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Impact of laboratory mixing procedure on the properties of reclaimed asphalt pavement mixtures

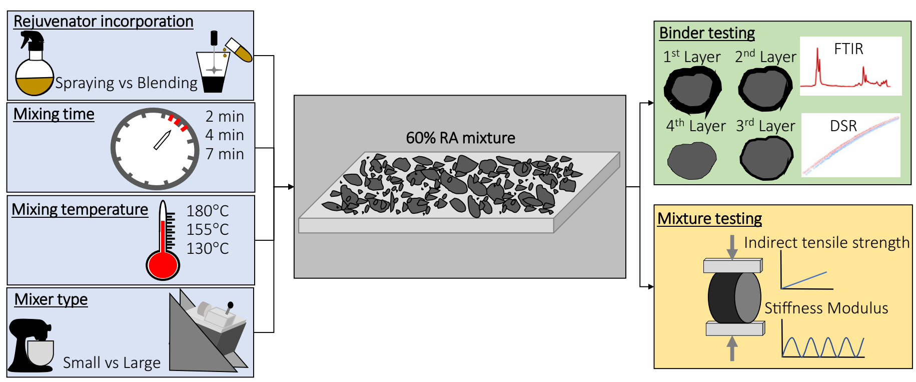
Mukul Rathore [a], Martins Zaumanis [b,*]

[a] Celu Eksperts, Aveņu iela 1, Ikšķile, Ikšķiles novads, LV-5052, Latvia
[b] Empa Swiss Federal Laboratories for Materials Science and Technology, Überlandstrasse 129, CH-8600 Dübendorf, Switzerland

HIGHLIGHTS

- Stage extraction method was used to obtain four layers of bitumen from mixtures.
- Rejuvenator addition method did not significantly affect the mixture properties.
- Increase in mixing time did not significantly increase the stiffness of mixtures.
- Mixing temperature was crucial parameter to control excessive aging of mixtures.
- Mixer type affected the degree of blending between aged binder and virgin bitumen.

GRAPHICAL ABSTRACT



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ABSTRACT

Reclaimed asphalt (RA) mixtures have been extensively studied over the last decade, but there is no common procedure for producing high RA mixtures in the laboratory. This study evaluates the effect of asphalt mixing parameters, including rejuvenator incorporation method, mixing temperature, mixing time, and mixer equipment on properties of high RA content mixtures. The asphalt mixtures containing 60% RA material were evaluated using the indirect tensile strength test and the stiffness modulus test. In parallel, a stage extraction method was developed to obtain four layers of bitumen from loose mixtures and then further analyzing them using the dynamic shear rheometer and the Fourier transform infrared spectroscopy. The results identified mixing and heating temperature as an important parameter that must be carefully controlled to avoid excessive aging of the binder. It was also found that laboratory mixer equipment could affect the degree of blending and hence should be taken into account for comparing mixtures. Based on this study, a mixing procedure is recommended for producing high RA mixtures using the rejuvenator in the laboratory.

1. Introduction

The use of reclaimed asphalt (RA) material in hot mix asphalt started in 1915 but became a common practice in the mid-1970s due to a rapid increase in asphalt binder prices [1]. Over the last decade, this topic has been extensively explored by researchers

* Corresponding author.
E-mail addresses: mukul.rathore@edu.rtu.lv (M. Rathore), martins.zaumanis@empa.ch (M. Zaumanis).

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and asphalt producers due to its environmental and economic benefits. The current emphasis is on increasing the percentage of RA material in asphalt mixtures to maximize the benefits of RA usage. There have been several attempts to demonstrate that mixtures containing even up to 100% RA material can perform equal to or better than conventional hot mix pavements [2–4].

In most of the studies, however, the laboratory mixing parameters such as rejuvenator incorporation method, mixing temperature, mixing time, and mixer equipment are only vaguely described. If these parameters are not properly controlled, the results might not be reproducible. In addition, the mix design of high RA mixtures involves certain assumptions [5,6]. Researchers generally agree that the RA binder does not behave as a "black rock" (a scenario when aged binder does not take part in mixing); rather, but the extent of active blending between RA binder and virgin binder depends on the mixing parameters. Imaging techniques have shown the existence of a partial black rock effect and a layered structure surrounding the RA aggregates, and the thickness of RA binder was found to be affected by mixing temperature [7]. This indicates that the chemical and physical properties of RA bitumen film may change as a result of the mixing process which needs to be taken into account for the mixture design.

Conventionally, for production of hot mix asphalt in the laboratory, at first, all mineral aggregates are mixed (dry mixing), followed by pouring the bitumen on aggregate mixture (wet mixing) which is continued for the desired mixing time (until a homogeneous mix is obtained) [8,9]. In high RA mixtures, a rejuvenator is often added to restore the viscosity and elasticity of the aged binder [10]. These rejuvenators are generally pre-blended with virgin bitumen but can also be mixed with heated or unheated RA [11]. In a plant study, a conventional approach of adding rejuvenator on hot RA into mixer was compared to a rather innovative approach where rejuvenator was sprayed on cold RA over a conveyor belt [12]. No significant difference was observed from extracted binder properties between the rejuvenation methods. However, the mixture test results demonstrated potentially improved fatigue life when the rejuvenator was sprayed on RA, due to an improved blending of the materials [13]. The mixing conditions in the asphalt plant are completely different from the laboratory. Therefore, the impact of the rejuvenator incorporation method on asphalt mixture properties may be explored in laboratory-scale studies. A study has shown that some variation in the traditional mixing method can also significantly improve the performance of high RA mixtures [9]. Therefore, different sequences of mixing the aggregate components and bitumen may also be investigated to optimize the mixing method for high RA mixtures.

It is preferred to avoid high heating temperatures for RA material to limit the aging of the already oxidized RA binder and to prevent sticking of the RA binder to the heating facility [14]. In the laboratory, a heating temperature of 110 °C for a time of no more than 2 h was recommended since higher temperatures and longer heating times have shown to substantially change the properties of RA materials [15]. However, to achieve a high RA content in the mixture, it becomes practically impossible to ensure the required mix discharge temperature without heating the RA to a high temperature. A laboratory study on 50% RA mixtures showed that virgin aggregates were required to be heated at 220 °C to reach a resultant mix temperature of 150 °C, when the RA material was heated at 120 °C [16]. Additionally, lower preheating temperatures of RA may also result in higher air voids which may lead to consolidated rutting in the mixture and increase in moisture damage [9,17].

Mixing time could be another important parameter to be controlled in the laboratory studies. The mixing time in an asphalt plant is much shorter compared to mixing time normally used in a laboratory setting. Therefore, the laboratory studies could be overestimating the degree of blending between the RA binder and virgin binder. It was shown in a study that during the short blending process, the complete diffusion of rejuvenator into the aged binder is not possible. As a result, outer layers of bitumen were less rut resistance as compared to the inner layers of bitumen [18].

Very few studies have investigated the effect of mixing time on the properties of asphalt mixtures. A laboratory study using imaging techniques has shown that doubling the mixing time enhanced the homogeneity of the mixture and reduced the variation of air voids [19]. In line with these findings, another study has reported improvement in homogeneity of the mixture observed as a result of increasing the mixing time [20]. Another study showed using X-ray CT system analysis that the optimum mixing time for regeneration of AC mixture with rubberized asphalt should not be <90 s to achieve good distribution of components [21]. It should also be noted that longer mixing time in the laboratory may be accompanied by extra oxidation of binder and consequential stiffening of the mixture.

As seen from this summary, some studies have shown that mixing parameters could have an important impact on evaluating the performance of high RA mixtures in the laboratory. At the same time, the effect of different mixing parameters on the mixture properties is largely unknown because to best of our knowledge, no systematic study has been performed to evaluate it. Importantly, there is no common procedure for preparing mixtures containing RA in the laboratory, and thus the results from different research projects might be difficult to compare or replicate.

2. Objective

The objectives of this study are:

(a) To evaluate the effect of mixing parameters on mechanical properties of high RA content mixtures produced in the laboratory.
(b) To investigate physical and chemical changes in different layers of bitumen as a result of using different mixing processes.

3. Experimental procedure

This study evaluated the impact of laboratory mixing parameters on volumetric and mechanical properties of 60% RA content mixtures. In addition, binder evaluation was performed to examine the RA mixture bitumen layers and investigate the physical and chemical changes in binder characteristics as a result of the mixing process.

The laboratory experiments designed for this study can be divided into two stages which are illustrated in Fig. 1. In the first stage, all the compacted RA mixtures produced with different mixing parameters were analyzed using indirect tensile strength (ITS) test and stiffness modulus test. Stage two consisted of extracting four layers of binders from mixtures and further evaluating binder using the dynamic shear rheometer (DSR) and the Fourier transform infrared (FTIR) spectroscopy. For binder evaluation, the mixing time parameter was not considered in this study.

In this study, nine different mixtures were prepared as summarized in Table 1. The experimental plan was developed in such a way that two of the nine mixtures were used for evaluating more than one mixing parameter. To simplify result expression, these mixtures are reported in the results with a name that corresponds to the variable that is evaluated at that time.

It should be noted that for each group only the parameter that is being analyzed was varied, while the other parameters were kept constant to represent a "typical" mixing procedure. In this study, a "typical" procedure was defined as mixing at 155 °C for 4 min using the virgin binder blended with the rejuvenator.

3.1. Materials

Reclaimed asphalt pavement originating in Switzerland was screened on an 11 mm sieve at the RA processing facility resulting in RA 0/11 and RA 11/22 fractions. The two fractions of RA material were combined along with virgin aggregates

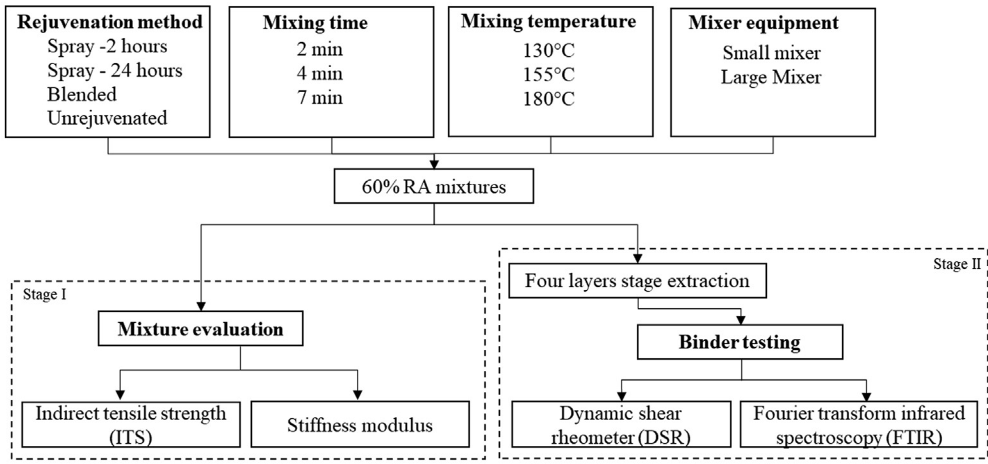

Fig. 1. Experimental plan.

Table 1
Mixture information.

Mixture ID	Rejuvenation method	Mixing temperature	Mixing time	Mixer equipment
Spray- 2 hours	Sprayed on RA material with 2 h rest period	155 °C	4 min	Small mixer
Spray- 24 hours	Sprayed on RA material with 24 h rest period	155 °C	4 min	Small mixer
Blended/Small mixer	Blended with the virgin binder	155 °C	4 min	Small mixer
Unrejuvenated	No rejuvenator added	155 °C	4 min	Small mixer
2 min	Blended with the virgin binder	155 °C	2 min	Large mixer
4 min/155 °C/Large mixer	Blended with the virgin binder	155 °C	4 min	Large mixer
7 min	Blended with the virgin binder	155 °C	7 min	Large mixer
130 °C	Blended with the virgin binder	130 °C	4 min	Large mixer
180 °C	Blended with the virgin binder	180 °C	4 min	Large mixer

to design final mixtures. RA content of 60% was used since this is typically the maximum RA content that can be added in a parallel drum plant setup. An AC-16 surface grading curve was selected as the target gradation (see Fig. 2). The virgin binder used in this study is a 70/100 penetration grade bitumen and the total binder content for each mixture was 5.5% by weight of the mixture. The recommended practice for the incorporation of recycling agents in RA mixtures is the reduction of the base binder by the full recycling-agent amount [6]. Hence for mixture design, full binder replacement was considered which means the rejuvenator quantity and available RA binder were deduced from the total binder content of the mixtures. A commercial rejuvenator based on distilled tall oil was used in the study. The rejuvenator dosage of 4.8% by weight of RA binder was selected from binder tests by targeting the virgin binder penetration grade. The properties of the RA material, virgin aggregates, and RA binder are given in Table 2.

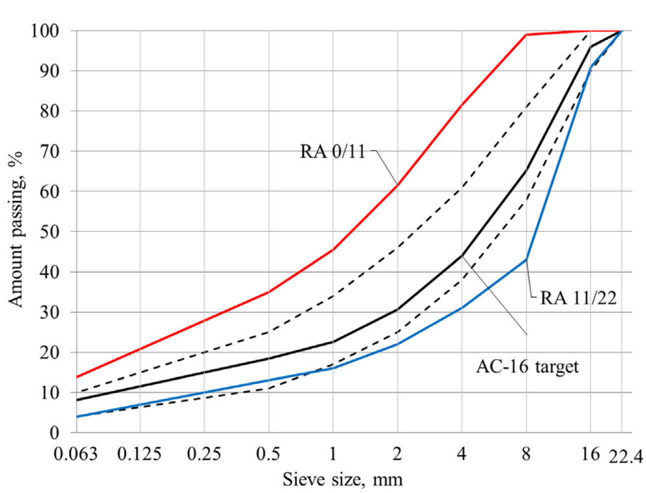

Fig. 2. Aggregate grading curves.

3.2. Laboratory mixing

The rejuvenator was either sprayed on the RA material or blended with the virgin binder, as shown in Fig. 3 (a). For spraying, the RA material was distributed evenly on a sufficiently large tray to maximize the exposed surface area. A manual pressurizing oil spray bottle was used for spraying rejuvenator on RA material which was placed on a weighing balance. First, half the quantity of rejuvenator was sprayed followed by thorough mixing, and then the rest of the rejuvenator was applied. Three different rejuvenator incorporation cases were considered as follows:

- *Spray- 2 hours*: The rejuvenator was sprayed on cold RA material and then it was put in the oven for 2 h heating, giving it a rest period of 2 h with the rejuvenator before mixing.
- *Spray-24 hours*: The rejuvenator was sprayed on cold RA material and then given a rest period of 22 h at room temperature (20 ± 5 °C). The mixture was finally heated for 2 h in the oven before mixing, giving it a total rest period of 24 h with the rejuvenator.
- *Blended*: The virgin binder was heated at a temperature of 155 °C for 2 h followed by addition of the rejuvenator and mixing them for 5 min using a high rate mixer.

All the materials were heated at their respective mixing temperature for 2 h before the mixing process. The mixtures to study the effect of different rejuvenation incorporation methods were produced in a small mixer while the mixtures considering different mixing temperature and mixing time were produced in a large mixer, as illustrated in Fig. 3 (b). The small mixer is an open mixing system where the mixing bowl is kept in the oven for heated mixing. The large mixer is a closed mixing system with a controlled heating mechanism. After mixing, the loose mixtures were stored for 24 h and then reheated for 2 h in a covered pan for compaction.

3.3. Test methods

3.3.1. Indirect tensile strength
Indirect tensile strength (ITS) was performed to evaluate the effect of the rejuvenator incorporation method on stiffness of the mixture at 5 °C. This test was conducted according to EN 12697-23 by applying a diametrical loading across the

Table 2
Material properties.

Sieve size (mm)	Passing, %					
	RA 0/11	RA 11/22	AG 11/16	AG 4/5.6	Stone dust	Filler
22.4	100	100	100	100	100	100
16	100	91	100	100	100	100
8	99	43	–	100	100	100
4	81.5	31	–	20.5	95.4	100
2	61.5	22	–	–	64.6	100
1	45.5	16	–	–	34.8	100
0.5	35	13	–	–	22.6	100
0.063	13.8	4	–	–	2.1	82
RA binder content, %	5.6	3.2				
RA binder penetration, 0.1 mm	23	34				
RA binder softening point, °C	60	65.4				

(a) (b)

Fig. 3. Laboratory mixing process (a) Spraying of rejuvenator on RA (left); Blending rejuvenator into the virgin binder (right), and (b) Small mixer (left) and Large mixer (right) used in the study.

circular cross-section of the sample at a constant speed of 50 mm/min and recording the load to failure. The test samples were compacted using the Marshall compactor according to EN 12697-30 with 50 blows on each side.

The lower mixing temperature and mixing time can lead to an insufficient coating of aggregates and allow moisture to damage the mixtures. Thus, moisture susceptibility was evaluated for these parameters according to EN 12697-12 at a test temperature of 22 °C as recommended in EN 12697-23. For moisture susceptibility evaluation, Marshall samples were prepared using 35 blows on each side. The average of three specimens was reported in the results. The ratio of the indirect tensile strength of wet specimens and dry specimens is calculated and expressed as a percentage to determine the moisture damage in the mixtures.

3.3.2. Stiffness modulus test

The stiffness modulus for all the mixtures was determined according to EN 12697–26. The load level was chosen by doing a pre-test inducing horizontal strain in the specimen in the range of 0.05%–0.10% to ensure that stiffness is in the linear viscoelastic range. This test was carried out at three temperatures, −10 °C, 10 °C and 25 °C and three frequencies, 0.1, 1, and 10 Hz. This temperature range was chosen to obtain a wide range of stiffness values and draw the mastercurves. The data obtained from testing at multiple frequencies and temperatures was shifted to a reference temperature of 20 °C by performing a temperature − time superposition principle. In this analysis, the sigmoidal model proposed by Witzack and Fonseca [22] was used and shift factors were calculated from the following relation given by Williams–Landel–Ferry [23]:

$$\log a_T = \frac{-C_1(T - T_{ref})}{C_2 + (T - T_{ref})} \quad (1)$$

where a_T is the shifting factor and C_1 and C_2 are material constants.

3.3.3. Stage bitumen extraction

Stage extraction method has been used to recover the bitumen present in different layers of asphalt mixtures and RA material. The method works on the principle that by immersing a loose asphalt sample into a solvent for a small period, a certain thickness of the bitumen layer can be dissolved. This process can be applied sequentially for a sample to extract various layers of the bitumen into the solution. Noureldin and Wood [24] used successive incremental of solvent quantity with a constant soaking time of 5 min for each layer to extract four layers of bitumen. Bower et al. [25] used incrementally increasing soaking time to extract four layers of bitumen. Other studies have kept the solvent quantity and soaking time constant for each layer [26,27]. The determination of solvent quantity and soaking time for each extraction depends primarily on the quantity of bitumen required in each layer and the number of layers required. The stage extraction method developed for the current study is illustrated in Fig. 4 and can be described as follows:

- The asphalt mixture obtained after mixing was cooled down while loosening and separating to reduce the agglomeration of particles.
- 1400 g of this mixture was taken in a mesh bucket, and four cylindrical vessels large enough to accommodate the mesh buckets were filled with 1400 ml of toluene.
- For extraction, the mesh bucket was lowered down slowly into the first vessel and kept immersed for 1 min. After 1 min, the bucket was lifted to drain the toluene under gravity and immersed into the second vessel for another 2 min. This process was repeated for the third and fourth vessels with a soaking period of 3 min and 60 min, respectively.
- The solution obtained from all the four vessels was then transferred to a centrifuge pump for removal of fines.
- Finally, the bitumen was recovered from the filtered solution through a rotary evaporator according to EN 12697-3.

The rest period for each bucket was determined by preliminary trials to ensure that approximately equal quantity of bitumen is recovered in each layer. The 60 min time for the fourth vessel was chosen to ensure removal of almost all of the traces of bitumen from the aggregates. The quantity of loose asphalt and toluene were selected to ensure complete immersion of asphalt, which is dependent on soaking vessel and mesh bucket dimensions.

3.3.4. Fourier transform infrared (FTIR) spectroscopy

FTIR test was used to investigate the changes in the chemical composition of binder due to oxidative aging as well as the effect of rejuvenator addition. This test is based on the amount of infrared radiation a bitumen sample absorbs under a given wavelength. The spectra for all the bitumen samples were recorded in reflective mode from 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans for each measurement. The raw data obtained from the test was subjected to baseline correction and normalization. For normalization, the asymmetric stretching vibration at 2920 cm^{-1} was taken as unit value, and normalized absorbance for all wavelengths was calculated as per Eq. (2).

$$A_{wn} = A_w * \frac{1}{A_{2920}} \quad (2)$$

where A_{wn} = normalized absorbance at any wavelength,

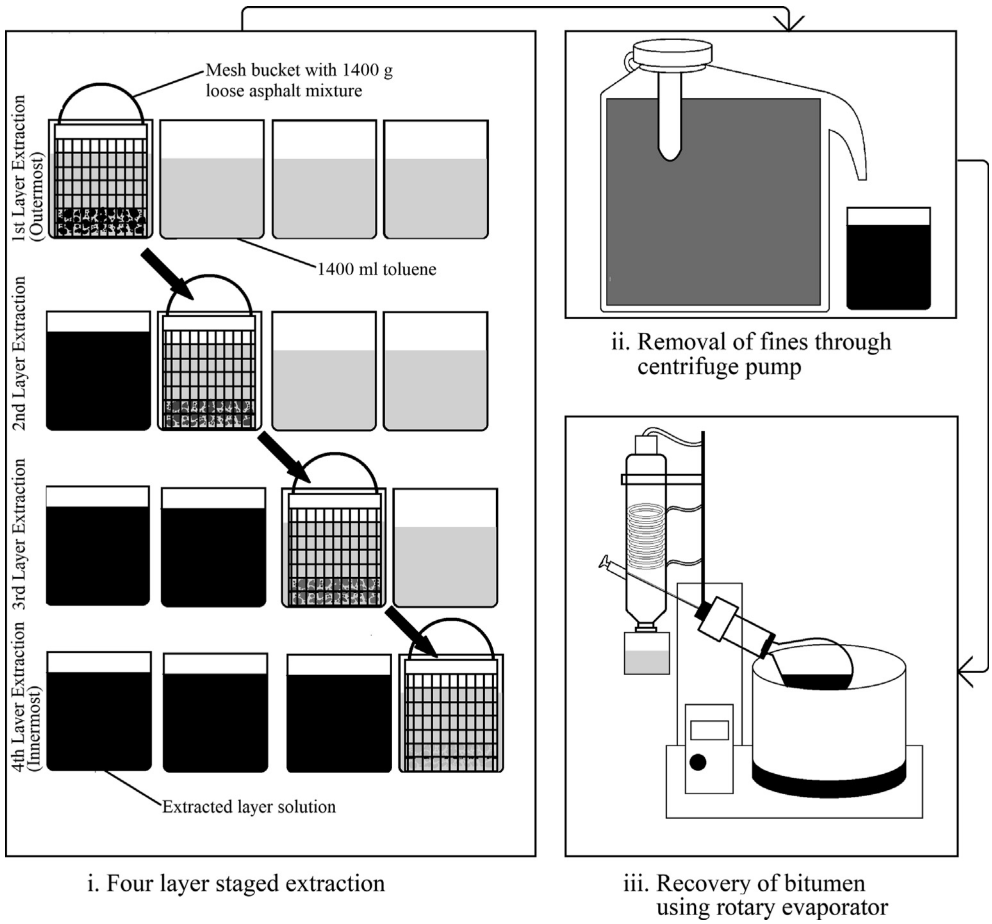

Fig. 4. Four layers stage extraction method adopted in the study.

A_w = original absorbance at any wavelength,
A_{2920} = original absorbance at 2920 cm^{-1} wavelength,

The carbonyl index has been identified in past studies to indicate the level of asphalt oxidation [28,29]. For the given sample of bitumen, the carbonyl peak was observed from 1675 cm^{-1} to 1725 cm^{-1}. From tangential integration around this region, the carbonyl index ($I_{C=O}$) was calculated as per Eq. (3).

$$I_{C=O} = \frac{\text{Area between } 1675 - 1725\ cm^{-1}}{\text{Reference area } 1220 - 1525 cm^{-1}} \quad (3)$$

The rejuvenated mixtures showed a distinct peak on FTIR spectra in the region from wavelength 1725 cm^{-1} to 1765 cm^{-1} as shown by the shaded area in Fig. 5. Therefore, the area along this region was also computed and divided by the reference group area to develop a rejuvenation index (I_R) indicating the presence of the rejuvenator in the binder, which is calculated as per Eq. (4).

$$I_R = \frac{\text{Area between } 1725 - 1765\ cm^{-1}}{\text{Reference area } 1220 - 1525\ cm^{-1}} \quad (4)$$

3.3.5. Dynamic shear rheometer test

The complex shear modulus (G*) was measured for four layers of binder using a dynamic shear rheometer as per EN 14770. The bitumen samples were conditioned at 20 °C and were subjected to a set of 22 frequencies ranging from 0.1 Hz to 20 Hz. The test was conducted in the temperature range of 20 °C–80 °C with intervals of 10 °C. A 25 mm diameter spindle with 1 mm gap width was used under strain-controlled testing mode. The obtained data were shifted to the reference temperature of 20 °C using temperature – time superposition principle to prepare mastercurves of complex shear modulus (G*). The shift factors were calculated from the same Eq. (1) as used for stiffness modulus mastercurves. A sigmoidal function as defined by Eq. (5) was used for fitting the shifted data.

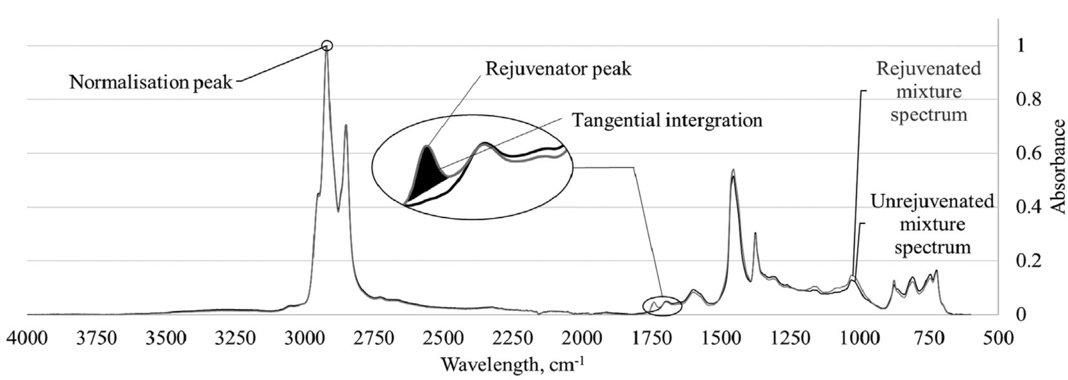

Fig. 5. Typical FTIR spectrum results for unrejuvenated and rejuvenated mixture.

$$\log G^* = \delta + \frac{\alpha}{1 + e^{\beta + \gamma(\log a_T + \log g)}} \quad (5)$$

where a_T is shifting factor, and δ, α, β, γ are fitting parameters determined using the least squares method.

3.3.6. Statistical analysis

In order to reveal the significant effect of mixing parameters on mixture properties, a one-way ANOVA was conducted for all the mixture tests along with Tukey–Kramer grouping analysis. This test uses pairwise comparisons for all the sets simultaneously to identify if the difference between two means is greater than the expected standard error. A level of significance (α) of 0.05 was used which indicates a 5% risk of concluding that a difference exists when there is no actual difference. If the mixtures share a common group letter, this means that their means are not statistically different from each other; in other words, the effect of that mixing parameter is not significant.

4. Results and discussion

4.1. Volumetrics

4.1.1. Effect of rejuvenation method on air voids

The volumetric analysis test results are summarized in Table 3. Due to the excessive fines in RA, lower air voids have been reported as a typical problem for high RA mixtures [14]. The air voids were found to be 33.1% higher in *Spray- 2 hours* and 19.9% higher in *Spray- 24 hours* mixture than the *Blended* mixture. One of the reasons for this could be that the spraying of rejuvenator may have resulted in more activation of RA material and consequently more fines are taking part in the mixing process and adhering to the coarse aggregates. Though, the Tukey-Kramer statistical groupings show no significant effect of the rejuvenation method on air voids.

4.1.2. Effect of mixing time on air voids

As seen in Table 3, the air voids slightly reduced by increasing mixing time from *2* min to *4* min. The increase in mixing time from 2 min to 4 min might have improved the homogeneity of mixture and resulted into enhanced compaction. However, a further increase in the mixing time to 7 min did not affect the air voids in the mixture. This shows that increasing the mixing time after a certain level of homogeneity is achieved, will not have any effect of compaction properties. It may be noted that the Tukey-Kramer groupings did not show any effect of mixing time on air voids of the mixture.

4.1.3. Effect of mixing temperature on air voids

Table 3 shows that an increase in mixing temperature resulted in a reduction of air voids in the mixture. At higher mixing temperature, the bitumen viscosity is reduced, and the compaction is enhanced. The Tukey-Kramer groupings for the mixing temperature showed that air voids for *180 °C* mixture were significantly different from *130 °C* and *155 °C* mixture. This shows that there is a substantial effect on compaction characteristics when mixing temperature is increased above 155 °C.

4.1.4. Effect of mixer equipment on air voids

As seen in Table 3, the air voids for the mixture produced in the *Small mixer* were significantly lower than that produced in the *Large mixer*. This was also confirmed from the Tukey-Kramer statistical groupings for the mixer equipment parameters. As all other parameters were same for these mixtures, this difference in air voids indicates that the degree of blending between RA binder and virgin binder could be significantly different for various mixing equipment. The material quantity mixed in the *Small mixer* is much less than the quantity in the *Large mixer*. Therefore, the *Small mixer* may have more homogeneously mixed the material and resulted in enhanced compactibility compared to the *Large mixer*.

4.2. Indirect tensile strength (ITS) test

4.2.1. Effect of rejuvenation method on ITS

The results of the indirect tensile strength test for different rejuvenation methods are shown in Fig. 6. From Table 4, it is confirmed that none of the rejuvenation methods had any significant effect on the indirect tensile strength of the mixtures. Although the *Unrejuvenated* mixture showed the highest ITS value, it was not statistically different from any of the mixtures containing rejuvenator. It may be noted from Fig. 6, that *Spray- 2 hours* and *Spray- 24 hours* showed slightly higher fracture energy compared to the other two mixtures mixture. The higher fracture energy for *Spray- 2 hours* and *Spray- 24 hours* mixtures could be related to higher activation of aged RA binder when the rejuvenator was sprayed directly over RA material as opposed to blending the rejuvenator into the virgin binder. However, this effect was not statistically visible from Tukey-Kramer groupings as shown in Table 4.

4.2.2. Effect of mixing time on ITS

The results of the indirect tensile strength test for different mixing time are shown in Fig. 7. It can be observed from Fig. 7, that the mixing time did not seem to have any effect on the indirect tensile strength for dry as well as wet conditioned specimens. For this reason, all the mixtures share the same group in statistical analysis for dry indirect tensile strength, as shown in Table 5. The high TSR ratios for all the mixtures indicate very low moisture damage in the mixtures. This is a common observation for high RA mixtures,

Table 3
Volumetric properties of mixtures.

Mixtures	Air voids, %	VMA, %	VFB, %	Tukey-Kramer group
Rejuvenation methods				
Spray- 2 hours	2.7	16.0	83.5	A
Spray- 24 hours	2.4	15.7	84.8	A
Blended	2.0	15.3	87.0	A
Unrejuvenated	1.8	15.1	88.0	A
Mixing time				
2 min	3.3	16.4	80.1	A
4 min	2.8	16.1	82.7	A
7 min	2.9	16.1	81.9	A
Mixing temperature				
130 °C	3.1	16.3	80.9	A
155 °C	2.8	16.1	82.7	A
180 °C	2.1	15.4	86.6	B
Mixer equipment				
Small mixer	2.0	15.3	87.0	A
Large mixer	2.8	16.1	82.7	B

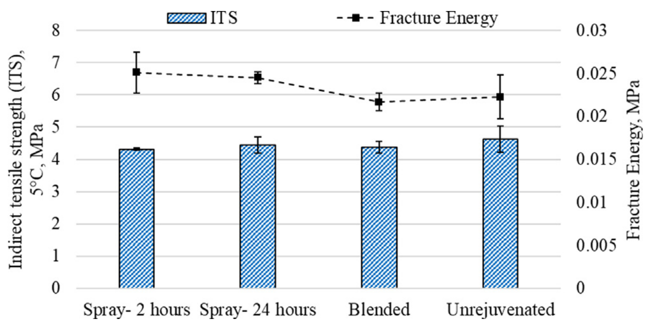

Fig. 6. ITS results for different rejuvenation techniques. The error bars indicate one standard deviation.

and one reason is that lower air voids do not allow moisture to damage the aggregates [10]. Additionally, the double coating of bitumen on RA aggregates supports the moisture resistance.

The fracture energy for all the mixtures was found to be significantly different from each other as none of the mixtures share the same group (see Table 5). This difference could be due to area calculation errors or test variability. The fracture energy shows large deviations for *2* min mixture which could be related to the non-homogeneity of the mixture. This observation is supported by other studies that suggested to avoid very low mixing time in the laboratory as it could lead to increased non-homogeneity of mixture [19,20].

4.2.3. Effect of mixing temperature on ITS

The results of the indirect tensile strength test at 22 °C for different mixing temperatures are shown in Fig. 8. It can be observed that the mixture produced at 180 °C resulted in a higher ITS value compared to other mixtures, and the ITS values for *130 °C* and *155 °C* mixtures were not much different. The fracture energy and toughness for the mixture produced at 180 °C were also significantly higher compared to other mixtures as observed from statistical grouping given in Table 6. This shows that the mixture stiffness increases substantially as a result of excessive oxidation when the temperature is raised above a certain threshold. This demonstrates that the mixing and heating temperature need to be carefully controlled when preparing mixtures in the laboratory. These results also show that the typical mixing temperature in the range of 155 °C considered in asphalt plant is justified.

4.3. Stiffness modulus of asphalt mixture

This test was conducted to evaluate the change in stiffness of the mixture by varying the mixing parameters. The results of the stiffness modulus test were used to construct mastercurves for all the mixtures, which can be seen in Fig. 9. For statistical evaluation of these results, the stiffness was divided into three zones, low stiffness zone (25 °C + 0.1 Hz), intermediate stiffness zone (10 °C + 1 Hz), and high stiffness zone (−10 °C + 10 Hz) as shown in Table 7.

4.3.1. Effect of rejuvenation method on stiffness modulus

It is clear from Fig. 9 (a) that all the three mixtures containing rejuvenator were less stiff as compared to the *Unrejuvenated* mixture. This was also confirmed from statistical grouping for low and intermediate stiffness zones as given in Table 7. The rejuvenator had a softening effect on the binder and reduced the overall stiffness of the mixture. However, none of the rejuvenation methods was statistically different from each other.

4.3.2. Effect of mixing time on stiffness modulus

Fig. 9 (b) shows that the increase in mixing time did not have any effect on the stiffness modulus of the mixtures. This was also confirmed from the Tukey-Kramer groupings shown in Table 7. These results are in agreement with the indirect tensile strength test where mixing time did not have any impact on mixture properties. Therefore, it is confirmed that additional oxidative aging that may occur by increasing mixing time up to 7 min is not enough to have a significant effect on the overall stiffness of the mixtures.

4.3.3. Effect of mixing temperature on stiffness modulus

Fig. 9 (c) shows the mastercurves for mixtures at different mixing temperatures. In the low stiffness zone, 130 °C and 155 °C mixtures were statistically similar, which is in agreement with ITS test results (see Table 7). However, at the intermediate stiffness zone, all the mixtures were statistically different, and the increase in temperature has resulted in a higher stiffness indicating increased oxidative aging. The effect of mixing temperature was not statistically significant in the high stiffness zone.

4.3.4. Effect of mixer equipment on stiffness modulus

The effect of mixer equipment on the stiffness modulus of mixtures is shown in Fig. 9 (d). It shows that the stiffness modulus mastercurve for the small mixer was slightly above the large mixer. This could also be observed statistically from Table 7 at intermediate stiffness grouping. It can be seen in Fig. 3 that the large mixer produces asphalt in a closed system while the small mixer is an open system with an abundant supply of oxygen and therefore this result could be explained by excessive oxidation of bitumen in the small mixer. Since the difference was not substantial for all the stiffness zones, it cannot be confirmed if this is the effect of the mixer equipment or inconsistency in test results.

4.4. Complex modulus of binder

4.4.1. Effect of rejuvenation method on complex modulus mastercurves

The rheological mastercurves obtained for four layers of bitumen for mixtures with different rejuvenation methods are shown in Fig. 10. It can be observed from Fig. 10 (a), (b), and (c) that complex modulus mastercurves for four layers of bitumen overlap for all the three rejuvenation methods. This indicates that there were no significant differences in stiffness among the four layers of bitumen for all the rejuvenation cases. A phenomenon defined by various researchers as the “black rock effect”, where the RA material acts as a black rock and the RA bitumen either does not at all or

Table 4
Tukey–Kramer Statistical Groupings (α = 0.05) for the effect of rejuvenation method on ITS value.

Mixture	Indirect tensile strength, 5 °C, MPa			Fracture energy, MPa			Post cracking energy, MPa			Toughness, MPa		
	N	Mean	Group	N	Mean	Group	N	Mean	Group	N	Mean	Group
Spray-2 hours	3	4.314	A	3	0.0251	A	3	0.0290	A	3	0.0513	A
Spray- 24 hours	3	4.446	A	3	0.0245	A	3	0.0306	A	3	0.0551	A
Blended	3	4.373	A	3	0.0217	A	3	0.0335	A	3	0.0552	A
Unrejuvenated	3	4.619	A	3	0.0223	A	3	0.0369	A	3	0.0620	A

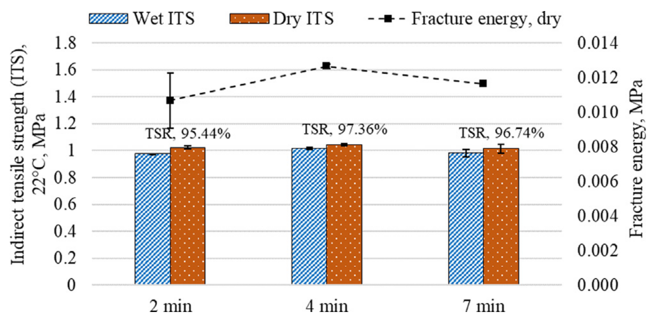

Fig. 7. ITS results for different mixing times. The error bars indicate one standard deviation.

partially blends with the virgin bitumen. Clearly, the effect of black rock aggregates was not observed in this case, as inner layers of bitumen showed equivalent stiffness to that of outer layers.

On the other hand, the *Unrejuvenated* mixture, as observed in Fig. 10 (d), shows non-homogeneous stiffness between the four layers. The absence of rejuvenator resulted in inadequate blending between the RA binder and the virgin binder in this case.

The mean complex modulus of four layers was calculated to see the overall effect of the rejuvenation method, as shown in Fig. 10 (e). The spraying of rejuvenator has resulted in lower overall stiffness of the *Spray- 2 hours* and *Spray- 24 hours* mixtures as compared to the *Blended* mixture. This may indicate that the softening effect of rejuvenator was higher when the rejuvenator was sprayed.

4.4.2. Effect of mixing temperature on complex modulus mastercurves

The rheological mastercurves obtained for four layers of bitumen with different mixing temperatures are illustrated in Fig. 11. It can be observed from Fig. 11 (a) and (b), that the third layer of bitumen showed higher stiffness compared to other layers of bitumen. The higher stiffness of the third layer in *130 °C* and *155 °C* mixtures may be an indication of the black rock effect where the stiff unactivated RA binder did not take part in the mixing process. The reason for the lower stiffness of the fourth layer could be explained by one of the drawbacks of the adopted stage extraction method. It is hypothesized that some virgin bitumen-fine aggregates clusters remain bonded during the short period of the first three extractions and were dispersed during the 60 min soaking period in the fourth extraction. As a result, a blend of RA binder and virgin bitumen was obtained in the fourth layer.

From Fig. 11 (c), the complex modulus mastercurves for bitumen extracted from mixtures produced at 180 °C show that the third and the fourth layers were stiffer than the first and the second layers. One of the possible reasons for this could be that higher heating temperatures of the RA aggregates stiffened the RA binder and resulted in a lower degree of blending between virgin and RA binder.

When the average value of the complex modulus of four layers of bitumen for all the three mixing temperatures was plotted as illustrated in Fig. 11 (d), an increment in mixing temperature shifted the mastercurve upwards, indicating increased overall aging of the bitumen which also agrees with the mixture test results.

4.4.3. Effect of mixer equipment on complex modulus mastercurves

Mixers with internal heating are normally considered superior compared to small unheated mixers. It can be seen from Fig. 12 (a) that the *Small mixer* resulted in homogenous stiffness throughout the four binder layers, while as seen in Fig. 12 (b) the layers from the *Large mixer* have a much higher stiffness range. This may indicate an incomplete blending of virgin and RA bitumen (black rock effect). It is concluded that mixer equipment may affect the degree of blending between the RA binder and the virgin binder even when other parameters are kept the same.

4.5. FTIR characterisation

4.5.1. Effect of rejuvenation method on FTIR indices

The FTIR characterisation results for different rejuvenation cases are shown in Fig. 13. It can be observed from Fig. 13 (a), that all the rejuvenated mixtures show lower carbonyl indices compared to the *Unrejuvenated* mixture except for the innermost layer. This could be due to chemical changes in the binder that occurred as a result of rejuvenator addition. The inner layers are expected to contain more oxidised binder compared to outer layers. Contrary to this, the carbonyl index is reducing for all the mixtures moving from the first (outermost) layer to the fourth (innermost) layer except in the *Spray- 2 hours* mixture, where the differences were negligible amongst the four layers. These unexpected results may be due to the presence of two different sources of binder that may show different intensities around the carbonyl peak.

A rejuvenation index (I_R) was developed in this study to indicate the presence of the rejuvenator in binder layers. It is calculated by dividing the tangential area around the distinct rejuvenator peak with the reference area. Fig. 13 (b) shows the rejuvenation index calculated for different rejuvenation methods. The presence of the rejuvenator was detected in all the layers (indicated by I_R values) of different mixtures except for the *Unrejuvenated* mixture (where $I_R \approx 0$). For the outermost layer, the rejuvenation index was the same for all the rejuvenated mixtures, but the difference was increasingly visible moving towards the inner layers. The higher difference in innermost layer I_R value among different rejuvenated mixtures may indicate that the diffusion of rejuvenator could be different for each rejuvenation method.

4.5.2. Effect of mixing temperature on FTIR indices

Fig. 14 shows the indices for four layers of stage extracted bitumen for different mixing temperatures. Since the bitumen tends to age more at a higher temperature, the carbonyl index for mixtures produced at high temperatures were expected to be higher. Unexpectedly as seen in Fig. 14 (a), except the outermost layer, the carbonyl index for *180 °C* mixture, was found to be lower compared to *130 °C* and *155 °C* mixtures. Carbonyl peak is an indicator of relative oxidation and not the absolute oxidation of bitumen [30].

Table 5
Tukey–Kramer Statistical Groupings (α = 0.05) for the effect of mixing time on ITS value.

Mixture	Indirect tensile strength, 22 °C, dry, MPa			Fracture energy, dry, MPa			Post cracking energy, dry, MPa			Toughness, dry, MPa		
	N	Mean	Group	N	Mean	Group	N	Mean	Group	N	Mean	Group
2 min	3	1.022	A	3	0.0107	A	3	0.0139	A	3	0.0245	A
4 min	3	1.044	A	3	0.0127	B	3	0.0164	A	3	0.0290	B
7 min	3	1.015	A	3	0.0116	C	3	0.0147	A	3	0.0263	A/B

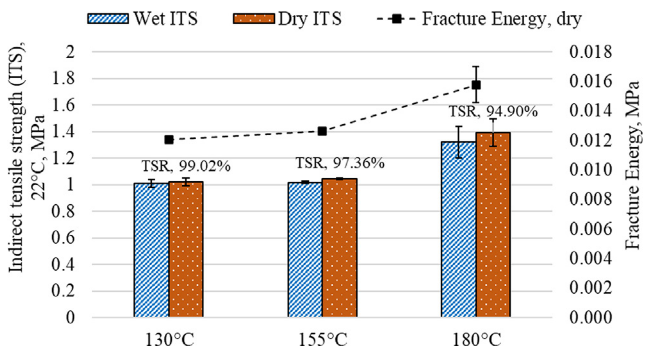

Fig. 8. ITS results for different mixing temperature. The error bars indicate one standard deviation.

The presence of two different binders may be one of the reasons that carbonyl index was not able to show increased oxidation as a result of increased mixing temperature. Another reason may be due to presence of rejuvenator. The functional groups including aldehydes, acid, anhydride, amides, and esters, overlap in the carbonyl band with binder oxidation products, which are common in bio-oils used as asphalt modifiers [29]. The rejuvenator presence may have complicated the chemical analysis and hence the changes in carbonyl area as a result of mixing process could not be observed in this analysis. In the future, it is recommended to analyze the FTIR spectra of binder without adding rejuvenator to have a more accurate assessment of the effects of mixing parameters.

There was no significant difference in carbonyl indices amongst the four layers of bitumen for *130 °C* (p_{value} = 0.465) and *155 °C* (p_{value} = 0.846) mixture. For the *180 °C* mixture, the carbonyl index among four layers was found to be significantly different (p_{value} < 0.05). A similar trend was observed for the rejuvenation index as reported in Fig. 14 (b), as rejuvenation index amongst four layers were statistically same for *130 °C* (p_{value} = 0.462) and *155 °C* (p_{value} = 0.497) and were different for *180 °C* mixture (p_{value} < 0.05). It can only be inferred that as the mixing temperature was increased, the difference between indices of the four layers was increased. This may indicate that at higher mixing temperature, the binder may get excessively oxidized and reduce the degree of blending in the mixtures.

4.5.2.1. Effect of mixer equipment on FTIR indices. A comparison between the carbonyl and rejuvenation indices for different mixer equipment can be seen in Fig. 15. It can be observed from Fig. 15 (a) that carbonyl indices for *Small mixer* were significantly higher compared to *Large mixer*. The higher index may be an indication of excess oxidative aging in *Small mixer* due to the open mixing system. This was in agreement with complex modulus bitumen results where *Small mixer* lead to higher stiffness of bitumen layers compared to *Large mixer*. As seen in Fig. 15 (b), the rejuvenation indices for the *Small mixer* was slightly higher compared to the Large mixers for all the layers except the innermost layer. Although the rejuvenation index was able to indicate the presence of rejuvenator in the bitumen, the earlier described complications of this analysis indicate that the rejuvenation index may not necessarily correspond to the quantity of rejuvenator present in the bitumen.

5. Conclusions

Laboratory mixing parameters can play an important role in evaluating the performance of high RA mixtures in the laboratory. This study considered 60% RA mixtures produced using a tall-oil based rejuvenator to evaluate the effect of laboratory mixing parameters, including rejuvenator application method, mixing temperature, mixing time, and mixer type. The mixtures properties were evaluated using the indirect tensile strength (ITS) test and the stiffness modulus test. A staged binder extraction method was used to study the properties of bitumen layers in RA mixtures and change in binder characteristics as a result of the mixing process. Binder evaluation was performed using Fourier transform infrared (FTIR) spectroscopy device and dynamic shear rheometer (DSR). Based on our analysis, the following conclusions can be made:

(1) The addition of rejuvenator reduced the stiffness of the mixtures and resulted in more homogeneous properties in bitumen layers. At the same time, none of the rejuvenation methods showed a significant impact on the mixture or binder test results.
(2) Mixing and heating temperature are critical parameters for high RA mixtures produced in the laboratory. Mixing at high temperature clearly increased the stiffness of the mixture and the binder layers. It is therefore important to carefully control the mixing and heating temperature of materials for evaluating high RA mixture performance in the laboratory. The simulation of the temperature used in the asphalt plant seems to be warranted.
(3) The effect of the mixing time was not observed on indirect tensile strength and stiffness modulus of mixtures.
(4) The rheological characterisation of different layers of bitumen shows that the black rock effect was partially observed in mixtures from the large mixer equipment, but not at all visible in the small mixer, which indicates that the degree of blending between the RA binder and the virgin binder may change with mixer equipment.
(5) The carbonyl and rejuvenation index calculated from FTIR spectra were not consistent with the rheological properties obtained from the dynamic shear rheometer nor with the mixture results. Therefore, in this study, these indices were found unsuitable to predict the chemical changes in the binder as a result of changing different mixing parameters.

In summary, based on the results of this study, there was no difference found between any of the rejuvenation methods adopted in this study. However, other studies provide evidence that early addition of rejuvenator facilitates the activation of the RA binder [13,31] therefore early addition on RA (instead of addition in bin-

Table 6
Tukey–Kramer Statistical Groupings (α = 0.05) for the effect of mixing temperature on ITS value.

Mixture	Indirect tensile strength, 22 °C, dry, MPa			Fracture energy, dry, MPa			Post cracking energy, dry, MPa			Toughness, dry, MPa		
	N	Mean	Group	N	Mean	Group	N	Mean	Group	N	Mean	Group
130 °C	3	1.020	A	3	0.0121	A	3	0.0175	A	3	0.030	A
155 °C	3	1.044	A	3	0.0127	A	3	0.0164	A	3	0.029	A
180 °C	3	1.392	B	3	0.0158	B	3	0.0184	A	3	0.034	B

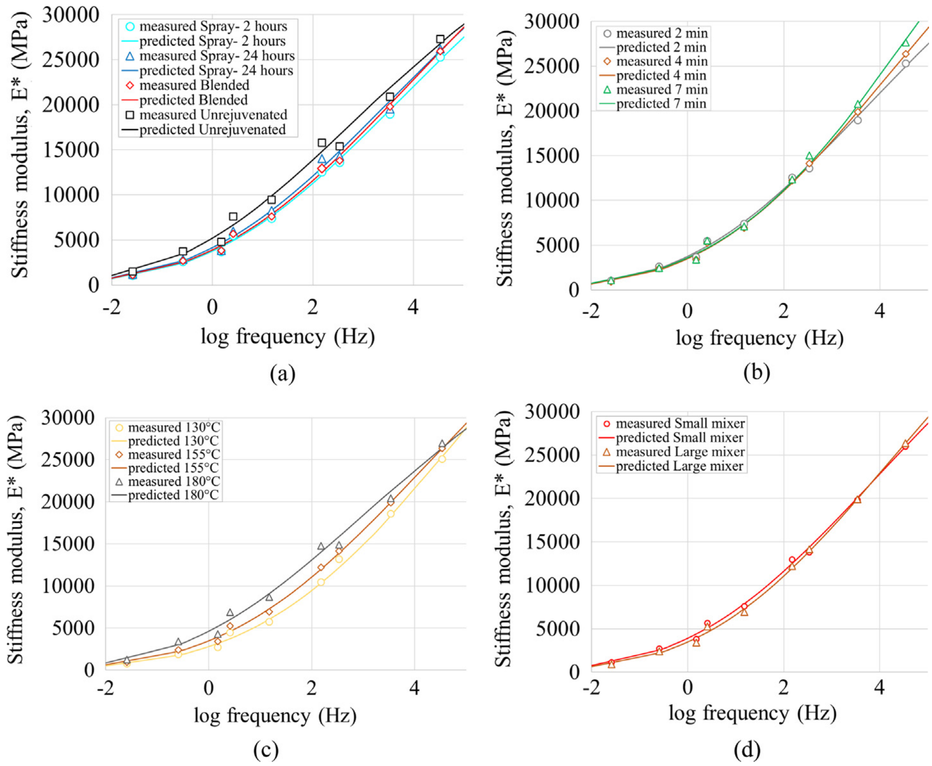

Fig. 9. Stiffness modulus mastercurves for effect of (a) rejuvenation method; (b) mixing time; (c) mixing temperature; (d) mixing equipment.

Table 7
Tukey–Kramer statistical groupings (α = 0.05) for stiffness modulus test.

	Low stiffness (25 °C, 0.1 Hz)			Intermediate stiffness (10 °C, 1 Hz)			High stiffness (−10 °C, 10 Hz)		
	N	Mean	Group	N	Mean	Group	N	Mean	Group
Rejuvenation method									
Spray-2 hours	3	1104.7	A	3	7336	A	3	25,788	A
Spray- 24 hours	3	1185.5	A	3	8285	A	3	26,191	A
Blended	3	1117.8	A	3	7604	A	3	25,958	A
Unrejuvenated	3	1519.6	B	3	9460	B	3	27,290	A
Mixing time									
2 min	4	918.1	A	4	7483	A	4	25,077	A
4 min	4	1045.0	A	4	7003	A	4	26,357	A
7 min	4	1074.2	A	4	7281	A	4	26,988	A
Mixing temperature									
130 °C	4	803.8	A	4	5937	A	4	25,077	A
155 °C	4	918.1	A	4	7003	B	4	26,357	A
180 °C	4	1282.6	B	4	8671	C	4	26,988	A
Mixer type									
Small mixer	4	1117.8	A	4	7604	A	4	25,958	A
Large mixer	4	932	A	4	7116.5	B	4	26,294	A

der) is recommended. The mixing and heating temperature of high RA mixtures should not exceed 155 °C to avoid excessive aging of RA bitumen. A mixing time of 4 min is recommended in the laboratory. The laboratory studies should account for the type of mixer equipment used for preparing the mixtures since different mixers could lead to a different degree of blending for the same material.

The above recommendations for the laboratory mixing procedure are based on the indirect tensile strength test and the stiffness modulus test. Hence, in the future, the effect of mixing procedure on performance parameters such as rutting, fatigue, and low temperature cracking may be evaluated. Moreover, the parameters considered in this study were limited to laboratory mixing, and these results do not extend to mixtures produced in a plant. Ideally, the plant conditions should be simulated when mixing in the laboratory. For this reason, future studies may consider simulating the plant mixing process to investigate the effects of mixing parameters on high RA mixtures.

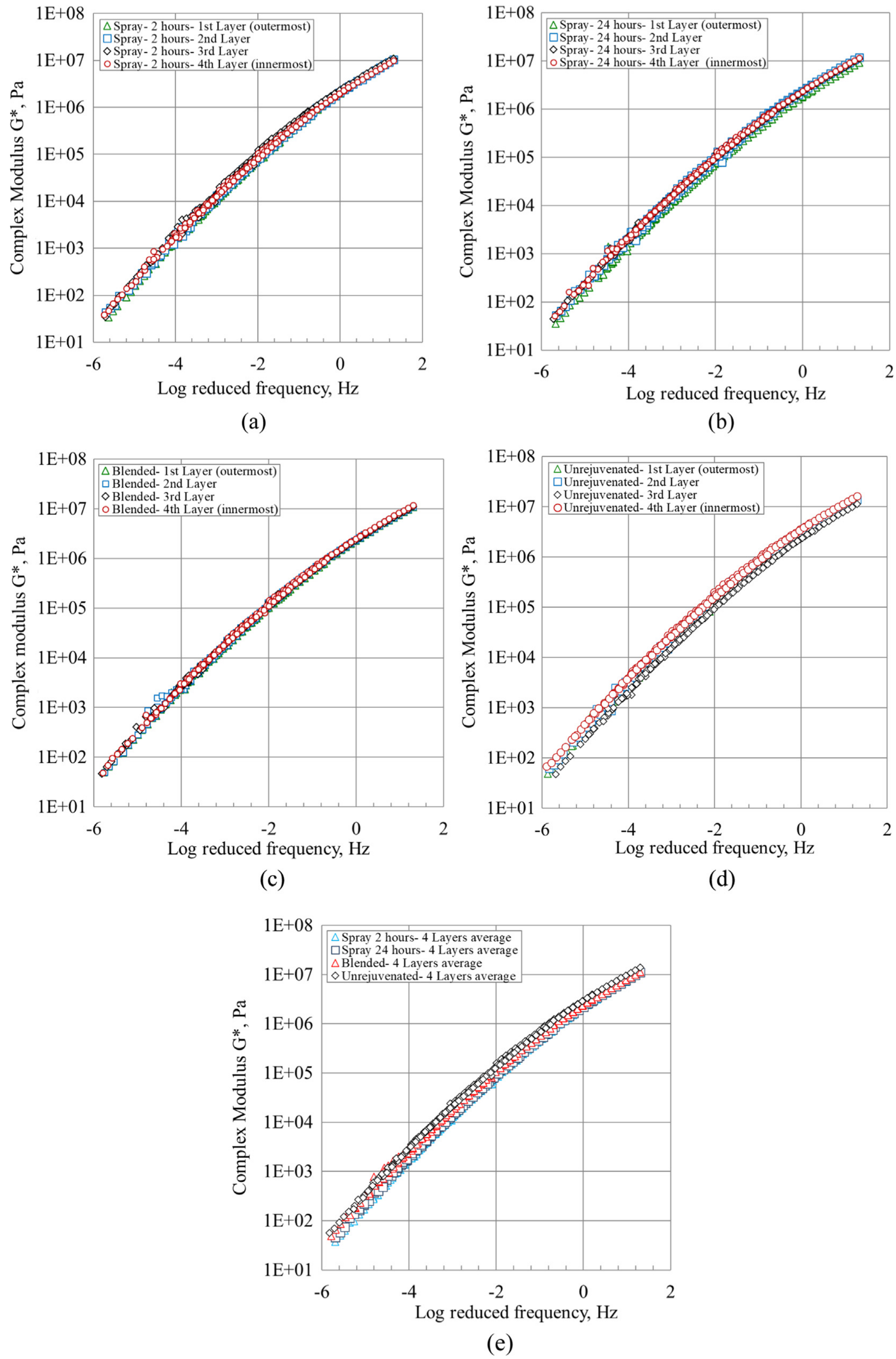

Fig. 10. Complex modulus mastercurves of stage extracted bitumen for (a) Spray- 2 h; (b) Spray- 24 h; (c) Blended; (d) Unrejuvenated; (e) average complex modulus.

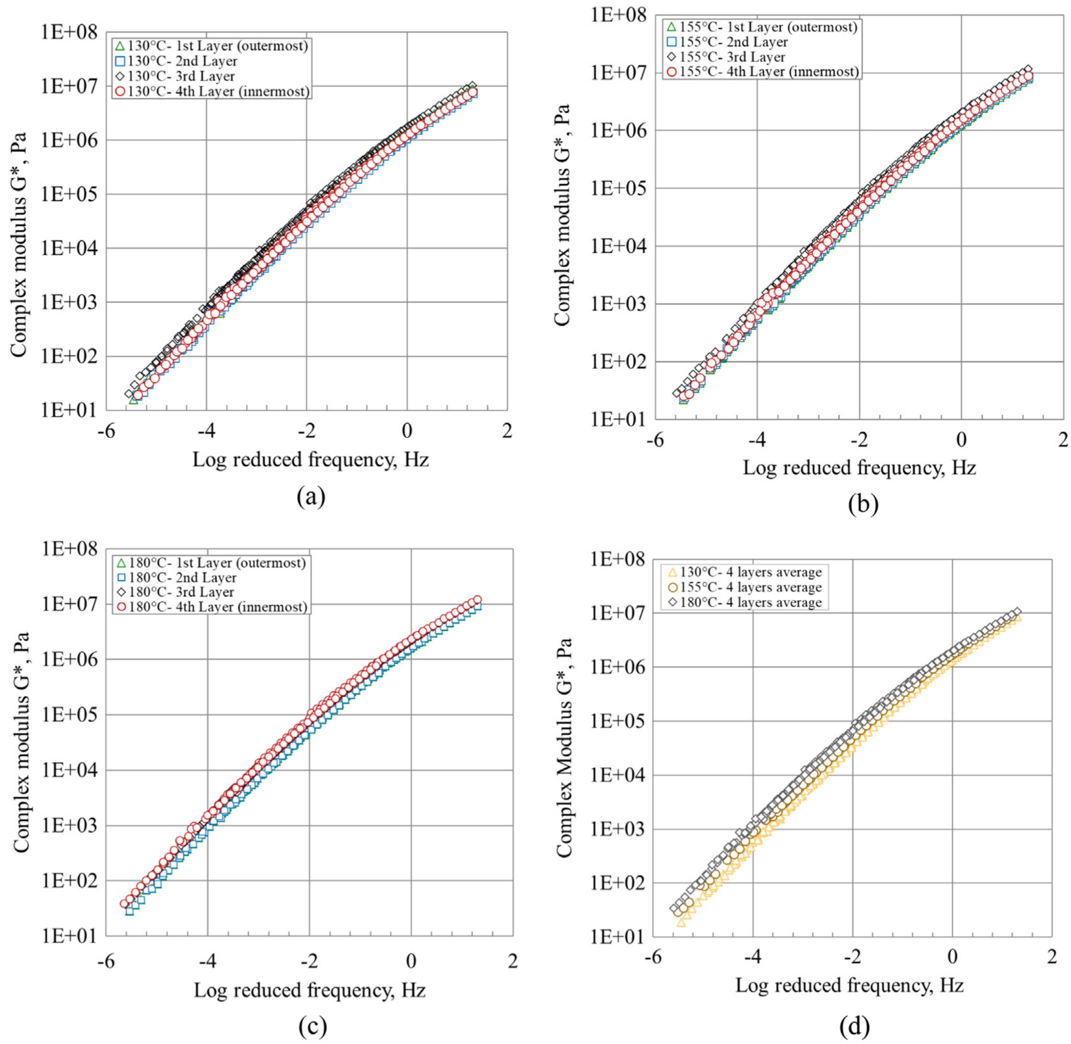

Fig. 11. Complex modulus mastercurves of stage extracted bitumen for (a) 130 °C; (b) 155 °C; (c) 180 °C; (d) average complex modulus.

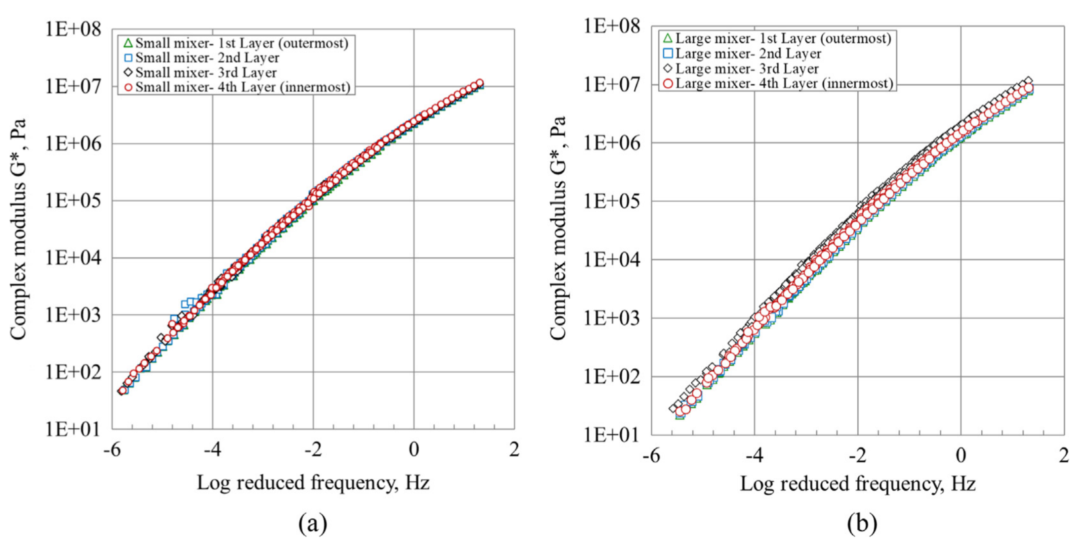

Fig. 12. Complex modulus mastercurves of stage extracted bitumen for (a) Small mixer; (b) Large mixer.

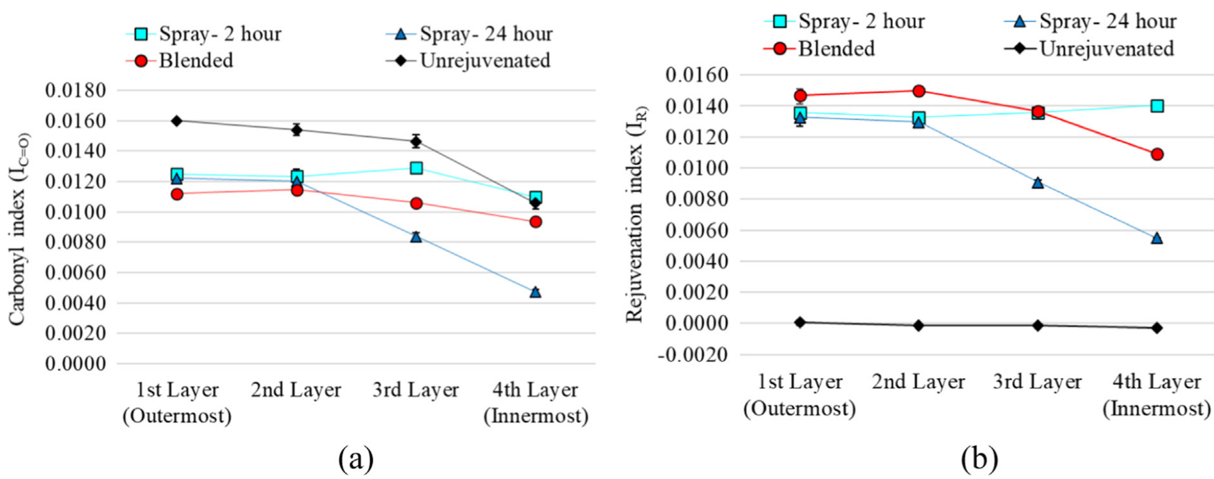

Fig. 13. Indices calculated from FTIR spectra analysis for different rejuvenation cases (a) carbonyl indices; (b) rejuvenation indices.

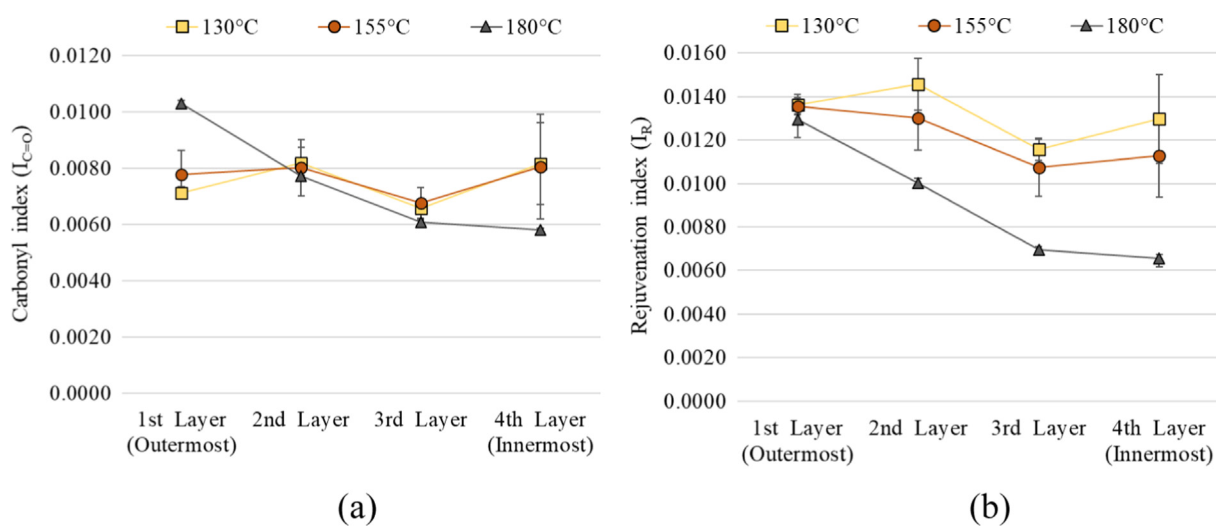

Fig. 14. Indices calculated from FTIR spectra analysis for different mixing temperatures (a) carbonyl indices; (b) rejuvenation indices.

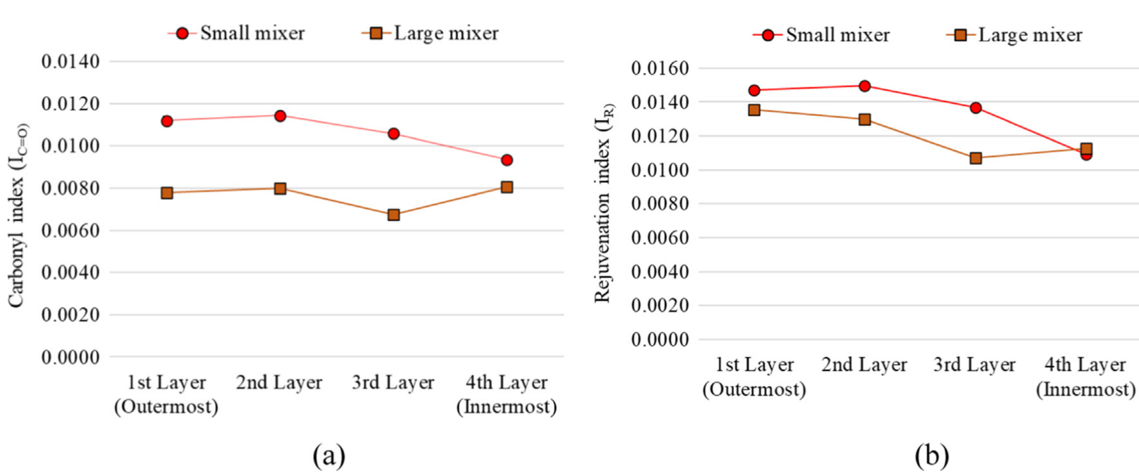

Fig. 15. Indices calculated from FTIR spectra analysis for different mixer equipment (a) carbonyl indices; (b) rejuvenation indices.

CRediT authorship contribution statement

Mukul Rathore: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing - original draft, Visualization. **Martins Zaumanis:** Conceptualization, Methodology, Resources, Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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