**Novel Methodology to Investigate and Obtain a Complete Blending between RAP and Virgin Materials**

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# Abstract

Pavement engineers are continually attempting to resolve the problems associated with disposal of highway construction materials and conserve the natural resources in line with producing a more economically and environmentally friendly asphalt mixture. This study presents a novel methodology to investigate and obtain a complete blending in the laboratory using nano-mechanical approach so that designers can be more confident in estimating the performance of asphalt mixtures incorporating RAP materials. The study also presents an effect of warm additives, Sasobit and Rediset LQ on the level of blending between RAP and virgin materials. Images obtained from nanoindentation were validated using scanning electron microscopy (SEM) while level of blending was linked to the overall fatigue-cracking of asphalt mixtures which were measured using Dynamic shear rhemoter (DSR). It was revealed that although Rediset LQ improved the fatigue life and nano- mechanical properties of virgin interfacial transition zone (ITZ) and mastic phases, it has no effect on the diffusion of virgin binder into aged binder (binder surrounding RAP aggregate) or rejuvenating of aged binder while as Sasobit decreased the viscosity of binder, it has a great influence in accelerating complete blending between RAP and virgin materials. In summary, nanoindentation integrated with an optical microscopy is a powerful technique to investigate and obtain complete blending between RAP and virgin materials.

**Keywords:** HMA, WMA, Nanoindentation, SEM, DSR, RAP, Fatigue

# Introduction

Reclaimed asphalt pavement (RAP) is one of the most important recycling materials used to produce a sustainable asphalt mixture. The use of RAP is a valuable approach for paving, economic and environmental reasons. It was estimated that in 1999, the UK used of some 26 million tonnes (Mt) of hot mix asphalt (HMA) which can lead to the assumption that around 20 Mt of aggregate were consumed. Such using the amount of aggregate does not seem to be in line with the country’s strategy for sustainable construction that requires for minimising the consummation of natural resources (Huang *et al.* 2007). The reuse of recycled asphalt mixtures decreases the amount of waste produced and significantly helps to resolve the problems associated with disposal of highway construction materials and conserve the natural resources. In UK, the average RAP content increased to approximately 10% in 2015, and it is anticipated to continue rising (Hanson 2016). In Europe, data from 19 countries showed that 47% of the available RAP was reused in HMA and WMA, while about 22 million tonnes were used in other applications (Zaumanis *et al.* 2014). In the USA, approximately 100 million tonnes of reclaimed asphalt pavements are annually produced. Sixty million tons are reused in the construction of new asphalt pavement, while 40 million tonnes could be employed in other pavement applications. In Canada, over a period of 17 years, RAP was used to pave approximately 3,500,000 m2 (Association 2009,Jamshidi *et al.* 2013). Moreover, it was reported that using RAP in the base and sub-base layers of the pavement structure potentially reduced global warming by 20%, energy consumption by 16%, water consumption by 11% and hazardous waste generation by 6% (Lee *et al.* 2010). Despite many states in USA have reported limits on the maximum percentage of RAP that can be reused in the asphalt pavement structure in the range between 10 and 50% (Al-Qadi 2007), high percentages of RAP are not ordinarily used in practice because of owing to durability problems.

However, the level of blending between RAP and virgin (aggregate and binder) materials is one of the most important factors which directly influence the overall performance of asphalt mixtures and the economic competitiveness of the recycling process. The level of interaction between RAP and virgin materials is still ambiguous. To date, this interaction has not been investigated at the nano-scale, which can be of particular importance in reflecting the real level of interaction between materials.

An increasingly popular construction practice is to incorporate and increase the amount of RAP in WMA to produce a more economically and environmentally friendly asphalt mixture. However, two issues regarding the inclusion of RAP materials in asphalt mixtures need to be further investigated: the fatigue behaviour of RAP mixes and the degree of blending between RAP and virgin materials (Nahar *et al.* 2013). A recent study by Zaumanis and Mallick (2015) recommended that there is the need for a methodology to evaluate the level of blending between RAP and virgin materials in the laboratory. In the current study, the effect of warm additives and production temperature on the level of interaction between RAP and virgin material is investigated in detail using the advanced technology of nanoindentation, which can effectively investigate the real interaction between RAP and virgin materials based on nano-mechanical properties of the interfacial transition zone (ITZ) and mastic.

# Level of blending between RAP and virgin materials

The level of interaction that occurs between the RAP materials and the virgin materials directly influences the performance of asphalt mixtures and the economic competitiveness of the recycling process. If RAP materials are considered as a black rock and do not mix with the virgin materials during the mixing process, while RAP binder may mix and blend with virgin binder, then the total binder will stiffen, resulting in a stiffer mixture overall. However, if the designer assumes that the RAP will totally blend with virgin materials and actually it behaves as black rock, the mixture will not be stiff enough as insufficient asphalt binder is used. The issue is more complicated if there is a partial interaction between materials.

A limited number of researchers have investigated the level of interaction between aged and virgin asphalt binders (Al-Qadi 2007). McDaniel *et al.* (2000) in National Cooperative Highway Research Program (NCHRP) 9-12 investigated three possible levels of interaction between RAP and virgin materials: black rock (no blending), total blending (100%) and actual practice (mixing as it usually happens in practice). Two RAP percentages were considered in this study: 10 and 40% as the minimum and maximum percentage used practically. In all scenarios, the gradation and the total asphalt binder content were constant. It was found and concluded that, at 10% of RAP, no remarkable difference was reported between the various blends, whereas the black rock assumption was statistically not similar to the actual practice and the total blending at 40% of RAP content. In other words, out of 66 possible comparisons made, there were 11 and 16 inconclusive cases at 10% and 40% RAP, respectively. With 10% RAP, the majority of the cases (about 70%) supported the conclusion that all cases were similar; on the other hand, only 42% of the comparisons supported the assumption of total blending. However, the authors believed that it was unlikely that total mixing occurred in all scenarios, even with high RAP content. From the results, it was indicated that there is no requirement to change the binder grade at low RAP content, and total blending between RAP and virgin materials can be assumed at higher RAP percentages.

Oliver (2001) also investigated the influence of RAP binder on recycled asphalt pavement. Artificial RAP material was manufactured in the laboratory by using bitumen class 320 similar to a 50/65 binder grade. The loose mix was exposed to high temperatures in the oven in order to oxidise and harden the binder film. It was then compacted and broken up to produce artificial RAP. After that, 50% of the manufactured RAP was mixed with 50% of new HMA (binder grade 180/200) and compacted again. Binder was recovered from the recycled asphalt mixture in order to prepare a binder with similar viscosity to the recovered-recycled binder. The viscosity of the extracted binder was 29500 Pa.s at 45$°$C. Class 600 bitumen was oxidised by heating and then blended with 23% of Class 170 bitumen in order to obtain the same viscosity value of extracted recycled binder. In this way, a manufactured mix identical to the recycled asphalt was produced but with virgin materials. The performance of the two mixes should have been similar; however, their performance in terms of rutting and fatigue resistance was different, and it is postulated the recycled and virgin binders may not completely mix.

Stephens *et al.* (2001) also conducted a study to evaluate the degree of blending between RAP and virgin binders in terms of the RAP preheating time before being mixed with conventional materials. The RAP preheating time was varied from zero to 540 minutes. The preheating time should not have any effect on the mix properties if RAP is considered as black rock. It was in fact found that the preheating time had a considerable effect on the mixture strength, indicating that blending does occur between aged and virgin binders. This finding corresponds with the findings of a study conducted by Nguyen (2009), who also reported that long RAP reheating time, which never occurs industrially, enhanced the reaction between RAP material and virgin materials. Based on his findings, he deduced that RAP material does not act as black rock, nor is it fully blended in recycled asphalt production.

Huang *et al.* (2005) also conducted a study to investigate the level of interaction. RAP materials (passing through No. 4 sieve only) were mixed with virgin coarse aggregate (retained by No. 4) to assess the blending due to pure mechanical mixing. The aim was to find out to what extent the aged binder can get away from the RAP particles under pure mechanical blending, as RAP materials and virgin coarse aggregate can easily be separated after mixing to assess the residual binder content in the RAP. It was found that the asphalt binder content in the RAP was decreased only by about 11% due to pure mechanical mixing. However, the use of pure mechanical mixing alone is not sufficient to determine the level of interaction between RAP materials and virgin materials because asphalt binder can diffuse in the RAP, and the intermixing between recycled and virgin binders can also rejuvenate the aged binder, resulting in more interaction between aged and virgin binders. The researchers also conducted staged extraction and recovery for a mixture of one type (20%) of screened RAP with virgin coarse aggregate. After blending, it was found that the outside layers of the RAP particles were much softer than the inside layers in terms of binder stiffness. In other words, the experiment indicated that only a small portion of recycled asphalt binder in the RAP (40%) indeed participated in the remixing process, while other portions (approximately 60%) did not have any interaction with the virgin binder and acted as composite black rock.

Shen *et al*. (2007a) studied the effects of rejuvenator agents on Superpave mixtures containing RAP materials. They found that the properties of the recycled mixtures using oily rejuvenator were better than that containing softer binders. Furthermore, Shen *et al*. (2007b) also investigated the effects of rejuvenator agents on aged crumb rubbers modified binders. Their results indicated that the compositional changes of the blends containing various percentages of either the rejuvenator or the softer binder were well reflected by the permeation chromatography. However, those studies by Shen *et al.* (2007a, 2007b) did not address how much aged binder can get away from RAP aggregate to change the properties of virgin binder.

More recently, experimental investigation of the homogeneity of aged binder and virgin binder has been conducted by Eddhahak-Ouni *et al.* (2012) using conventional tests and infrared spectroscopy analysis to assess and quantify the evaluation of the mass proportions of RAP and virgin binders during the stripping process (percentage of extracted bitumen). They found that poor remobilisation of the virgin binder in the blended binder correlated with the hypothesis of the heterogeneous asphalt.

With more recent innovations in WMA technologies, the issue of the level of interaction between RAP and virgin materials is more complicated because of the lower production temperature of the WMA. Research carried out by the NCHRP to assess the level of interaction between aged and conventional binders at WMA temperatures revealed that the aged binder and conventional binder do mix when they are subjected to elevated temperatures; however this may not be valid in the actual mixture as the study only used solvent casting of a thin film of new binder on a simulated RAP binder (Bonaquist 2008). Furthermore, as shown by Nahar *et al*. (2013) that the interface between the RAP binder and the virgin binder was completely blended. They used a combination of DSR and atomic force microscopy (AFM) to show that. Although, a complete blending was observed in AFM images and confirmed by DSR, the results did not rule out the black rock hypothesis altogether.

Bowers *et al.* (2014) also investigated the effect of mixing time, mixing temperatures and the inclusion of WMA additives (surfactant beads and wax-based additives) on the efficiency of blending between RAP and virgin materials. The study involved a large virgin aggregate, a PG64-22 virgin asphalt binder and a fine RAP aggregate. Upon separation, rheology and molecular weight distribution of the recovered binder from coarse and fine aggregate were determined using DSR based on master curves, and Gel Permeation Chromatography (GPC) based on large molecular size was conducted. Several conclusions were reported in this study. Firstly, mixing temperature has a higher impact on the level of blending that the effect of mixing time. It was also reported that the blending ratio increased from 59% at 130$°$C to 70% at 180$°$C. Secondly, including warm additives had a positive effect on blending ratio: a 76% blending ratio was achieved using surfactant-based WMA produced at 130$°$C for 105s, which was equivalent to that of the control mixture, mixed at 160$°$C for 150s. It was also found that the wax-based WMA was the most workable mixture; therefore, a suggestion was made to investigate the effect of wax-based additives on the level of blending at higher temperatures.

Diffusion of virgin asphalt binder to the recovered binder of RAP is further being investigated. It has been reported that the current AASHTO (American Association of State Highway and Transportation Officials) *M323* specification which recommends using one softer performance grade asphalt at higher than 15% of RAP may not be justified. This fact has been exhibited by Kriz *et al.* (2014), who demonstrated that it is unnecessary to change the binder grade up to 25% of RAP. The ability of RAP binder to blend with virgin binder by diffusion mechanism was measured in a DSR and the results showed that complete binder blending in both HMA and WMA applications was reached a few minutes after mixing. A similar study was conducted by Rad *et al.*(2014). The rate of diffusion of virgin asphalt binder into the RAP binder was investigated using two layers of binder (1mm of virgin binder and 1mm of artificial RAP binder) tested in a 2mm gap of DSR. It was reported that the rate of diffusion increased with increasing temperature as the viscosity of asphalt binder decreases at higher temperatures. More importantly, the chemical compositions of the asphalt binder had an effect on the rate of diffusion. Therefore, it is hypothesised that other factors rather than viscosity can have an effect on the rate of diffusion of virgin binder into RAP binder.

Mobilisation rate of recycled binder was investigated by Zhao *et al.* (2015). In that study, a laboratory procedure to quantify the rate at which aged binder could be mobilised and made available to coat aggregate was developed based on a new term called large molecular size percentage (LMSP) derived from GPC testing for recovered binder. The researchers reported the RAP binder mobilisation rate decreased with increase in the percentage of RAP. It was noticed the RAP binder mobilisation rate was close to 100% at low RAP mixtures (10% and 20%); however, as the RAP percentages increased from 30% to 80%, the rate of mobilisation decreased from 73% to 24%.

Based on the above information, one should note that the degree of blending between aged and virgin binders still needs to be further investigated. In fact, most studies investigating the level of blending have been based on the traditional mechanical properties or only the level of blending between aged and virgin binders, which does not rule out the black rock hypothesis or complete blending assumptions. Therefore, one of the main objectives of this study is to directly investigate the level of blending between RAP and virgin materials using the advanced technology of nano-mechanical properties, as the author believes the sophisticated technology of nanoindentation can be practically adopted in order to characterise the mechanical properties of the interfacial zone between RAP materials and virgin binder with the advantages of including warm additives in order to gain more understanding about the level of interaction at the micro- and nano-scales.

# Materials and methods

## *Materials*

Two binder grades and two warm additives are used in this study; the virgin aggregate type adopted in this study is granite, which was supplied by Croft Quarry, Aggregate Industries, UK. Asphalt binders were supplied by Nynas, while warm additives were supplied by their subsidiary companies, Naylor Chemicals Ltd, UK and AkzoNobel, Sweden. It should be noted that the recommended dosages of Sasobit, and Rediset LQ which were adopted in this study are 2% and 0.5% by the weight of the bitumen respectively. Reclaimed asphalt pavement (RAP) was supplied by Hanson Aggregates, UK and binder content and recovered aggregate gradation were determined by Pavement Testing Services Ltd (pts), UK. The penetration and softening points were identified by the authors. **Table 1** illustrates the properties of virgin binders and recovered RAP binder.

## *Mix Design*

A gap-graded hot rolled asphalt was adopted to manufacture HMA, WMA HMA-RAP and WMA-RAP. The nominal maximum aggregate size is 10mm, which is suitable for surface courses. The recipe specification method was adopted in the design of the mixes; this method provides details for the aggregate type and gradations and binder grades for particular mixes. The HMA and WMA incorporating RAP mixtures were manufactured according to the British Standards recipe (BS 597-1 2005). It should be noted that virgin aggregate was heated overnight at the required mixing temperature whereas, RAP was at the required production temperature four hours prior to mixing process. **Fig.1** illustrates the particle size distribution of the virgin aggregates and RAP aggregate for HMA and WMA. For each mixture, the mixture code was defined as follows H: Hot Mix Asphalt; W: Warm Mix Asphalt; Second H: hard binder (40/60) grade; S: Soft binder (100/150) grade; Sa: Sasobit; Rl: Rediset Liquid. For example, HH155 and HS145 are the controlled hot mixes manufactured using (40/60) and (100/150) binder grades respectively. WSSaP125 and WSSaP155 are the Sasobit-modified warm asphalt mixtures manufactured at 125$°$C and 155$°$C including RAP respectively.

## *Assumptions for including RAP*

A complete blending between RAP and virgin materials was assumed in all mixture. It was found that, when 40% of recovered RAP binder was mixed with 60% of 100/150 binder, the new binder behaved as the 40/60 binder. Frequency sweep tests were performed under controlled strain, and frequencies between 0.1 and 10 Hz were adopted over a temperature range between 0$°$C and 60$°$C at intervals of 10$°$C. **Figs 2** and **3** illustrate the master curves of complex shear modulus and phase angle vs. frequency for the control 40/60 binder and the new binder obtained from mixing 40% of the recovered RAP binder and 60% of the 100/150 binder. It is clear that the properties (complex shear modulus and phase angle) of the new binder are exactly same as the virgin 40/60 binder, and its viscoelastic response behaviour during the dynamic test under different loading conditions and testing temperatures is equivalent. Consequently, for all mixtures containing RAP materials, through manufacturing asphalt mixtures, a complete blending was assumed. Five mixtures were manufactured incorporating RAP materials: HSP145, WSSaP125, WSSaP155, WSRlP125 and WSRlP155. For example, HSP145 is hot mix asphalt manufactured at 145$°$C using 60% of virgin materials (100/150 binder and virgin granite) with 40% RAP material, while WSSaP125 is a warm mix asphalt manufactured at 125$°$C using 60% of 100/150 and 60% of virgin aggregate with the addition of 2% of Sasobit mixed with 40% of RAP.

## *Samples preparation*

After manufacturing the asphalt mixture slab, firstly a beam of (305 × 65 × 25 mm) was discarded from one side of the slab and then two beams of (305 × 65 × 50 mm) were cut using a diamond cutter. The beams were tightly fixed on the wooden prisms using tape to prevent them moving whilst the cylindrical samples were being cored as shown In **Fig 4.a**. This ensures that damage to the samples is avoided when the coring pet punches from the top face to the bottom face of the beam, and moreover reduces the vibration. The cylindrical samples of 25 mm in diameter and 50 mm in height were cored from the beam to prepare disc samples for nanoindentation. In this regard, the cylindrical DSR samples (12 mm in diameter and 50 mm in height) were also cored using an electric coring machine with continuous water. 34 cylindrical DSR samples and two cylindrical nanoindentation specimens were obtained from each beam. Four cylindrical samples were obtained from each mix as presented in **Fig 4.b**.

A small cutter was used to prepare nanoindentation disc samples 25 mm in diameter and 10 mm in height as shown in **Fig 5.a**. The nanoindentation disc samples (NDSs) as presented in **Fig 5.b** and DSR samples were left to dry for 24h at ambient temperature. They were then put in an oven maintained at 20$°$C for half an hour to ensure they were completely dry. After that, bulk densities for all DSR samples were measured based on (BS EN 12697-6 2012), and, during the measurements, the samples were coded and kept in PVC tubes, and then stored in a fridge at 10°C.

It is essential that asphalt mixture samples tested in nanoindentation have a very smooth surface because the contact area is measured indirectly from the depth of penetration. Therefore, if the surface is rough or not adequately polished, errors in determining the area of contact may lead to obtaining inaccurate results for nano-mechanical properties. A custom specimen preparation holder as shown in **Fig 6** was used for polishing the nanoindentation disc samples. Each nanoindentation disc sample was fixed on the brass bar using double-sided tape. The brass bar was used to load the sample onto the grinding paper of polishing machine.

In order to adequately level the sample during polishing, slices of Alumina sheet were used. Firstly, a grinding machine as shown in **Fig 7.a** rotating at an angular speed of 300 rpm was used to polish the sample to approximately 5 $μm$ using a sequence of Silicon Carbide Abrasive (SiC) papers of decreasing abrasiveness (120, 180, 320, 600, 1200, 2500 and 4000) under continuous cold water and then a grinding machine rotating at an angular speed of 150 rpm using fine aluminium powder and wet polishing cloth paper was used to obtain a finishing of 0.05 $μm$ (approximately 49 nm) as presented in **Fig 7.b**.

## *Methods*

Nanoindentation testing was conducted using a Nano Indenter G200 (Keysight Technologies, Chandler, AZ, USA) with a Berkovich indenter probe. The indenter was used as per the Hardness-Modulus at depth method based on Oliver and Pharr’s theory (Oliver and Pharr 1992). The strain rate target was 0.05 1/s, while peak hold time was 10s and surface approach velocity was 10 nm/s. Calibration was also conducted using standard fused silica before and after testing each batch of samples to ensure that there was no dust on the head of probe, and also to ensure that all obtained results were reliable. Poisson’s ratio was also assumed to be 0.3 for all asphalt mixture settings. All tests were conducted at 22°C and the temperature was controlled using an A/C. 20 points of measurement were conducted in each phase for each sample. Three samples were tested for each mixture

A JSM 6610 SEM was used to further investigate the level of blending between RAP and virgin materials and validate images obtained under the optical microscope of nanoindentation. The electron beam was focused on spot of 50nm while the accelerating voltage was 20Kv (20000 volts) at working distance of 9-10mm.It should be noted that, for this purpose, samples were coated with a thin layer (20-100nm) of conductive material, chromium, in order to prevent charge build-up.

In order to conduct fatigue testing and assess the overall performance of the mixtures, modifications were conducted on the DSR in order to use it to run sequences on cylindrical DSR samples. These modifications included design and manufacturing end alterations and adjusting the holder connections that are used to hold and set up samples in the proper position for testing. Moreover, in order to control the sample temperature during testing, a temperature controller unit was designed and added to the DSR as an essential component. Fatigue measurements were conducted using controlled-stress mode at 250KPa and 425KPa for control mix HS145 and HH155 respectively, while the testing temperature was 25$°$C. Five samples were tested for each mixture.

# Results and discussion

## *Nano-mechanical properties of mixture phases*

### *Mechanical properties of aggregate*

The RAP aggregate was a combination of sandstone, limestone and gabbro. RAP aggregates were identified by a geologist in the Geology Department, University of Liverpool, UK. Elastic modulus and hardness of the RAP aggregate were measured and compared to those of the virgin granite. **Fig 8.a** and **Fig 8.b** show the elastic modulus and hardness of sandstone, gabbro and limestone. The elastic modulus of all RAP aggregate was equal to or higher than the elastic modulus of the virgin granite. However, it was expected that the hardness of limestone would be less than that of the other types of aggregate, as limestone is considered weaker than granite. The most important consideration in this paper is that the vast majority of the RAP aggregate was a combination of Sandstone and Gabbro. Therefore, in the matter of investigating the level of blending between RAP and virgin materials, the effect of the aggregate can be neglected as the properties of the RAP aggregate are approximately equal to the properties of the virgin granite.

### *Mechanical properties of ITZ and mastic*

The level of blending between the RAP and the virgin materials was investigated using nanoindentation. **Fig 9.a** illustrates the ITZ (interfacial transition zone between virgin aggregate and virgin binder). As can be seen from that figure, there was no gap between the virgin aggregate and the binder/mastic, and so the elastic modulus and hardness of ITZ in this mixtures can be measured directly. However, in the case of incorporating RAP materials, **Fig 9.b and Fig 9.c** explain the ITZ virgin and ITZ RAP (interfacial transition zone between RAP aggregate and RAP binder). It is clear that the binder surrounding the RAP aggregate was not completely affected by the virgin binder. In other words, there was no complete interaction between the old and the virgin binder, as the virgin binder was not completely diffused into the old binder and the old binder did not get way from the RAP aggregate due to the high stiffness of the ITZ RAP. However, in the same mixture, a nice interaction between the virgin binder and virgin aggregate can be observed, as shown in **Fig 9.c**

This phenomenon was also noticed in mixtures WSRlP125, WSRlP155 and WSSaP125. **Figs 9.d** and **9**.**e** illustrate the ITZ RAP of mixtures WSRlP125 and WSSaP125 respectively. In those figures, complete blending was not satisfied. However, the effect of Sasobit on the level of blending in mixture WSSaP155 was very considerable. Sasobit has the potential to significantly decrease the viscosity of the binder, which increases diffusion of the virgin binder into the old binder; also, it is expected to decrease the viscosity of the old binder; therefore, the old binder can easily get away from the RAP aggregate, and so a complete blending can occur. This phenomenon was confirmed as presented in **Fig 9.f**. It is very clear that complete blending occurred and the ITZ RAP disappeared and therefore a complete interaction between the aggregate and the binder/mastic can be noticed.

Confirmation of the aforementioned scenarios can be made based on the mechanical properties of the ITZ RAP, ITZ virgin and mastic. **Figs 10.a** and **10.b** show the elastic modulus and hardness of the ITZ RAP compared to the ITZ virgin of the control mix HH155. It is clear that the ITZ RAP of mixtures HSP145, WSSaP125, WSRlP125 and WSRlP155 was statistically stiffer and harder than the ITZ virgin of HH155 (P $<$0.05) as shown in **Table 2.** This is expected because the binder surrounding the RAP aggregate must be aged for a long time due, perhaps, to some factors such as: oxidation, volatilisation, polymerisation, thixotropy, etc. (Al-Qadi 2007,Roberts *et al.* 1996,Karlsson and Isacsson 2006). However, it can be observed that, at a mixing temperature of 155$°$C, the mechanical properties of the ITZ RAP of mixture WSSaP155 are a bit higher than those for the ITZ virgin and lower than those for mixtures HSP145, WSSaP125 and WSSaP125. The reason for that is, at high mixing temperatures, the old binder covering the RAP aggregate starts melting and getting away from the old aggregate. The reduction in the viscosity because of Sasobit has also accelerated this process and the new binder (blending old and virgin binders) with the effect of Sasobit has enhanced the elastic modulus and hardness of the ITZ virgin of mixture WSSaP155, as presented in **Figs 11.a** and **11.b** respectively. It is therefore clear that the elastic modulus and hardness of the ITZ virgin of WSSaP155 is the same to that of HH155 as there was no significant difference in the mean elastic modulus and hardness between ITZ of WSSaP155 and that of HH155 (P $> $0.05). Moreover, there was no significant difference in the mean elastic modulus and hardness of mastic for WSSaP155 and HH155 as presented in **Figs 12.a** and **12.b** and **Table** **2** which shows the results of ANOVA test for every mixture phase. The reason of that is because the extracted aged binder contributes to increase the elastic modulus and hardness of mastic due to the complete blending between RAP and virgin materials. It can therefore be concluded that complete blending was achieved using Sasobit.

Although at a mixing temperature of 155°C, the ITZ RAP of mixture WSRlP155 seems partially melted, as shown in **Figs** **10.a** and **10.b**, elastic modulus and hardness of ITZ virgin of WSRlP155 were statistically less than those for HH155. The reason for that is because Rediset LQ has no effect on the viscosity of the binder, in which blending between old and virgin binder is unlikely to occur. Furthermore, there was no extracted binder from the old aggregate contributing to improving the properties of the ITZ virgin and mastic as presented in **Figs 11** and **12**. Despite the fact that the mastic properties of the HSP145 were statistically the same as the properties of HH155, because of the presence of fine particles of RAP aggregate, the properties of the ITZ virgin of HSP145 were statistically less than those of HH155 due to the same reason mentioned previously in the case of mixture WSRlP155 and the mean elastic modulus and hardness were significant (P$<$0.05). On the other hand, because Sasobit has a superior performance in improving the mixtures stiffness, the ITZ virgin of WSSaP125 is stiffer and harder than that of HSP145 but still statistically lower than those of HH155 because of the effect of production temperature and incomplete blending between old and virgin binders.

As presented in **Table 2**. It can be noticed that complete blending was only obtained in one scenario in the case of mixture WSSaP155, as there was no statistical difference between the mechanical properties of ITZ and mastic of this mixture and those for control mix HH155 (P$>$0.05) as mentioned previously. It can therefore be concluded that Sasobit has the potential to help the virgin binder diffuse into the old binder, and also has an effect on the mobilisation of the old binder only if the effect of production temperature is considered.

## *SEM*

Further investigation on the level of blending between RAP and virgin material was conducted using a scanning electron microscopy (SEM). The SEM offers high-resolution images to investigate the degree of blending between RAP and virgin materials. Although the images obtained using optical microscopy integrated in nanoindentation were very clear in showing the interfacial transition zone between the virgin aggregate and virgin binder and also between the aged binder and RAP aggregate, those images can also be validated using a SEM. As can be seen in **Fig 13.a**, there is complete interaction between the virgin aggregate and virgin binder, as was previously seen in **Fig 9.a**. However, **Fig 13.b** clearly illustrates the ITZ RAP at magnifications of x1000 and x5000. It is obvious that the aged binder still covered the RAP aggregate and there is no complete blending between virgin and old binders.

As obtained and proved previously, Sasobit has a superior performance in satisfying complete blending between RAP and virgin materials. As presented in **Fig 13.c**, the region representing the old binder covering the RAP aggregate disappeared and complete interaction between the mastic and RAP aggregate can be seen, as was also noticed under the optical microscope of nanoindentation, as aforementioned and shown in **Fig 9.f**.

***Fatigue***

More attention should be paid to the fatigue life of asphalt mixtures because the greater the RAP content, the more brittle are the materials included in the mixture. It is therefore important to investigate the level of blending based on the fatigue characterisation of asphalt mixtures. As proved previously, partial blending between RAP and virgin materials was noticed in all HMA-RAP and WMA-RAP apart from WSSaP155, as its nano-mechanical properties of mixture phases were higher than the control mix HH155, which means complete blending occurred. Fatigue tests were conducted for those mixtures to estimate the fatigue life of WMA-RAP. Investigations were carried out under two scenarios, as follows:

* First scenario: fatigue tests were conducted for mixtures HSP145, WSSaP125 and WSRlP125 at controlled stress value of 250KPa and testing temperature of 25$°$C so a comparison could be made with the control mix HS145.
* Second scenario: fatigue tests were conducted for mixtures HSP145, WSSaP125, WSRlP125, WSRlP155 and WSSaP155 at controlled stress value of 425KPa and testing temperature of 25°C so a comparison could be made with the control mix HH155.

In the first scenario, **Fig 14.a** shows number of cycles at a reduction of stiffness modulus of the sample to 50% of its initial value, because it was impractical to obtain a complete fracture failure or reduction to 10% of its initial value as the fatigue test took a very long time. It was therefore decided to stop the fatigue test when the complex shear modulus reached 50% reduction from its initial value. As can be seen in that figure, HSP145 exhibited a higher number of cycles until reaching 50% compared to HS145. And, although WSSaP125 and WSRlP125 were manufactured at 125$°$C, practically, their fatigue lives were longer than that of HS145. This finding agrees with Huang *et al.* (2004a, 2004b). Consequently, the common belief that the more RAP, the more brittle the mixture, thus, the lower the fatigue resistance, is contradicted. It can be therefore confirmed that there was a partial blending between RAP and virgin materials in those mixtures as the fatigue life of HSP145, WSSaP125 and WSRlP125 were longer than that of control mix HS145. However, in order to further investigate whether a complete blending occurred or not, fatigue tests were conducted for those mixtures at 425KPa so that a decision could be made according to their fatigue performance with HH155.

**Figs 14.b** and **15** present the results of the fatigue tests in the second scenario. It is clear that the number of cycles at the failure point of mixtures HSP145, WSSaP125, WSRlP125 and WSRlP155 was lower than that of HH155, which confirmed the fact that there was no complete blending between RAP and virgin materials. From a fatigue perspective, this region may be considered as a weak region because of the incomplete blending; therefore, under apply higher load, cracks will likely start to initiate and develop over time. Furthermore, the deficiency of mastic to resist the applied higher load accelerates initiating and developing of cracks over time. This assumption is supported by the conclusion study of Mohajeri *et al.* (2012) who studied the relation between cracking resistance of asphalt mixtures and the degree of blending between RAP and virgin binders. It was reported that the degree of blending between RAP and virgin binders seems to governing the mechanical properties of asphalt mixtures.

 Despite the fact that WSRlP155 was manufactured at 155°C with the addition of 0.5% of Rediset LQ, no complete blending was observed, whereas WSSaP155 exhibited approximately the same fatigue life as HH155. The reason for that is because Sasobit is a viscosity reducer and stiffness improver. It can therefore be concluded that a complete blending between RAP and virgin materials occurred in the mixture of WSSaP155.

## *Proposed methodology to obtain complete blending*

Based on what has been presented in this study, a simple methodology is proposed to obtain a complete blending between RAP and virgin binder, which is a key component of suitable practices in the pavement industry. If a complete blending can be reached, natural resources can be significantly conserved and also a more sustainable and cost-effective pavement structure can be constructed. Moreover, pavement designers can be more confident to incorporating RAP materials and accurately estimate the overall performance of the asphalt mixture. **Fig 16** illustrates the steps that can be used to reach a complete blending between RAP and virgin materials.

**Conclusion**

Level of blending between RAP and virgin materials is a point of debate among pavement researchers. As aforementioned, to date, this interaction has not been investigated at the nano-scale, which can practically reflect the real level of materials’ interactions. In the current study, the effect of warm additives of Sasobit and Rediset LQ and production temperatures on the real interaction was also investigated using nano-mechanical properties of ITZ and mastic and fatigue. Results were further validated using scanning electron microscopy. The main findings of this study are:

1. It was revealed that, in the incomplete blending scenario, the old binder is still covering the RAP aggregate. However, it was found that the production temperature and Sasobit have a substantial effect on the mobilisation of aged (old) binder and the diffusion of virgin binder into old binder.
2. Once the aged binder covered RAP aggregate disappears, complete blending between RAP and virgin materials can be reached, which can be proved by the mechanical properties of ITZ RAP, ITZ virgin and mastic.
3. Although Rediset LQ can improve the performance of WMA-RAP compared to the control mix, it has no effect on the rejuvenation of aged binder as the purpose of this additives is to decrease the fractional forces at the interfaces of the aggregate and binder without reducing the viscosity of the binder, which is the most important factor that affects the rejuvenation of aged binder and the diffusion of virgin binder.
4. Inclusion of up to 40% of RAP materials with WMA can produce an asphalt mixture with better performance than the traditional HMA in terms of fatigue life; therefore, RAP materials must not be considered as a black rock.
5. A simple methodology has been proposed to reach complete blending between RAP and virgin materials.

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**Table 1.** Properties of virgin and recovered RAP binders

|  |  |  |
| --- | --- | --- |
| Binder grade | Penetration at 25$°$C (mm) | Softening point $°$C |
| 40/60 | 45 | 54.3 |
| 100/150 | 104 | 43.0 |
| Recovered RAP binder | 15 | 65.5 |

**Table 2**. Results of ANOVA test for every mixture phase (α=0.05)

|  |  |  |  |
| --- | --- | --- | --- |
| Mixtures  | Elastic modulus  | Hardness | Significance |
| *ITZ virgin*  | *ITZ RAP* |
| *F* | *P-value* | *F critical* | *F* | *P-value* | *F critical* |
| *HH155* | *HSP145, WSSaP125, WSSaP155, WSRlP125, WSRlP155* | 26.05604 | 9E-06 | 4.091279 | 9.532115 | 0.003709 | 4.091279 | YES |
| *ITZ virgin*  |   |
| *HH155* | *HSP145* | 26.05604 | 9E-06 | 4.091279 | 9.532115 | 0.003709 | 4.091279 | YES |
|  | *WSSaP125* | 8.456838 | 0.006667 | 4.159615 | 14.16602 | 0.000701 | 4.159615 | YES |
|  | *WSSaP155* | 0.275265 | 0.602516 | 4.067047 | 0.270806 | 0.605461 | 4.067047 | NO |
|  | *WSRlP125* | 68.06081 | 6.58E-10 | 4.105456 | 43.10736 | 1.08E-07 | 4.105456 | YES |
|  | *WSRlP155* | 43.34783 | 2.93E-08 | 4.038393 | 15.69861 | 0.000241 | 4.038393 | YES |
| *Mastic*  |   |
| *HH155* | *HSP145* | 0.064074 | 0.804128 | 4.667193 | 4.323601 | 0.057944 | 4.667193 | NO |
|   | *WSSaP125* | 0.087542 | 0.771132 | 4.493998 | 8.300429 | 0.01086 | 4.493998 | YES |
|   | *WSSaP155* | 3.158423 | 0.094548 | 4.493998 | 0.166487 | 0.688661 | 4.493998 | NO |
|   | *WSRlP125* | 27.6608 | 1.91E-05 | 4.241699 | 75.58687 | 4.99E-09 | 4.241699 | YES |
|   | *WSRlP155* | 4.902428 | 0.039245 | 4.38075 | 0.946548 | 0.342824 | 4.38075 | NO |