a similar trend observed in terms of energy increasing with temperature. However, unlike the ITS results, the RAs show lower energy absorption capacity compared to the Artificial RAs at 170°C. This difference in behaviour may be explained by analysing the Pre and Post peak load regions (through the Total Energy) that consider the load-displacement connected to the energy absorption for each sample, before and after crack initiation.

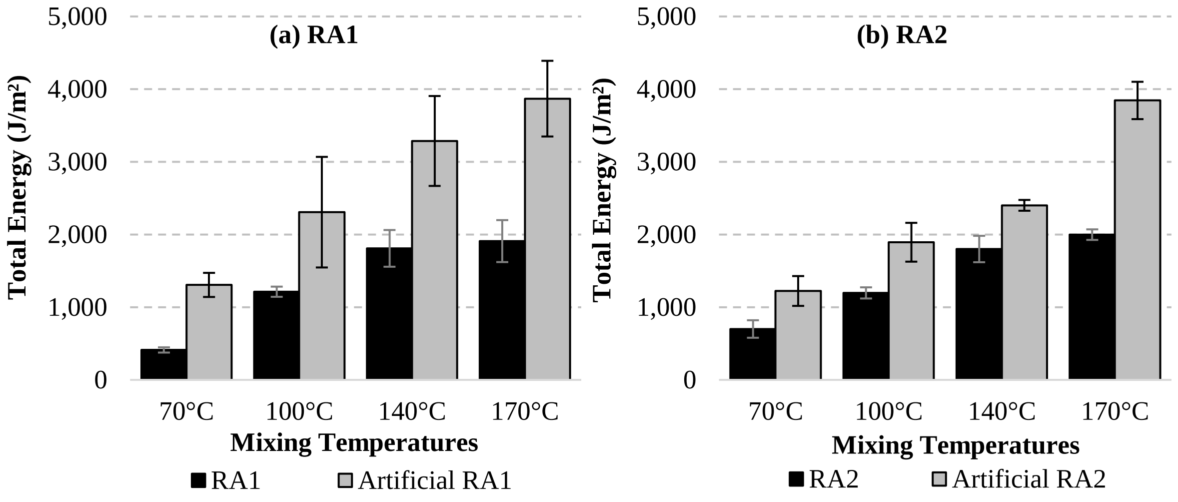

Figure 9. Cohesion test RAs and Artificial RA – Total Energy

The main differences that can be observed in Figure 10 are for the $E^{Post\text{-}peak}$ results for Artificial RA1 which are substantially larger than the values seen for both the other mixtures as well as the materials $E^{Pre\text{-}peak}$ results. There also seems to be high variability associated with the $E^{Post\text{-}peak}$ values when comparing the RA and artificial RA materials. The influence of temperature also appears to be less significant for these energy parameters compared to the ITS results.

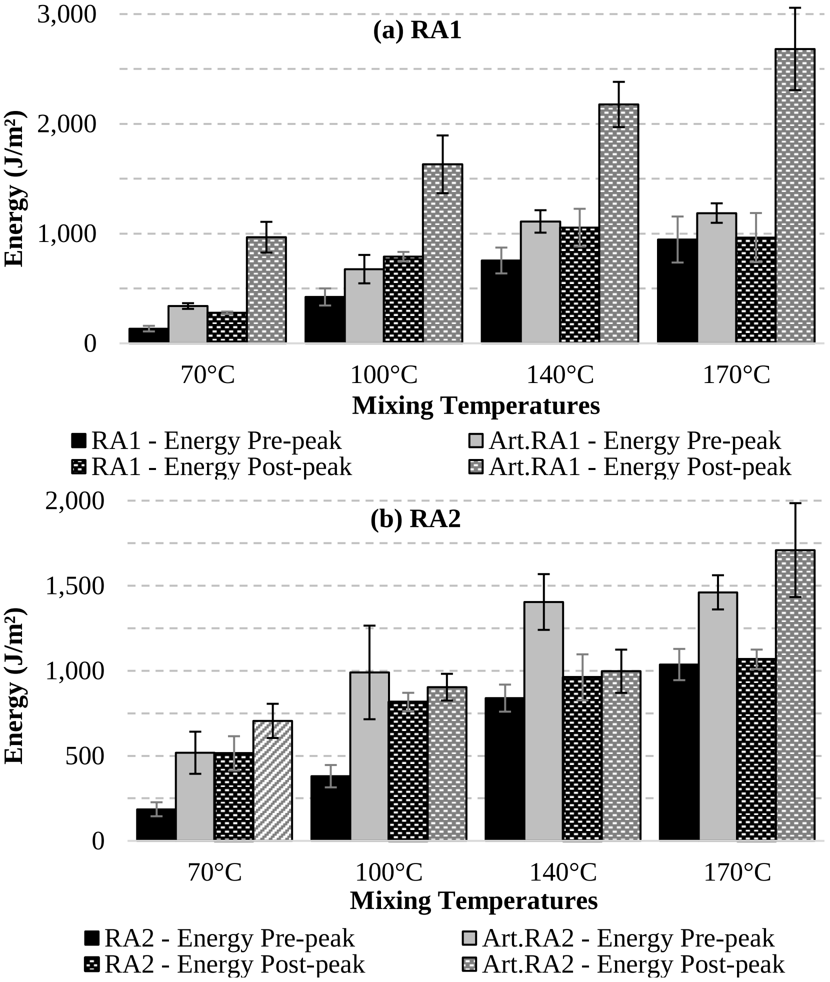

Figure 10. Cohesion test RAs and Artificial RA – Pre Peak and Post Peak Energy

Finally, Figure 11 shows the Flexibility Index (FI) results for the RA and Artificial RA mixtures. The FI is a simple index parameter that can be correlated to fundamental crack growth mechanisms and has the ability to distinguish mixtures with distinct design characteristics that may influence cracking resistance.

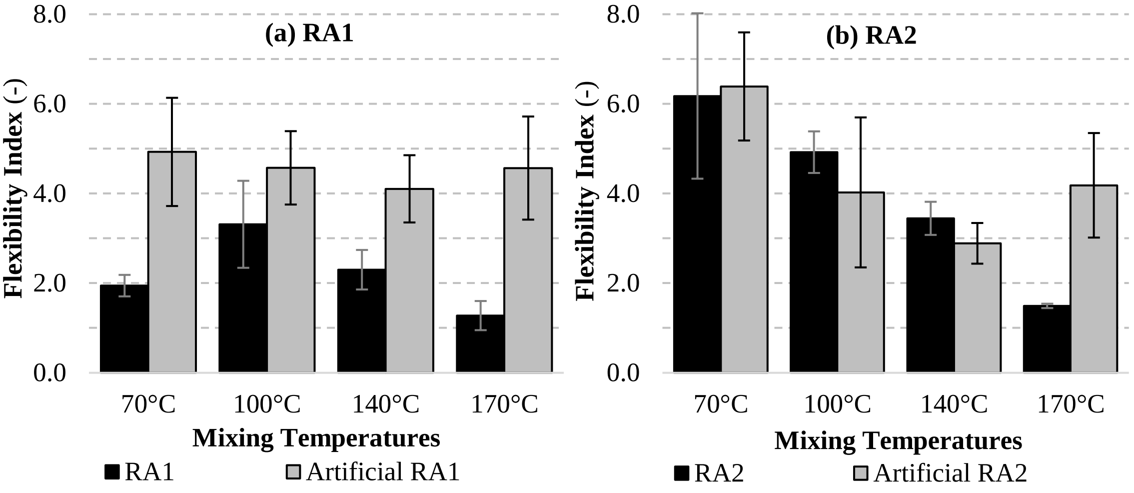

Figure 11. Cohesion test RAs and Artificial RA – Flexibility Index

The FI values for the mixtures ranged from 1 to 6 with increasing brittleness associated with lower FI values. Both RAs behave differently when comparing the FI results. RA1 shows some variation when the specimens were conditioned and produced at different temperatures with the brittleness increasing from 100°C to 170°C (lower FI values). The laboratory artificial RA1 mixtures present an almost constant index for all four temperatures, where the samples do not seem to be affected by the production temperature. However, in the case of mixtures RA2 and Artificial RA2, both mixtures show a decrease in FI with increasing production temperature. In addition, all the mixtures presented a high degree of variation of individual results with large standard deviations making FI difficult to use as a suitable parameter for evaluating RA binder activity.

RA DoA Labelling

In order to estimate the DoA for each temperature and material, the parameters investigated were inputted into Equation 7. The calculated DoA's are presented in Tables 4 and 5 for RA1 and RA2 respectively. The results assume that Artificial RA reached the maximum DoA value for each analysis, with the DoA being a mechanical relationship and the ratio between both materials (RA and Artificial RA). In addition, because the results were obtained through mechanical tests, the DoA from these outcomes are termed DoA'.

$$DoA' \, (\%) = 100 \times \frac{Y_{RA} \, (X°C)}{Y_{Art.RA} \, (X°C)} \tag{7}$$

Where YRA (X°C) is the parameter result "Y" for the RA at a specific temperature "X"; Art.RA (X°C) is the result for the artificial RA at the same temperature "X" analysed for RA.

Table 4. DoA' estimation for RA1

Temperature (°C)	Analysis	RA1	ART. RA1	DoA' (%)
70	ITS Peak Stress (MPa)	0.302	0.639	47.2
	E_{Total} (J/m²)	412.0	1306.8	31.5
	$E^{Pre\text{-}peak}$ (J/m²)	134.1	339.9	39.5
	$E^{Post\text{-}peak}$ (J/m²)	277.9	966.9	28.7
	Flexibility Index (-)	1.949	4.928	39.5
100	ITS Peak Stress (MPa)	0.760	1.192	63.8
	E_{Total} (J/m²)	1212.9	2307.4	52.6
	$E^{Pre\text{-}peak}$ (J/m²)	422.7	676.2	62.5
	$E^{Post\text{-}peak}$ (J/m²)	790.2	1631.2	48.4
	Flexibility Index (-)	3.313	4.573	72.4
140	ITS Peak Stress (MPa)	1.502	1.864	80.6
	E_{Total} (J/m²)	1809.3	3287.2	55.0
	$E^{Pre\text{-}peak}$ (J/m²)	754.9	1110.8	68.0
	$E^{Post\text{-}peak}$ (J/m²)	1054.5	2176.4	48.5
	Flexibility Index (-)	2.300	4.104	56.0
170	ITS Peak Stress (MPa)	1.980	1.949	101.6
	E_{Total} (J/m²)	1909.4	3869.4	49.3
	$E^{Pre\text{-}peak}$ (J/m²)	945.9	1187.0	79.7
	$E^{Post\text{-}peak}$ (J/m²)	963.5	2682.4	35.9
	Flexibility Index (-)	1.276	4.567	27.9

Table 5. DoA’ estimation for RA2

Temperature (°C)	Analysis	RA2	ART. RA2	DoA’ (%)
70	ITS Peak Stress (MPa)	0.330	0.637	51.2
	E_{Total} (J/m²)	700.1	1223.2	57.2
	$E^{Pre\text{-}peak}$ (J/m²)	185.8	518.0	35.9
	$E^{Post\text{-}peak}$ (J/m²)	514.3	705.2	72.9
	Flexibility Index (-)	6.176	6.389	96.6
100	ITS Peak Stress (MPa)	0.744	1.221	61.0
	E_{Total} (J/m²)	1196.8	1893.7	63.2
	$E^{Pre\text{-}peak}$ (J/m²)	380.2	990.2	38.4
	$E^{Post\text{-}peak}$ (J/m²)	816.6	903.5	90.4
	Flexibility Index (-)	4.923	4.025	122
140	ITS Peak Stress (MPa)	1.490	1.922	77.5
	E_{Total} (J/m²)	1800.7	2401.5	75.0
	$E^{Pre\text{-}peak}$ (J/m²)	839.2	1404.4	59.8
	$E^{Post\text{-}peak}$ (J/m²)	961.5	997.1	96.4
	Flexibility Index (-)	3.446	2.889	119
170	ITS Peak Stress (MPa)	2.158	2.018	107.0
	E_{Total} (J/m²)	2104.0	3844.6	54.7
	$E^{Pre\text{-}peak}$ (J/m²)	1036.6	1461.2	70.9
	$E^{Post\text{-}peak}$ (J/m²)	1067.4	1709.4	62.4
	Flexibility Index (-)	1.492	4.183	35.7

From the tables, the charts in Figure 12 – RA1 and Figure 13 – RA2 were produced representing the DoA’ percentage ranges as well as the active binder content for the studied temperatures and mixture cases. The five parameters are plotted following the principle presented in Equation 7.

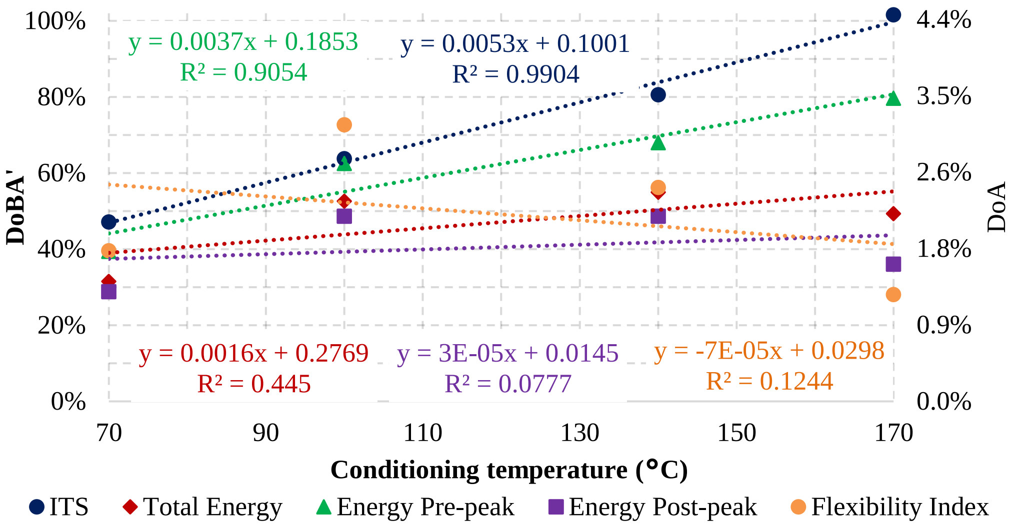

Figure 12. DoA’ estimation for RA1.

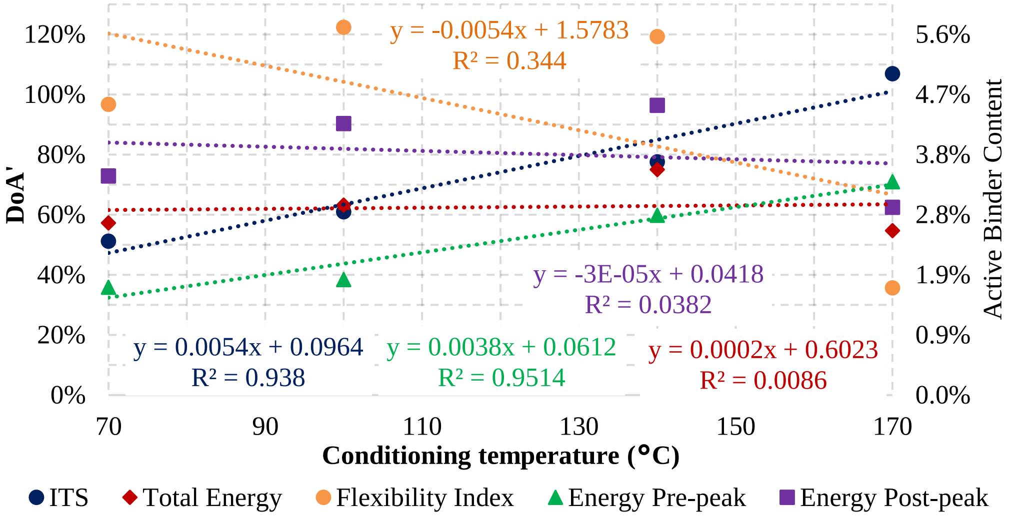

Figure 13. DoA’ estimation for RA2.

Analysing the estimated DoA’ from RA1 and RA2, it can be seen that there is a similar trend in the regression lines for ITS and EPre-peak. Moreover, it is possible to observe a relatively good correlation and high value of R2 (>0.9) from the linear regression analysis for both RA1 and RA2. However, the results of ETotal, EPost-peak and FI do not show the same tendency seen for the previous two parameters, where from 100°C upwards the values of the DoA’ ratios start to decrease. Furthermore, the RA2 ETotal and EPost-peak results are higher at lower temperatures regarding DoA which is opposite to what has been observed to occur during the conditioning, mixing and compacting of the samples. What may explain this dispersion in the results is the increased activation of the aged RA binder, which when coupled with its inherent brittle nature, affects the rate of crack initiation and propagation during the ITT. Therefore, ETotal, EPost-peak and FI cannot be considered suitable for the determination of the DoA ratio for the RAs investigated.

In summary, the analysis of the parameters is promising since the trends found are consistent with the expected values and confirm the main philosophy, already observed in the literature review, that the temperature is the most important factor in the activation of RA bitumen. Inevitably, the temperature increase should gradually boost the DoA and not diminish it as was found for three of the ITT parameters (E_{Total}, $E^{Post\text{-}peak}$ and FI). Moreover, the total control of conditioning, mixing and sample production leaves no room for rapid ageing of the material in the oven, which could drastically alter the properties of the RA binder at high temperatures.

RA DoA Labelling – Supplementary analysis using (ITS)

In addition to the approach already described, another way of analysing the data was investigated. This consisted of a more straightforward method to use the results obtained from the Cohesion Test but only for the RA mixtures. In order to create the index to estimate the DoA, the results of the RA samples produced at 170°C were used as a reference, meaning that 100% DoA is assumed for those materials conditioned at this temperature. This assumption of full binder activity at 170°C is based on the ratios calculated using the ITS parameter and summarised in Table 6.

Table 6. DoA estimation for RA’s – ITS

Analysis	Temperature (°C)	DoA RA1	DoA RA2
ITS Peak Load	70	47.2%	51.2%
	100	63.8%	61.0%
	140	80.6%	77.5%

	170	101.6%	107%

The following analysis is elaborated according to the previous results, by comparing the RAs and the Artificial RAs for each case at 170°C (highest activity), making it possible to assume these values as a reference. Applying Equation 8 and using the ITS results for the RA at 170°C as reference, the DoA” can be estimated as:

$$DoA''\ (\%) = 100 \times \frac{ITS_{RA}(X°C)}{\max \quad ITS_{RA}} \tag{8}$$

Where RA (X°C) is the ITS result of the RA at a specific temperature “X”.

The calculated DoA” results are shown as a function of conditioning temperature in Figure 14. The results show the high similarity for both the RA materials used in this study. A good correlation was found when using the 170°C ITS value as a reference.

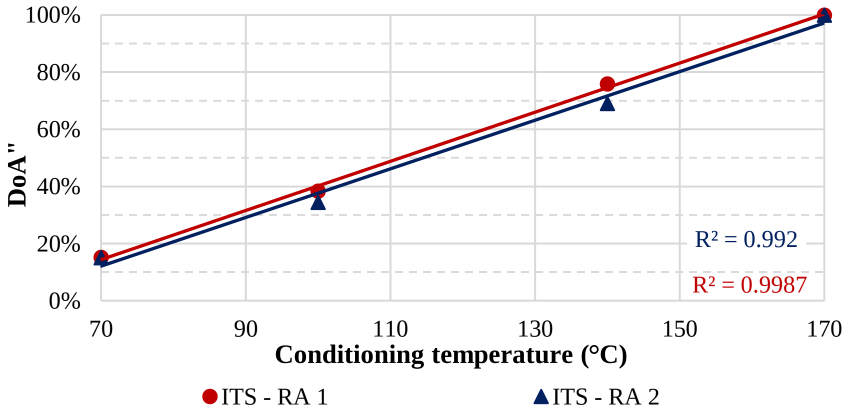

Figure 14. DoA estimation using RA at 170°C as reference.

Conclusions

The paper presents a methodology to assess the feasibility of using high RA percentages in asphalt mixtures. It is based on the availability (reactivation) of RA binder which can reduce the amount of virgin binder that needs to be added. According to the laboratory investigation, the following conclusions can be drawn:

- RA characterisation is essential when attempting to maximise RA content in new asphalt mixtures. This is especially relevant in terms of the recovered bitumen that guides the binder design for the new mixtures using virgin materials, rejuvenators or other available technologies.
- The high shear ageing procedure adopted to age the virgin bitumen to create an artificial RA binder proved to be efficient when adequately controlled. The procedure provided bitumen characteristics similar to that of the RA binder in terms of penetration and softening point as well as viscosity.
- Varying the conditioning and production temperature associated with the compaction of RA mixtures significantly affects the volumetric (air voids) and mechanical (ITS) properties with air voids decreasing and ITS increasing with temperature increases. The overriding mechanism responsible for these changes is the activity of the RA binder at higher temperatures and the consequential increase in mixture workability and compactability. The effect of increasing mixing times was found to be relatively insignificant.
- The ITS results showed a similar trend for 100% RA and Artificial RA with increasing peak loads in accordance with temperature rise. The Energy Pre-peak parameter also proved to be a useful property when considering other aspects of the ITT such as the load-displacement and the energy absorption. However, the Flexibility Index, Total Energy and Energy Post-peak do not show consistent correlations with temperatures and these parameters were therefore not considered useful for the DoA prediction.
- The proposed methodology to quantify the degree of bitumen mobilised from RA for new asphalt mixtures has shown positive results and can be used as a tool to better

understand RA in order to improve the binder/blend design for recycled asphalt mixtures. The proposed RA-DoA labelling methodology was successfully demonstrated for two types of RA. The DoA’ index proposed to improve the binder and mixture design is an easy parameter to calculated and useful tool for RA characterisation. The DoA’ analysis can be conducted on RA samples without any need for binder extraction although procedure does require the RA compositional properties to be known.

- The supplementary DoA” analysis (using RA conditioned and compacted at 170°C as the 100% activity reference) also presented satisfactory results and a good correlation with the DoA’ index. This simplified analysis does not require the production of the Artificial RA mixture or the detailed compositional analysis of the RA material.

Acknowledgements

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