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ORIGINAL RESEARCH PAPER

Chemo-rheological Investigation on Waste Rubber-Modified Bitumen Response to Various Blending Factors

Muhammad Jamal[1] · **Filippo Giustozzi**[1]

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Abstract

In a continuous effort to promote the environmental sustainability of the road construction sector while increasing the durability of road pavements, a growing number of studies have focused on recycled materials to be used as polymer modification for asphalt. Australia is also following the 'sustainability' trend by reusing end-of-life tyres (EOLTs) in the form of crumb rubber (CR) for road applications. Blending conditions are influential in determining the final properties of waste rubber-modified bitumen and are explored in this study to promote the recycling of EOLTs further. Two shear mixing rates (700 rpm and 3500 rpm) and three mixing durations (30, 60 and 90 min) were selected at 7.5% (low concentration), 15% (medium concentration), and 22.5% (high concentration) of CR. Blends were prepared by mixing CR in C320 bitumen while keeping the temperature constant at 177 ± 10 °C. Physio-chemical, thermal, and rheological tests were conducted to characterize the optimal blending conditions. Although the current CR bitumen specifications are predominantly based on viscosity and bitumen–rubber interaction time, the present study highlights the impact of blending conditions on blends to be adopted for different purposes (i.e., 7.5% low-content CR for local roads with less traffic vs. 22.5% CR for medium–high-trafficked roads). It has been observed that mixing duration and applied shear rate significantly influence the rheological properties and are closely correlated. A mixing duration of 60 min is effective to swell rubber particles, although further increasing the mixing time breaks the polymeric chains and deteriorates the rheological properties. Increasing the shear mixer speed to 3500 rpm reduces the total time required to fully swell the CR particles by approx. 30 min. The shear rate can produce a relatively stiffer blend at higher mixing speeds, which was observed through the increase in the complex shear modulus and fatigue parameter values; this was further assessed through Fourier Transform InfraRed analysis and aging indices.

Keywords Recycling · Waste rubber · Asphalt · Bitumen · Multiple Stress Creep Recovery · Non-Newtonian fluids

1 Introduction

'Asphalt' the magazine of the Asphalt Institute mentioned that 'Asphalt is the pavement of choice in Australia' [1]. The Australian road network stretches for approximately 877,651 kms and is mostly paved with asphalt [2]; rutting at high temperature and fatigue cracking at intermediate temperature are considered the main culprits for pavement distresses, as low-temperature cracking is generally not a concern. Asphalt pavements constructed using conventional bitumen are usually prone to these types of distresses, however, it was observed that the addition of polymers can substantially improve the overall pavement durability [3–8]. Although Styrene Butadiene Styrene (SBS) copolymer has largely proven to be an effective bitumen modifier, the recent push for sustainability has attracted many state and local road authorities to adopt recycled polymer technologies on their roads [9–13]. In this sense, the use of CR from shredded tyres is an interesting alternative from both economic and environmental perspectives [14–16]. In Australia, due to the recent bans on the export of whole used tyres to begin in December 2021, all the EOLTs must be reprocessed locally [17, 18]. In 2018–2019, almost 70% of the 466,000 tons of EOLTs generated in Australia were recycled into tyre-derived products or thermal processing. Unfortunately, approx. 140,000 tons of tyres were landfilled, buried on-site or illegally dumped [19]. The regulation about EOLTs in roads generally lacks proper specifications and guidelines,

✉ Filippo Giustozzi
filippo.giustozzi@rmit.edu.au

1 Civil and Infrastructure Engineering, RMIT University, Melbourne, VIC 3000, Australia

as also emphasized by the 'National market development strategy for used tyres 2017–2022' [17].

CR can be added into the asphalt mixture either directly as coarser particle (dry process) or by first interacting with bitumen in the form of fine powder (wet process) to produce CR-modified bitumen (CRMB). The final characteristics of CRMB depend on the extent of the interaction between CR and bitumen with the interaction being more significant for the wet process, compared to the dry process [20]. However, this complex process depends on several material properties and processing conditions. Whenever bitumen and rubber interacts, two types of mechanisms are observed; swelling and chemical degradation [21]. Generally, unvulcanized rubber will dissolve, while vulcanized rubber swells quickly in a compatible liquid [22]; in case of bitumen, the solubility of the maltenic fraction is similar to that of the rubber, which makes them compatible [23]. However, different rubber sources may show significant variability of their chemical footprint and the compatibility with bitumen may vary. If the material properties are constant, then the processing conditions such as mixing device, mixing temperature, mixing time, shear mixer speed and CR dosage play a significant role in defining CRMB properties.

Navarro et al. [24–26] evaluated the effect of different mixing devices (four-blade impeller, helical and anchor stirrer and a pilot plant with rotor and stator setup) and mixing temperatures on the rheology of rubberized bitumen; the study showed that the rheological behavior of CRMB is not influenced by the specific processing device and impeller geometry used for blending bitumen and CR at 180 °C. However, depolymerization of CR particles starts at approx. 180 °C, though smaller in magnitude. Similar conclusions were conveyed by Jamrah et al. [27] while studying the effect of temperature on bitumen–CR interaction. Aflaki and Memarzadeh [28] studied the influence of shear rate on the intermediate and low temperature properties and reported that a higher shear rate enhances the low temperature properties as compared to lower shear rates. However, the reason behind this improvement was not fully provided in their study. Billiter et al. [29] showed an increase in CR dissolution with increased mixing speed, time and temperature. Li et al. [30] recommended a blending temperature of 180 °C, mixing duration of 40 min and a shear rate of 7000 rpm for proper interaction of CR and bitumen. Such a high mixing rate ultimately disintegrates the CR particles, hence a reduction in CR particle size enhances the CR–bitumen interaction. However, Kok et al. [31] reported that blends prepared at higher mixing speed are susceptible to oxidation and aging can be induced during the blending process. Therefore, many studies on CRMB were found using a rather low shear rate of approx. 700 rpm [32–34]. It should be noted that for the manufacturing of CR terminal blends, very high shear rates are found in a number of studies [35, 36]. Mashaan et al. [34] investigated the influence of mixing duration and CR quantity by adding CR up to 20% at 180 °C with a blending rate of 200 rpm mixed for 30 and 60 min. Ductility, elastic recovery, and rutting rheological parameter ($G^*/\sin\delta$) were evaluated and the significance of each factor was analyzed using ANOVA method. Statistical analysis showed that CR content significantly influences CRMB properties while mixing duration had minimal influence. Thives et al. [37] used SEM to assess the digestion time of CRMB prepared from two CR sources (cryogenic and ambient grinding). CR was mixed with soft (Pen 50) and hard (Pen 30) bitumen for 30, 45, 60 and 90 min at 180 °C. A mixing duration of 60 min was found to suffice for the digestion of ambient grinded CR particles, while 90 min was optimal for cryogenic grinded CR instead.

As it can be inferred, the influence of temperature and mixing processes/devices is greatly controversial; in addition, very few studies are available that include at the same time a broader range of mixer speeds (including both high and low speed), blending duration and various CR concentrations. In this study, various mixing speeds (i.e., up to 3500 rpm) and CR concentrations (up to 22.5%) were evaluated to assess how the variation of the single parameters involved in the blending protocol affects the physiochemical, thermal and rheological properties of CRMB. The broadening of the blending parameters also allowed to investigate how different potential applications of the crumb rubber technology (i.e., low CR content on local roads vs high content on medium–high-trafficked roads) were distinguished by the blending variables. Hence, in this study, the effect of mixing duration, applied shear rate and CR concentration on the final properties of CRMB was evaluated.

In Australia, most CR applications are related to spray sealings; however, significant effort is being put to introduce and regulate its adoption in asphalt mixes. Commonly, local roads are constructed adopting standard bitumen, however, an attempt has been made in this paper to additionally introduce low-content CRMB as a possible alternative to use recycled materials and facilitate the disposal of EOLTs. This is in line with the recent "Light-Traffic Crumb Rubber Asphalt" specification released by the Department of Transport—Victoria (Australia) [38].

2 Material and Methods

2.1 Crumb Rubber

CR used in this study was collected from a tyres recycling plant in Victoria (Australia) that produces it using the ambient grinding process. In this process, tyres are first converted into chips by feeding into a shredder and then passed through a granulator which converts them into particles. The size of these particles can be further reduced by passing it through secondary granulators and rotary mills. The difference between the ambient and cryogenic process of CR production is the production temperature. During ambient grinding, the CR is produced either at room temperature or above while the cryogenic process uses much lower temperatures—i.e., up to − 80 °C. Gradation, chemical composition and surface texture of CR particles are summarized in Table 1 and Fig. 1, respectively. Table 1 shows that a major portion of CR particles is below 0.6 mm. SEM micrographs were captured using Philips XL-30 at two magnification levels, 100× and 200×. Figure 1a shows that the relative proportion of finer CR particles is greater than the coarser, and this is also confirmed from Table 1, sieve analysis data. By magnifying CR particles up to 200× (Fig. 1b), the surface texture can also be studied. The coarser particles are smooth with sharp edges while finer CR particles are of irregular shape and rough surface texture, hence providing greater surface area.

The characterization of CR particles was conducted as per ASTM E-1131, by decomposing CR samples in the Perkin Elmer Pyris-1 varying the temperature from 50 to 850 °C at a rate of 20 °C/min. The decomposition was completed in two stages; (i) 50 °C–750 °C in Nitrogen atmosphere, and (ii) 750 °C–850 °C in standard air. The reason behind this two-step decomposition process can be explained from Fig. 2.

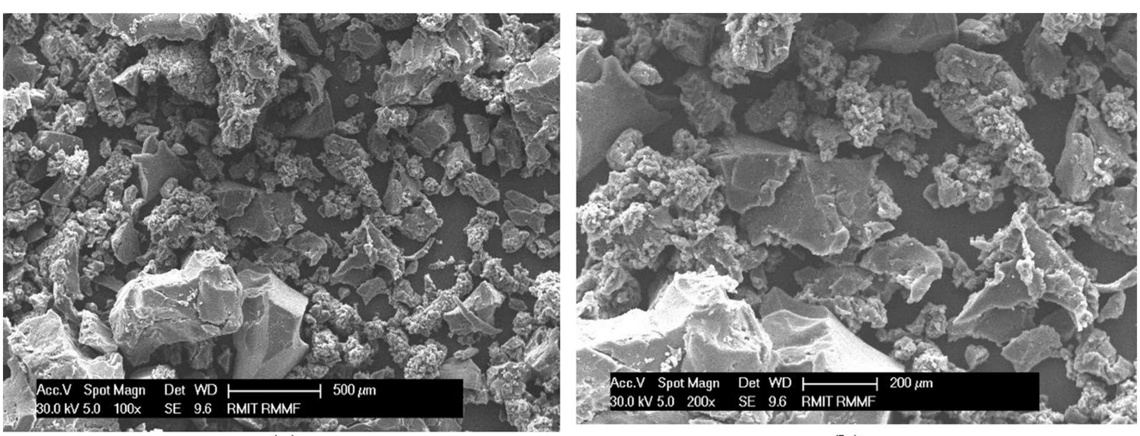
If samples are decomposed in air (as operational atmosphere) from 50 to 850 °C, synthetic rubber and carbon black decomposes simultaneously at approx. 550 °C due to the presence of oxygen in the air, which reacts with the carbon black [39, 40]. Hence, it is not possible to distinguish between the percentage of synthetic rubber and carbon black present in CR. On the other hand, if samples are decomposed in the nitrogen as inert atmosphere from 50 to 850 °C, though carbon black does not oxidize in this case but the separation of carbon black from the inert filler is not possible in this case. Therefore, a combination of both gases was used in which, the atmosphere was kept inert from 50 to 750 °C (to avoid the oxidation of carbon black) using nitrogen and was then switched to air after 750 °C (to distinguish between carbon black and inert filler). The sequence of decomposition peaks was identified as follows; oils (300 °C), natural and synthetic rubber (between 300 and 750 °C), carbon black (> 750 °C) and inert filler (Ash) [41].

2.2 Bitumen C320

C320 bitumen (equivalent to pen grade 50/70) was used as the base for CR modification. Its penetration (ASTM

Table 1 Physio-chemical properties of crumb rubber

CR gradation						
Sieve size (mm)	2.36	1.18	0.6	0.3	0.15	0.075
Passing (%)	100	100	89.8	29.6	9.0	4.2
Chemical composition of CR particles from Thermogravimetric analysis (TGA) analysis						
Constituent	Oils	Natural rubber	Synthetic rubber	Carbon black	Inert filler	
Weight (%)	6.5	40.0	17.5	29.3	6.7	



(a) (b)

Fig. 1 SEM Images of CR particles at magnification of **a** 100×, **b** 200×

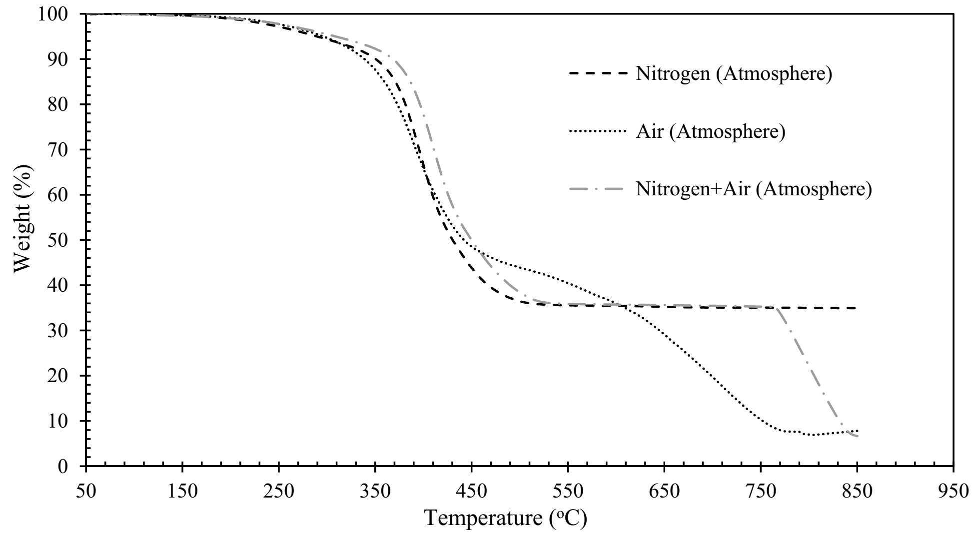


Fig. 2 Degradation of CR particles under nitrogen and air

D5), softening point (ASTM D36), and viscosity (ASTM D4402) at 135 °C was 59.3 (0.1 mm), 48.0 °C and 0.62 Pa·s, respectively.

2.3 Sample Preparation

CR was added into the bitumen at three different concentrations—7.5% (low), 15.0% (medium) and 22.5% (high) (by weight of bitumen)—and 177 °C. Several CRMB specifications around the world recommend dynamic viscosity of 1.5–4.0 Pa·s at blending temperatures for CRMB; this is probably due to the initial developments of the CR technology where contents greater than 18–20% were used in the mix. However, this range could not be achieved even when pushing the content up to 15% CR, as it can be seen from Fig. 3. Therefore, the upper limit of CR dose was set to 22.5%. The reason behind the selection of 7.5 and 15% of CR is that the local council roads in Australia are under pressure to provide service to new households, due to rapid increase in population and traffic. Commonly, local roads are constructed adopting standard bitumen; however, an attempt has been made in this paper to additionally introduce low and medium-content CRMB as possible alternatives to enhance the use of recycled materials and facilitate the disposal of end-of-life tyres. This agrees with the recent "Light-Traffic Crumb Rubber Asphalt" specification released by the Department of Transport—Victoria (Australia) [38]. Therefore, the 7.5% and 15% dosages were also considered and evaluated for the possible application of low content CRMB in low- to medium-traffic roads. CR was dispersed into the bitumen using Silverson L5M-A high shear mixer with a blending rate of 700 rpm (i.e., low shear rate) and 3500 rpm (i.e., medium–high shear rate). Samples were collected after 30, 60 and 90 min from the introduction of the rubber to study the effect of blending duration (reaction time) on the CRMB properties. The available body of literature shows a wide range of mixing speeds ranging from as low as 200 rpm [42] to as

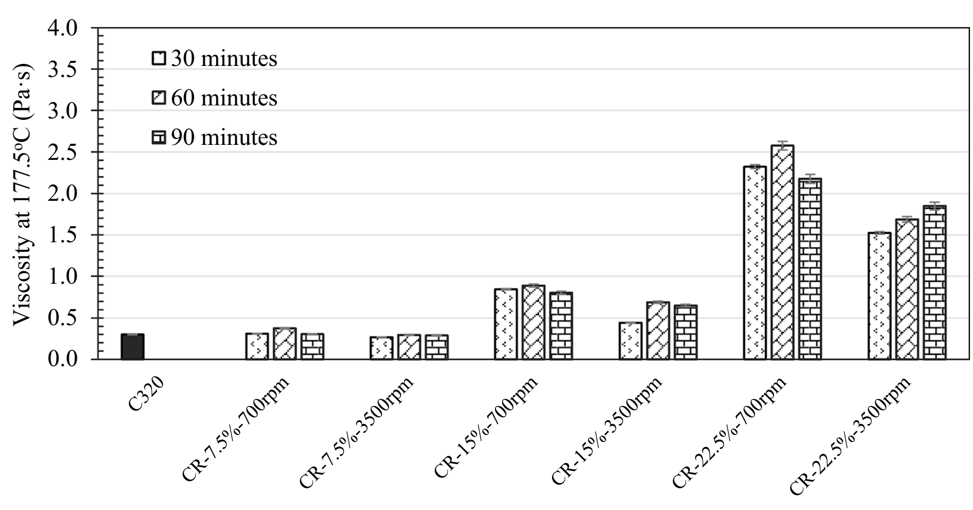


Fig. 3 Viscosity of crumb rubber-modified bitumen for various blending conditions

high as 8000 rpm [43]. The most commonly used mixing rate for CRMB is 700 rpm and, if SBS or other modifiers are to be added in the mix, then a mixing speed of 3000+ rpm has been used by various researchers. Therefore, for the present study, it was decided to use two shear mixing rates; namely 700 rpm (low) and 3500 rpm (high) to evaluate benefits and drawbacks associated with the mixing speed, if any. Although a number of specifications indicate approx. 45–60 min of reaction time, literature studies [44–48] show how shorter reaction periods can actually lead to insufficient penetration of the oily components into the rubber while an excessively long reaction time can cause depolymerization. Therefore, in this study, blending durations of 30 min (i.e., a shorter reaction time to evaluate whether the maltene fraction was able to penetrate the rubber particles), 60 min (i.e., commonly indicated by the standards worldwide) and 90 min (i.e., to evaluate the potential depolymerization) were selected to test CRMB.

2.4 Rheological Evaluation

Bitumen is sensitive to both loading time and temperature. The time- and temperature-dependent properties are termed as rheological properties and include; storage modulus (G'), loss modulus (G''), complex shear modulus (G^*), phase angle (δ), complex viscosity (η^*) and shear strain (γ). Viscosity at 177.5 °C was measured using Fungilab Viscolead rotational viscometer, while frequency sweep and multiple stress creep and recovery (MSCR) tests were performed using Dynamic Hybrid Rheometer-3 (DHR-3)—TA Instruments. Frequency sweep tests were conducted at 5, 20, 40 and 60 °C by varying the loading frequency from 0.1 to 15 Hz at a constant strain level of 0.01%. The 8 mm diameter parallel plate geometry with 2 mm gap was used for temperatures between 5 and 20 °C, while the 25 mm diameter plate with 1 mm gap was used from 40 to 60 °C.

MSCR test was conducted in accordance with AASHTO T350-19 at 60 °C using the 25 mm plate with a gap of 1 mm. Three stress levels (0.1, 3.2 and 6.4 kPa) were applied on each sample with a creep cycle of 1 s followed by 9-s recovery cycle. Twenty Creep and Recovery cycles were applied at 0.1 kPa, while ten cycles were applied for the other stress levels, according to the standard. Three replicates of each sample were tested, and their average value is reported in the paper with error bars showing the min–max range of measurements.

3 Results and Discussion

3.1 Viscosity at 177.5 °C

Viscosity of CRMB at 177.5 °C is a critical parameter because several current CRMB specifications use this property for evaluating CR blends. Viscosity of C320 bitumen modified at different CR concentrations and for various blending parameters is presented in Fig. 3.

Figure 3 shows that viscosity of C320 bitumen is around 0.30 Pa·s and, with the addition of 7.5% CR, the change in viscosity is negligible. When the quantity of CR is doubled, though the viscosity of CRMB is increased, but is below 1.0 Pa·s. Worldwide CR specifications usually recommend a viscosity range of 1.5–4.0 Pa·s; however, this value is achieved when CR is incorporated in more than 20%. At a fix CR concentration, the influence of mixing duration was found to be less significant; for instance, at 7.5%, 15% and 22.5% of CR concentration, viscosity of the blends varies from 0.30 to 0.37 Pa·s, 0.84 to 0.89 Pa·s, 2.18 to 2.58 Pa·s, respectively, when the blending duration is varied from 30 to 90 min. Nevertheless, a general trend of viscosity increase can be noted up to 60 min of reaction time whereas a slight viscosity decrease can be observed if the mixing is carried on further. The interaction between CR and bitumen is usually considered as a diffusion process by most and not chemical in nature. During this process, the oily components in the bitumen diffuse into the rubber which results in the swelling of rubber particles. The oily components continually decrease during this process while viscosity continuously increases. After an equilibrium is reached, the swelling process is replaced by depolymerisation, which results in a consequent decrease of viscosity [32, 49, 50]. On the other hand, increasing the shear mixer speed from 700 to 3500 rpm resulted in CRMB with relatively lower viscosity. Blending at high speed (i.e., 3500 rpm) may disintegrate the CR particles [30]; therefore, the undissolved CR particles of the blend prepared at 3500 rpm have smaller residual size as compared to the blends prepared at 700 rpm, hence offering reduced resistance to the rotating spindle of the viscometer.

3.2 Master Curves

The frequency sweep test measures the dependence of the binder shear strength (G^*) and viscoelasticity (δ) on temperature and loading frequency. The outputs of this test (G^* and δ) at different temperatures and frequencies are usually analyzed by plotting master curves. In this approach, data sets from frequency sweep tests are shifted via shift factors to a single reference temperature by the time–temperature superposition (TTS) principle. In this study, shift factors are calculated by regression analysis and the data is fitted into the Modified Sigmoidal Model using the following equation:

$$\log |G^*| = \delta + \frac{\alpha}{1 + e^{\beta-\gamma(\log f_r)}}, \tag{1}$$

where 'f_r' is the reduced frequency, 'δ' is the minimum complex modulus, and 'α' is the difference in the minimum and maximum modulus. 'β' and 'γ' are shape parameters of the curve, showing the horizontal position of the curve where it changes its shape and slope of curve, respectively [51]. According to Yusoff et al., the modified sigmoidal is the most accurate model for predicting the modulus of unaged, aged and polymer-modified bitumen [52, 53].

Figure 4 shows the comparison of CRMB master curves shifted to a reference temperature of 20 °C. The minimum complex shear modulus of C320 bitumen (i.e., high temperature, low frequency) is 369 Pa and when 7.5%, 15% and 22.5% CR is added into it at 700 rpm, it increased the shear modulus to 464, 1400 and 3000 Pa, respectively. However, when the same amount of CR is mixed at 3500 rpm, the minimum shear modulus at the corresponding dosages increased to 829, 2100 and 3400 Pa, respectively. Blends produced at high mixing speed generate stiffer CRMB than the one produced at low mixing speed. In addition, increasing the concentration of CR results in an enhancement of G^* at low reduced frequencies. Greater G^* at slow loading rates and high temperature provides betterments when coping with shear stresses and rutting.

The influence of CR at high frequencies (i.e., low testing temperature) is not clearly noticeable from the master curves plot as many curves seem to overlap in the log–log chart, especially at low CR content. To explore this further, complex viscosity has been plotted at 5 °C to show the CR effect at low temperature.

A general decrease in viscosity can be observed with increased frequency (Fig. 5), which shows the non-Newtonian or shear thinning behavior of bitumen; the rate of viscosity decrease is more pronounced in CRMB than C320 bitumen. Shear thinning in non-Newtonian fluids occurs due to the alignment of the particles or changes in the molecular orientation. Bitumen consists of asphaltene and maltenic fractions. Generally, asphaltenes are high-molecular weight micelles dispersed in the oily maltenic phase. When bitumen is subjected to a certain shear rate at low temperature, the asphaltenes are aligned in the direction of shear and there is a drop in viscosity with increasing frequency, which results in shear thinning behavior. In case of CRMB, the potential polymer entangled network—formed in the bitumen microstructure due to rubber–bitumen interactions—unfolds and stretches to adjust to the applied shear forces, which in turn makes the polymer network easy to deform [54].

It can be observed that the mixing duration does not have significant effect on complex viscosity when up to 15% CR is added into the bitumen at 700 rpm; however, when the mixer speed is increased to 3500 rpm, a reaction time of 30 min is enough for the swelling of CR particles. By increasing the mixing duration further, depolymerization of CR particles occurs and can be observed in the form of viscosity reduction. At greater CR concentration (i.e., 22.5%), at least 60-min reaction time is required for swelling CR particles due to the migration time of the oils from the bitumen to CR particles. Consistently, a decrease in viscosity is observed for longer reaction time (i.e., 90 min) due to the depolymerization process undergone by the rubber. Moreover, the addition of 22.5% CR in C320 bitumen has significantly reduced the viscosity of CRMB at low temperature in comparison to 7.5% content, which shows the effect of significant polymer modification—reduced thermal susceptibility—that was not sufficiently captured by the master curves plot.

The effect of the shear mixer speed at different CR concentration levels is shown in Fig. 6. The 60-min mixing time was selected as it is a common reaction time value in several CR standards worldwide [55, 56].

At low crumb rubber content (i.e., 7.5% and 15%) an upward vertical shift in the master curve is observed with increased mixer speed, which is more noticeable at lower frequencies. Baha et al. [31] and Tayh [32] used 10% CR and also noticed the stiffening effect in CRMB, when mixed at high mixing rates. However, when the CR concentration is increased from 15 to 22.5%, the effect of shear speed is minimal. The increase in G^* at high mixing rates can be related to oxidation. A non-negligible increase in complex viscosity was also observed in Fig. 5 at greater mixing speed. To analyze this further, an unmodified sample of C320 bitumen was heated under the same CR mixing conditions for 60 min at 700 rpm speed. Perkin Elmer Spectrum-100 Fourier Transform InfraRed spectroscopy (FTIR) Spectrometer was used to collect the spectra of: C320 bitumen, C320 bitumen conditioned for 60 min at 700 rpm, and CRMB at 7.5% concentration (700 and 3500 rpm for 60 min mixing). Samples were run in attenuated total reflectance (ATR) mode and a total of 32 scans were collected for each sample; the spectrum of each sample is presented in Fig. 7.

The strong peaks at 2920 and 2851 cm^{-1} show the presence of C–H stretches of alkanes, while a mild peak at 1599 corresponds to C=C in the aromatic structure. The asymmetric deformation of C–H in CH_2 and CH_3 and the symmetric deformation of C–H in the CH_3 were found in the range of 1410–1495 and 1360–1395 cm^{-1}, respectively. The small peaks between 650 and 810 cm^{-1} are the C–H vibrations in the benzene rings. The peaks in 1680–1750 and 980–1060 are usually associated with C=O (carbonyl) and S=O (sulfoxide) functional groups, respectively, and are often used to monitor the oxidation of bitumen [57–59]. The following equations are used to calculate the aging indices [60, 61].

$$\text{Carbonyl Index} = \text{CI} = \frac{A_{\text{carbonyl}(1680-1750)}}{A_{\text{CH2}(1410-1495)} + A_{\text{CH3}(1360-1390)}}, \quad (2)$$

$$\text{Sulphoxide Index} = \text{SI} = \frac{A_{\text{sulphoxide}(1060-980)}}{A_{\text{CH2}(1410-1495)} + A_{\text{CH3}(1360-1390)}}, \quad (3)$$

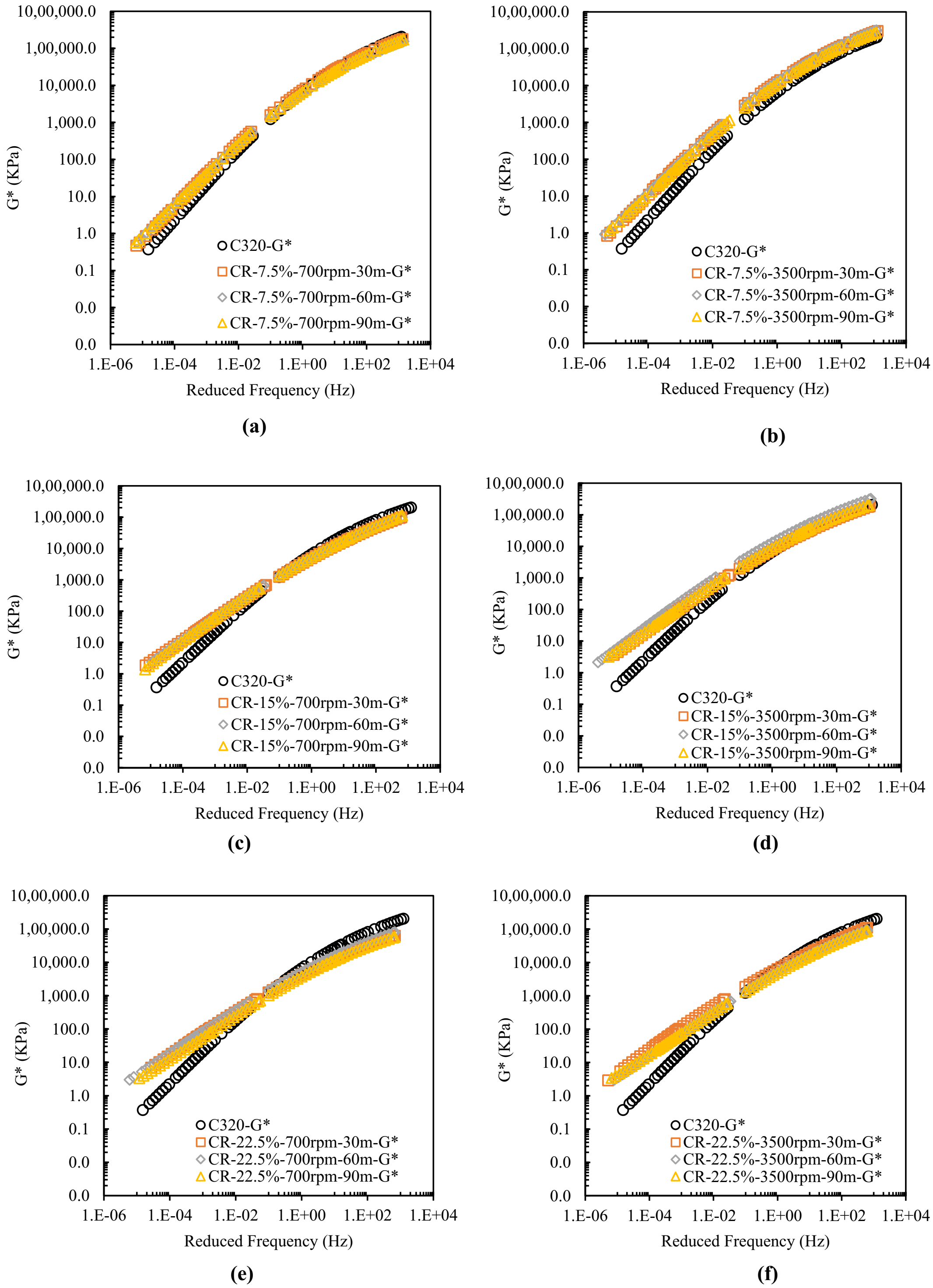
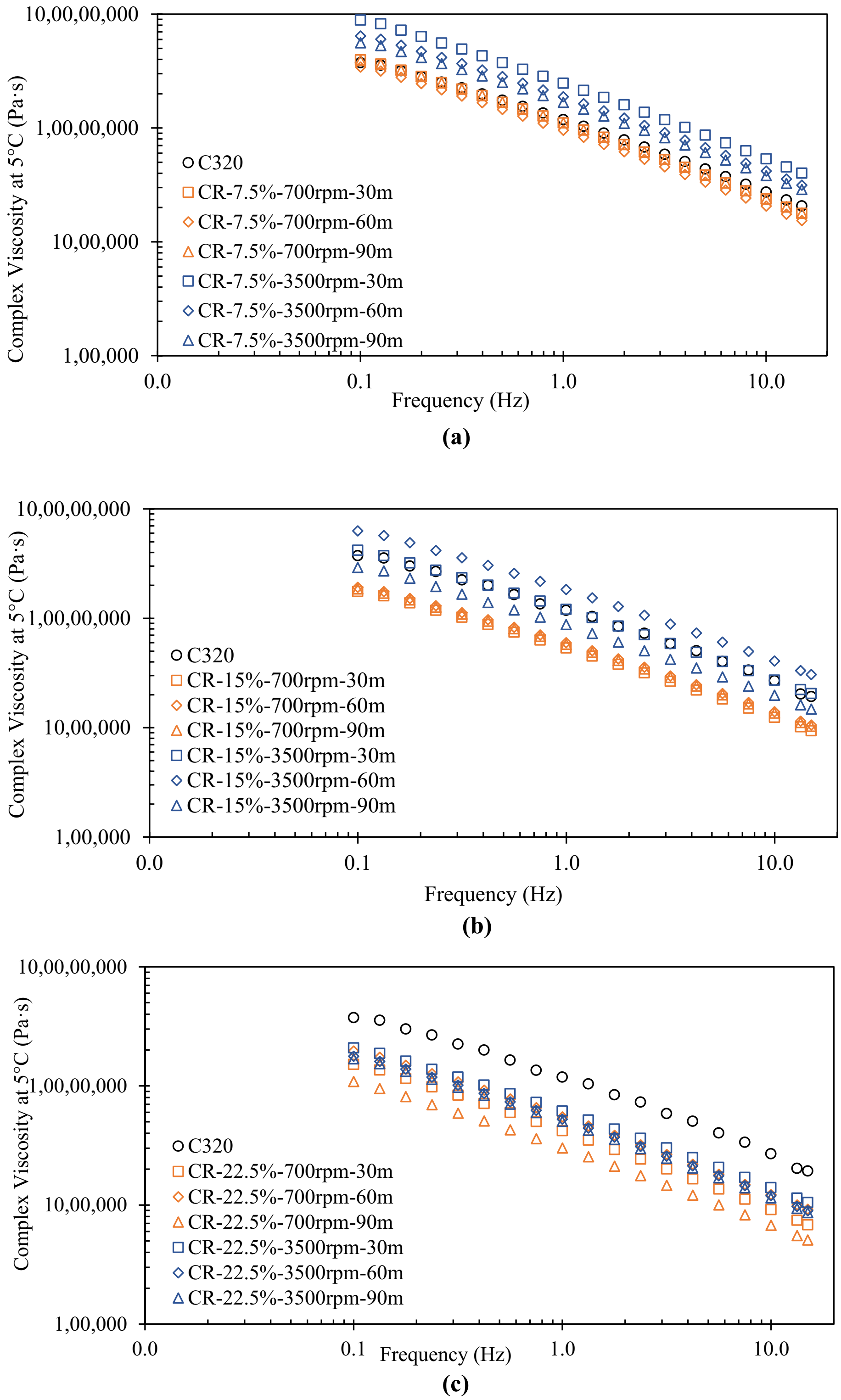


Fig. 4 Influence of digestion time, blending rate and CR content on CRMB master curves

where '*A*' is the area under the relative peaks. Areas under the peaks can be calculated using Spectragryph Optical Spectroscopy software.

CI and SI of neat C320 bitumen, and C320 bitumen blended alone for 60 min at 700 rpm (to simulate the same mixing conditions of CRMB) and 7.5% CRMB are shown in Fig. 8. It can be noted that C320 bitumen blended alone has the highest CI and SI values which shows the susceptibility of neat bitumen to aging as compared to CRMB. The addition of CR reduced the extent

Fig. 5 Complex viscosity plot at 5 °C



of oxidation of neat bitumen; a similar observation was also reported by Wang et al. [62]. On the other hand, if a comparison is to be made between the aging indices of the two CR-modified blends prepared at different mixing speeds, it can be noted that the shear mixer speed has a significant effect on the SI index of CRMB. Blends prepared at 700 rpm have SI value of 0.135, while SI is equal to 0.186 when the mixer speed is increased to 3500 rpm. Though no significant difference was noticed for the CI index. Overall, an increase in the value of the

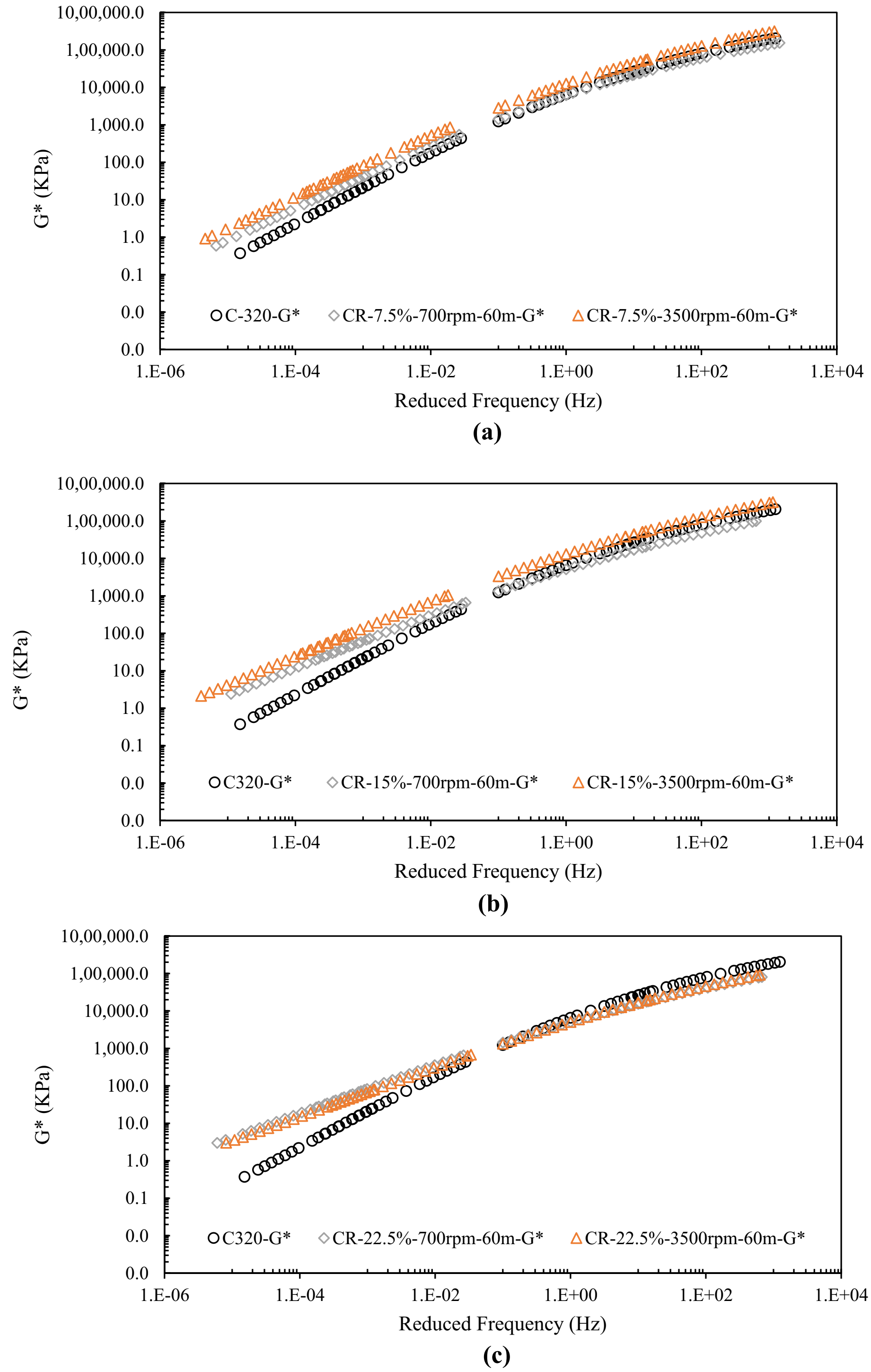


Fig. 6 Influence of the shear mixer speed on CRMB master curves (700 and 3500 rpm after 60 min)

aging indices at increased mixing speed was identified for low content CRMB.

The effect of various CR concentration levels on G^* of CRMB is shown in Fig. 9.

Figure 9 shows that increasing CR content generates greater G^* values for CRMB at high temperature, but reduces G^* at low temperature, hence reducing the thermal susceptibility of CRMB.

3.3 Black Diagram

Black diagrams are another way for analyzing data sets obtained from frequency sweep tests and help to distinguish between a thermo-rheologically simple and complex material, capturing any polymeric modifications of bitumen [63]. The influence of blending conditions on black diagrams is shown in Fig. 10. It may be noted from Fig. 10 that the

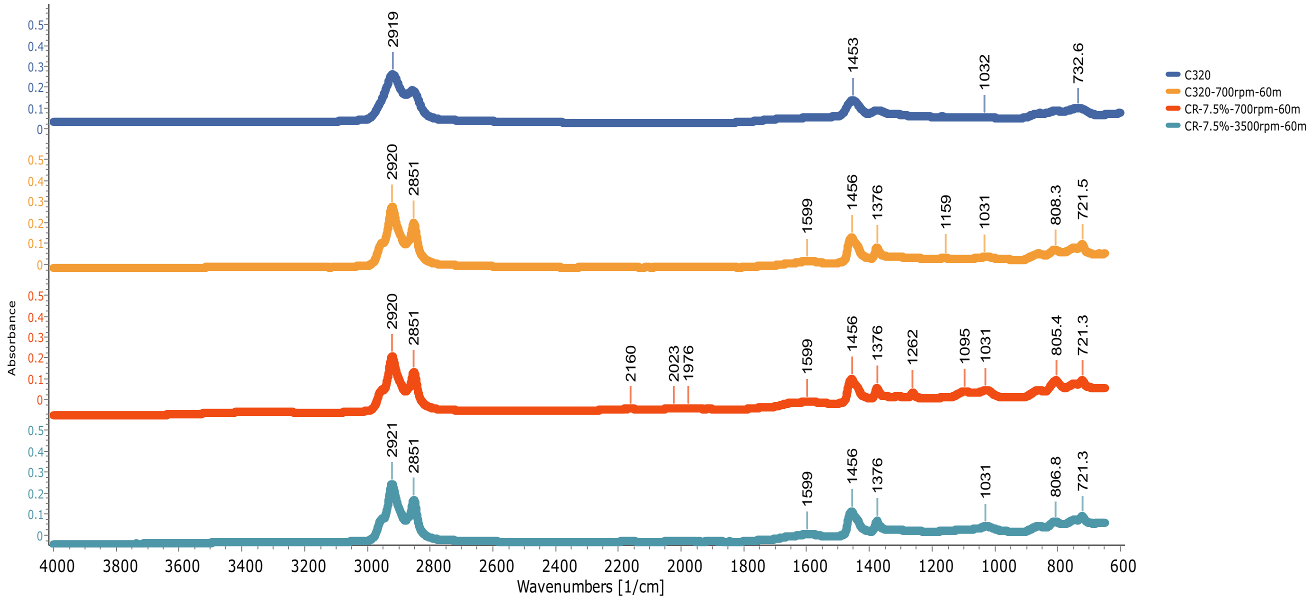
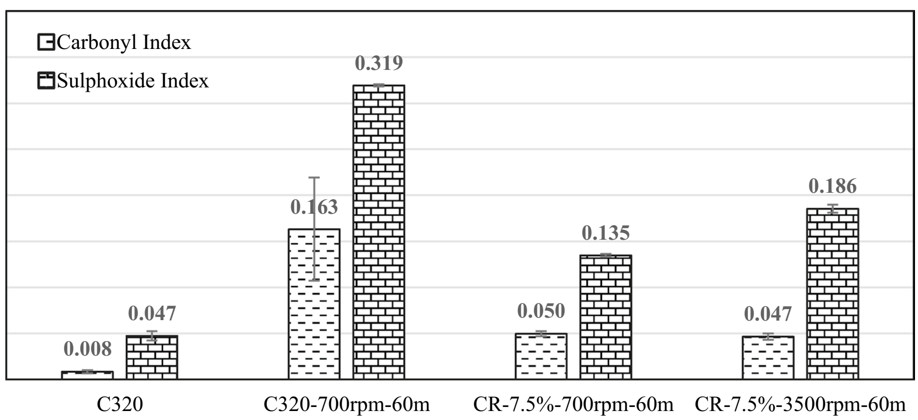


Fig. 7 Influence of shear mixer speed on the FTIR spectra of CRMBs

Fig. 8 Influence of shear mixer speed on the aging indices of C320 and CRMB



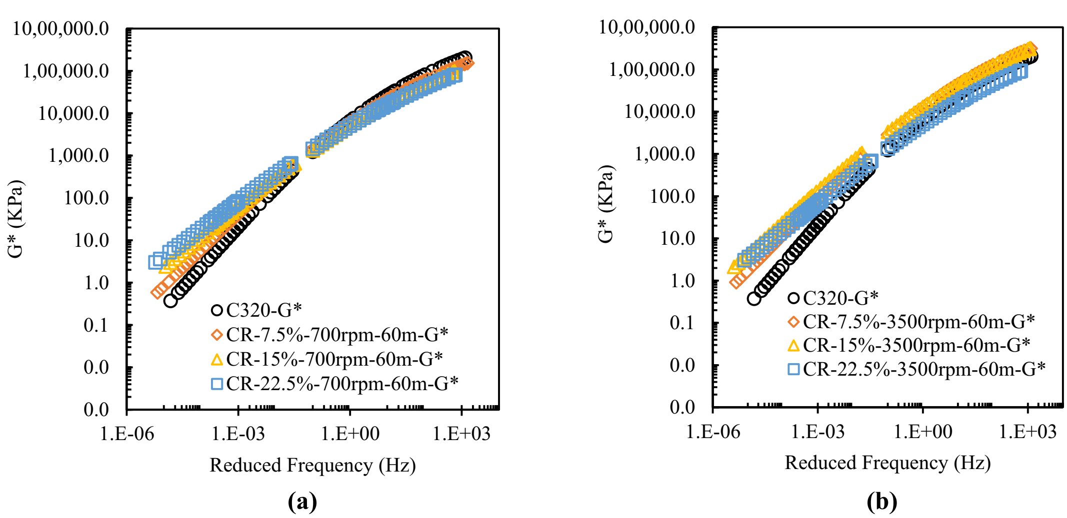


Fig. 9 Influence of CR concentration on the rheology of CRMB

blending duration is not an influential factor in affecting the shape and magnitude of CRMB black diagrams, but rather CR concentration is. The black diagram of C320 bitumen shows a smooth transition from a viscoelastic solid to viscoelastic fluid, hence showing its thermo-rheologically simple behavior [64]. The curves discontinue after reaching phase angle values of approx. 68°, 60° and 54° for 7.5%, 15% and 22.5% CR, respectively. This shows the biphasic nature of the material and the polymeric modification due to the presence of CR [63]. These phase angle values show a boundary line between the bitumen dominant phase and the polymer dominant phase. On the left side of these threshold values, bitumen is the main driver of the rheological behavior and the characteristics of the blends are similar to those of the unaged bitumen; however, on the right end side of these threshold values (high temperature), the base bitumen starts melting, hence leaving the CR particles to take the shear load. The combined effect of G^* and δ at different intermediate and high temperatures can be quantified using Strategic Highway Research Program (SHRP) parameters after Rolling Thin Film Oven test (RTFO) and Pressure Ageing Vessel (PAV) aging [65]; i.e., $G^*/\sin(\delta)$ (rutting parameter) and $G^*\cdot\sin(\delta)$ (fatigue parameter). Figure 11a, b shows the plotting of the rutting values at 60 °C and fatigue at 20 °C, respectively.

In the previous section i.e., 3.2, three types of phenomena were observed to occur during the blending process of CRMB; (i) swelling, (ii) depolymerization of CR particles and (iii) aging of CRMB. Generally, swelling and aging increases the rutting resistance of CRMB while depolymerization decreases the rutting resistance. Figure 11a shows the effect of blending conditions and CR concentration on rutting resistance of CRMB. On average, a mixing duration of 60 min seems to be optimal at low mixing speed (700 rpm) as it provides greater rutting resistance than 30 and 90 min; i.e., 6.6 kPa as compared to 6.5 kPa and 6.2 kPa at 7.5% and 25.4 kPa as compared to 24.3 kPa and 23.5 kPa at 22.5%. The 30-min reaction time is probably not enough for the development of proper polymeric network in CRMB while 90 min result in damaging the integrity of the polymer network. However, a negligible difference (0.1 kPa) between 30 and 60 min is observed for low content CRMB (i.e., 7.5%), meaning that a shorter reaction time could already be enough to trigger the beneficial effects of CR addition when the rubber/bitumen ration is small. This aspect should additionally be explored in relation to the size of CR particles and their chemical composition since a finer mesh with greater content of natural rubber is easier to digest than a coarser mesh with greater content of synthetic rubber. Further investigation on blending parameters with different sources of rubber is, therefore, recommended.

Increasing the mixer speed (i.e., 3500 rpm) resulted in greater rutting resistance values than the corresponding blends prepared at 700 rpm. In addition to a more thorough mixing effect, an aging component—due to higher mixer speed—was also noted to be responsible for greater stiffness and reduced phase angle, as discussed in the previous sections. In the case of 3500 rpm, the greatest rutting resistance was observed after just 30 min of mixing, therefore decreasing the average optimal mixing time by 30 min compared to 700 rpm. Greater blending shear rates favor the quick mechanical (i.e., through diffusion) swelling of CR, though a further increase in mixing duration deteriorates the developed network which appears as a drop in rutting resistance. The rise in the rutting resistance parameter during the last 30 min of mixing (i.e., between 60 and 90 min) is thought to be correlated with aging, although no specific test was conducted for the eighteen combinations studied in this paper.

Figure 11b shows how the influence of mixing shear speed is more of a concern than the mixing duration for the fatigue parameter, as it reduced the $G^*\sin(\delta)$ value showing poorer fatigue behavior of the blends prepared at higher mixing rates. A clear trend of the fatigue parameter with the reaction time was not identified. However, it was evident that increasing the CR concentration level improved the fatigue performance of CRMB. Further study should be dedicated to the analysis of SARA (Saturate, Aromatic, Resin, Asphaltene), hence coupling rheological findings at different blending conditions with the chemical composition of CRMB.

Finally, it can be inferred that adding a small amount of CR (i.e., 7.5%) helps reduce the rutting potential significantly; this can be beneficial for low-traffic environments and local roads. However, to produce some betterments in fatigue performance, at least 15% CR should be included in the blend. Significant discussion is available in literature about the ability of $G^*/\sin(\delta)$ and $G^*\sin(\delta)$ parameters to reflect field performance; MSCR tests were also conducted in the following sections to provide a more comprehensive analysis.

3.4 Cole–Cole Plot

Cole–Cole plot, commonly used for dielectric materials [66, 67] is a way to show a graphical description on a linear or log scale of the viscous and elastic part $[G'' = f(G')]$ of materials' stiffness. It evaluates the contribution of the elastic (shear storage modulus) and viscous (shear loss modulus) components that contribute to the overall stiffness of the material. The transition from liquid-like to solid-like behavior can also be characterized by the crossover frequency, which is the frequency at which $G' = G''$. A line at 45° angle is usually drawn to indicate the location of $G' = G''$ points. The 45° line identifies the border that distinguishes between the elastic dominant and viscous dominant behavior of the material. Cole–Cole plots for C320 and CRMB at various CR concentrations, mixing

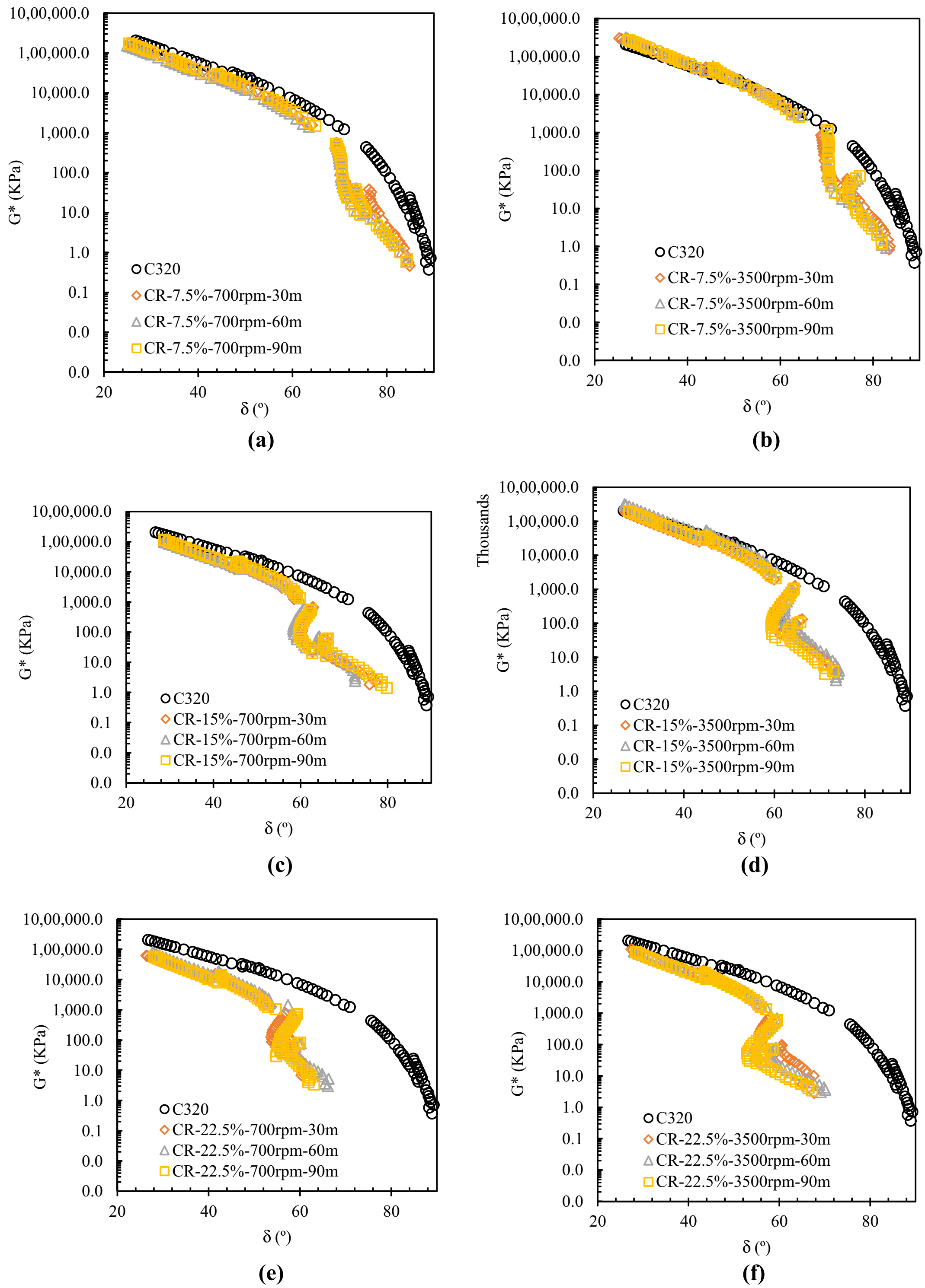
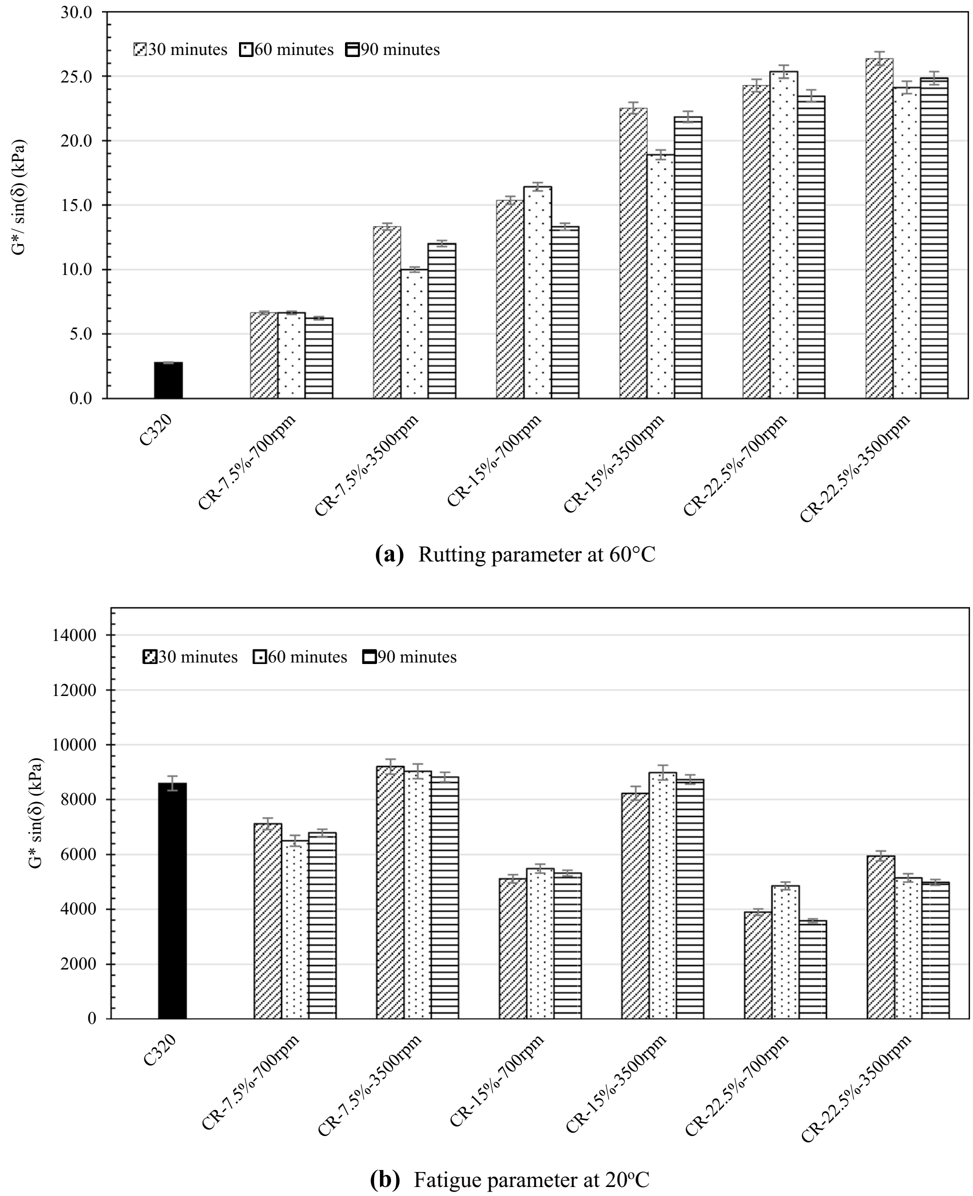


Fig. 10 Black diagrams of C320 bitumen modified with CR at 700 rpm (**a**, **c**, **e**) and 3500 rpm (**b**, **d**, **f**)

durations and shear rates are shown in Fig. 12. It can be noted that the blending duration has minimal effect on the relative contribution of viscous and elastic components over the entire range of stiffness values. However, most of the stiffness values of C320 bitumen show a significant viscous component compared to CRMB; this demonstrates the greater elastic response of CRMB, especially at high and intermediate temperature.

Fig. 11 Influence of CR blending conditions on SHRP parameters



Cole–Cole plots can also be used to check whether the material has an ideal Maxwellian behavior or not. An ideal Maxwellian fluid shows a semicircular Cole–Cole shape, if G' and G'' are plotted on a linear scale [68]. Perez et al. [69] proposed an equation to fit G', G'' with frequency close to the T_g glass transition temperature; however, Nait-Ali et al. [70] found that it is valid at temperatures far from T_g, also. The following equation is proposed by Perez:

$$G^* = G_c + \frac{G_L - G_c}{1 + H\left(i\omega\tau_{mr}\right)^{-h} + \left(i\omega\tau_{mr}\right)^{-k}}, \quad (4)$$

where ω is the angular frequency, G_C and G_L are the relaxed and unrelaxed modulus, respectively, and τ_{mr} is the time for molecular relaxation. Cole–Cole plot model parameters k, h and H can be obtained by plotting G' versus G''. The 'h' parameter of this equation has a significant importance and is assumed to be linked to topographic defects at the molecular scale. The term topographic defect was introduced by Perez et al. [69] to predict the mechanical behavior of amorphous polymers under the application of periodic stresses. At temperatures close to T_g, some sites of density fluctuations develop when the monomers get closely packed. Resistance to applied stress is very weak at these sites due to nucleation of shear microdomains. Rolere et al. [71] concluded that the gel fraction of natural rubber acts as a defect and limits the mobility of polyisoprene chains due to physical and chemical crosslinking and found a good correlation between the total gel content and h parameter. Nait-Ali et al. [70] observed a gradual decrease in the h parameter during

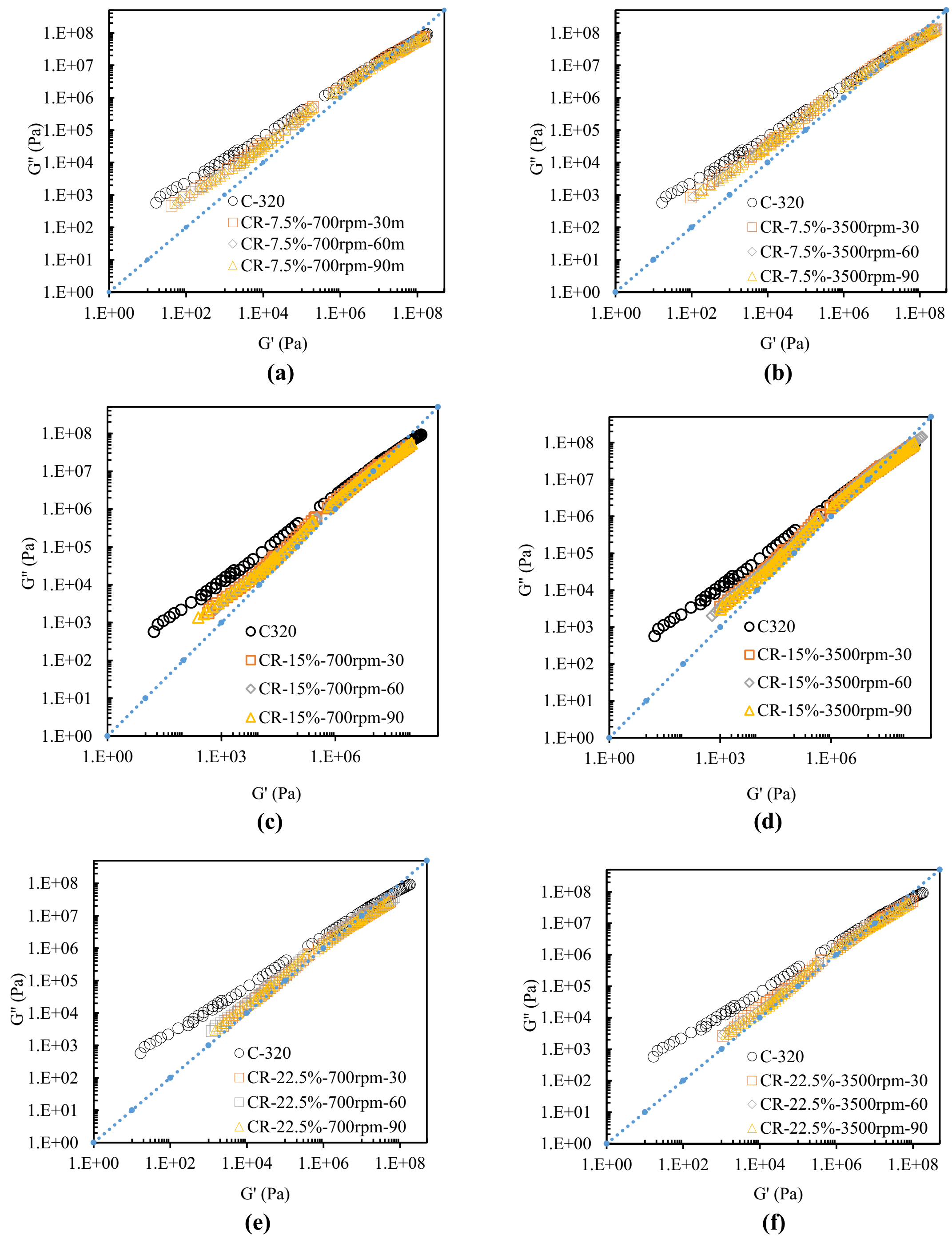


Fig. 12 Cole–Cole plot of C320 bitumen and CRMB for different blending conditions

the mechanical degradation of PET, indicating an increase of branched chains in the polymeric matrix. Bergeret and Alberola [72] used this concept in the study of the interaction between glass beads and the polymeric matrix and found a decrease in the h value when the interaction was improved. Therefore, it can be assumed here that there will be a decrease in h value with increasing CR–bitumen interaction and that an overall reduction in the polymeric network will increase the h value. Rolere et al. [71] proposed a simple technique to calculate the h value. The methodology could be summarized as follows; first, plot G' against G'' on a linear scale and fit it with a fourth-order polynomial. Finally, use the following equation to calculate h:

$$h = \frac{2}{\pi}\left[\arctan\left(\frac{dG'}{dG''}\right)\lim_{G'\to 0}\right]. \quad (5)$$

The h value for the different blending conditions are presented in Table 2.

Table 2 shows that—based on the interpretation of the model—the h value is influenced by the mixing duration, shear mixer speed and CR concentration. The h value decreases at 700 rpm and 60 min, which shows that CR particles are swelling and creating an effective polymeric network with the bitumen [70]; however, a further increase in the reaction time decreases the h value, which can be linked to the depolymerization of CR particles for longer blending durations. When the shear speed is increased from 700 to 3500 rpm, one can notice that the lowest h value corresponds to 30 min of mixing. This shows that the faster mixing speed makes the CR particles swell quickly and a further increase in mixing duration disturbs the polymeric network (increased h value). Generally, CR concentration is also influencing the h value and an increase in CR concentration provides more points of polymer–bitumen interaction, hence an overall decrease in h value with increasing CR concentration.

3.5 Rutting Potential (Multiple Stress Creep Recovery test)

J_{nr} (non-recoverable creep compliance) and %R (percent recovery) were determined for each stress level; results are plotted in Fig. 13. The temperature value of 60 °C was selected for all the blends for comparison purposes. As expected, C320 bitumen showed very high non-recoverable deformation and minimal recovery at 60 °C. With a softening point of just 48 °C, standard bitumen does not cope well with plastic permanent deformations at high temperature.

The small addition of 7.5% CR reduces the non-recoverable deformation by half. Moreover, a significant improvement can be observed for the percent recovery due to CR. The influence of different stress levels on the non-recoverable strain and %recovery of the blends—at 60-min reaction time—is shown in Fig. 13. It can be observed that C320 bitumen has a similar J_{nr} value at all the stress levels, which further evidences that the test temperature is excessive for it to resist to any applied stress. Similarly, %R of C320 is minimal and further reduced when the stress increased from 0.1 to 6.4 kPa. CRMB showed a significant decrease in J_{nr} and an increase in %R compared to the standard bitumen, hence possibly improving resistance to rutting in the field. In addition, the shear mixer speed was deemed to have an influence on J_{nr} and %R, although very small in magnitude and mostly depending on CR concentration. At low concentration of CR, the blends prepared at 3500 rpm exhibited lower J_{nr} value compared to blends produced at 700 rpm. This shows that the 3500 rpm blend holds on to a stiffer behavior at 60 °C as observed in the rheological section and FTIR analysis. However, the influence of the shear speed on J_{nr} becomes negligible at higher concentrations of CR.

Table 3 quantifies the effect of the blending parameters and various CR concentrations on CRMB and provides a comparison with C320 bitumen. In this table, the percentage change—between CRMB and C320—in J_{nr} and %R values under different stress levels is reported. It can be observed that the percent reduction in non-recoverable deformation is mainly dependent on CR concentration and is only slightly influenced by the shear rate and mixing duration. Although a dependence on blending parameters is more evident at lower concentrations of CR (i.e., 7.5%), this tends to reduce for 15% and 22.5% CR modification. For example, if the concentration is increased from 7.5 to 15%, the J_{nr} value is reduced from 57 to 88%, which holds to a relative change of 31%; this variation is much larger than that associated with the mixing duration and shear mixer speed for the 15% blend (i.e., +3% only). Similarly, %recovery increases mainly because of higher CR concentration (Table 3) in CRMB blends.

Compared to C320, 7.5% CR has proved to significantly improve MSCR parameters; in particular, J_{nr} reduces by approx. 64% and %R increases by approx. 1175%. The use of low-content CR asphalt mixes on local roads could represent an opportunity for local councils and road authorities to improve rutting resistance while reducing the volume of material to be sent to landfill.

Table 2 Influence of CRMB blending conditions on the value of the h parameter

Mixing speed (rpm)	Mixing duration (min)	CR concentration		
		7.5%	15.0%	22.5%
700	30	0.5564	0.5530	0.5423
	60	0.5515	0.5521	0.5375
	90	0.5572	0.5542	0.5425
3500	30	0.5649	0.5522	0.5547
	60	0.5716	0.5639	0.5570
	90	0.5793	0.5647	0.5574

3.6 Comparison with Outcomes from Similar Studies

Thives et al. [37] studied the effect of the rubber digestion time using penetration, softening point, viscosity at 175 °C, resilience at 25 °C and microscopy analysis. Ambient and cryogenic CR samples at 16%, 17% and 20% concentration were separately mixed in two types of base bitumen (pen 50 and pen 30) at 180 °C for 30, 45, 60 and 90 min. CR produced at ambient temperature had a surface area of 19.30 kg/

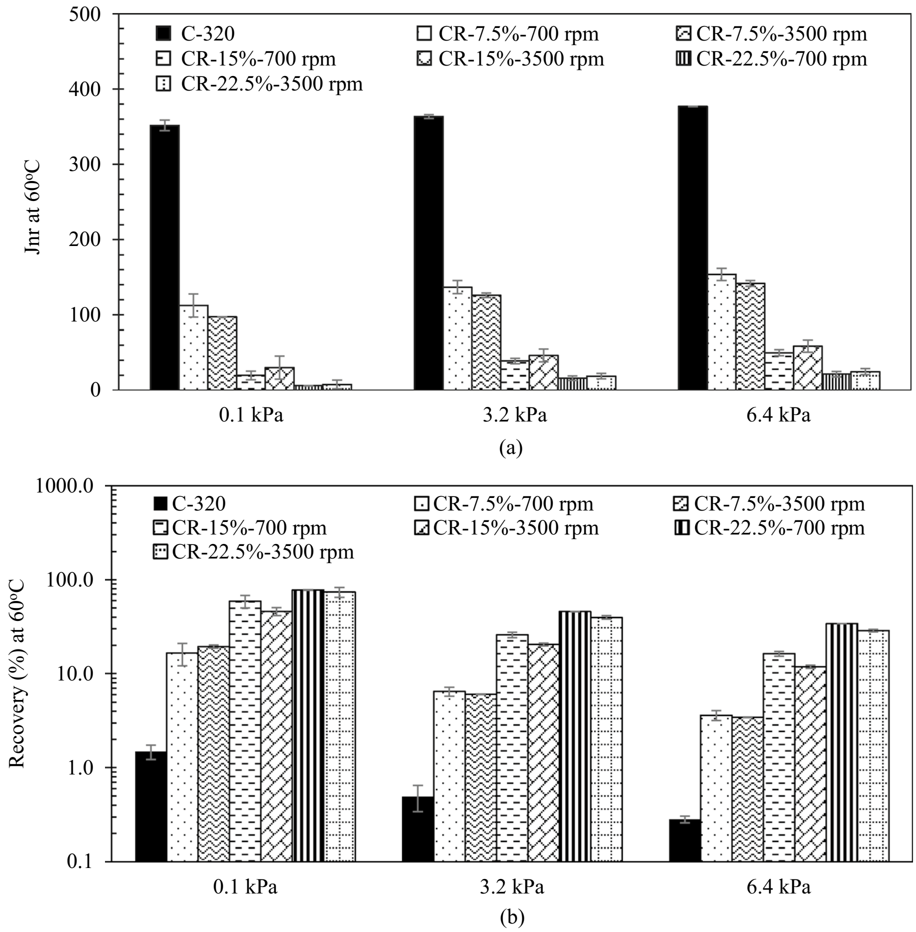


Fig. 13 Influence of various stress levels on J_{nr} and $\%R$ of CRMB at 60-min blending duration

Table 3 Percentage change in the J_{nr} and $\%R$ of CRMB with respect to C320 bitumen (*darker colors represent greater %change)

CRMB Blend	% change CRMB-C320 J_{nr}			% change CRMB-C320 %R		
	0.1 kPa	3.2 kPa	6.4 kPa	0.1 kPa	3.2 kPa	6.4 kPa
CR-7.5%-700rpm-30m	-63	-57	-54	837	829	766
CR-7.5%-700rpm-60m	-68	-62	-59	1015	1218	1172
CR-7.5%-700rpm-90m	-68	-62	-59	1093	1381	1388
CR-7.5%-3500rpm-30m	-72	-64	-61	1194	1077	1027
CR-7.5%-3500rpm-60m	-72	-65	-62	1208	1128	1112
CR-7.5%-3500rpm-90m	-75	-70	-67	1233	1701	1780
CR-15%-700rpm-30m	-94	-88	-85	3713	3760	3853
CR-15%-700rpm-60m	-94	-89	-87	3871	5148	5638
CR-15%-700rpm-90m	-94	-88	-84	4006	5097	5391
CR-15%-3500rpm-30m	-97	-89	-87	4795	4403	4378
CR-15%-3500rpm-60m	-91	-87	-84	3005	4036	4066
CR-15%-3500rpm-90m	-94	-89	-87	3524	4751	5012
CR-22.5%-700rpm-30m	-100	-96	-95	6370	8593	10811
CR-22.5%-700rpm-60m	-98	-96	-94	5132	9178	11888
CR-22.5%-700rpm-90m	-99	-96	-95	5840	9619	12508
CR-22.5%-3500rpm-30m	-99	-94	-93	6110	6288	7275
CR-22.5%-3500rpm-60m	-98	-95	-93	4878	7965	10026
CR-22.5%-3500rpm-90m	-98	-96	-95	5148	9366	12435

m^2, which is almost equal to the surface area used in this study. Similarly, pen 50 bitumen had a closer penetration and softening point to the C320 bitumen used in this study and these two can, thus, be compared. However, the speed of the mixer used for the blending process was not mentioned by the authors in their article. According to Thives et al., the effect of digestion time was only significant for up to 60-min blending duration. Samples were further studied by SEM analysis and a more uniform rubber dispersion was observed in the samples blended for 90 min; this value was selected as the optimal mixing duration. However, in the present study, an increase in rotational viscosity was observed up to 60 min of digestion time when blended at low shear rate (700 rpm), whereas viscosity reduced after that. By increasing the shear mixer speed up to 3500 rpm, the 60-min threshold dropped to 30 min.

The effect of the shear mixer speed was studied by Tayh and Yousif [32] by blending 10% of ambient grinded 40-mesh CR into 80/100 pen grade bitumen at 180 °C at a mixing speed of 250, 750 and 1250 rpm for a constant blending time of 60 min. The blends were characterized using softening point, rotational viscosity and SHRP rutting parameter ($G^*/\sin\delta$) tests. A decrease in rotational viscosity was observed with increasing shear speed. The reason provided by the authors was that the increasing mixer speed provides enhanced interaction and dispersion of CR into bitumen. A reduction in rotational viscosity due to increasing shear mixer speed was also observed in this study although two main distinctions can be provided: the maximum shear speed of 3500 rpm (almost threefold) and the type of mixer. While Tayh and Yousif [32] used a standard four-blade impeller which mainly blends—by dispersion—the CR into the bitumen, this study utilized a similar overhead stirring mixer but with a slotted disintegrated head, specifically developed for blending elastomers and rubbers by disintegration and solubilization. This could have facilitated the disintegration of CR particles and fostered the interaction and swelling of CR particles in bitumen, hence a reduction in viscosity. Similarly, Kok et al. [31] mixed 10% CR in 50/70 pen grade bitumen at 180 °C for 30, 60, 90 and 120 min using a shear mixer speed of 1000, 3000 and 5000 rpm; the modified binders were then evaluated for conventional and rheological properties. They concluded that the effect of rubber addition (10% CR content) is less significant as compared to the processing conditions due to the oxidation of the base binder. More oxidation at 7.5% CR content was also identified in the present study.

CRMB specifications around the world are mostly considering viscosity, reaction time, temperature and a fix concentration of CR (i.e., greater than 17%), which could potentially limit the scope of low-to-medium CR contents to be efficiently used in low-traffic roads. When low-to-medium CR concentrations are used, the blending conditions should also be considered by future specifications as the interaction between bitumen and rubber may significantly vary. For instance, the prescriptive 60-min reaction time and viscosity range of 1.5–4 Pa·s could not be achieved at low CR concentration (i.e., up to 15%—Sect. 3.1) and should, therefore, be revised. In addition, other parameters—such as the blending speed rate, not commonly considered by the standards—should also be evaluated as it can cause aging on low content CR blends and stiffening of the binder as compared to low shear blends. Viscosity of CRMB alone cannot be used as a prescriptive criterion to predict the in-service performance of CR-modified asphalt, especially if CRMB is to be used on minor roads with limited traffic and mainly for environmental purposes (i.e., fostering recycling).

4 Conclusions

This study evaluates the influence of blending conditions and CR concentrations on the rheological properties of CRMB. Mixing duration (reaction time), shear speed of the mixer and CR content all have an influence on the swelling process of CR particles and, thus, on the final rheological properties of CRMB. Based on the experimental results, it can be concluded that the optimal mixing duration is mainly dependent on CR concentration and shear mixer speed. On average, 60 min is a sufficient time to allow CR particles to fully provide rheological benefits at all CR contents. A mixing duration of more than 60 min or medium–high shear mixer speed (i.e., 3500 rpm) does not generally provide any rheological benefits, but rather produces depolymerisation (Cole–Cole *h* value) and aging (FTIR) effects, respectively; and this is more relevant for low-content CR blends (i.e., 7.5%). The aging effect associated with the high shear mixer speed increases the value of the Superpave rutting parameter ($G^*/\sin\delta$) but, at the same time, it also reduces the fatigue parameter (i.e., $G^*\sin\delta$) of the modified blends at all CR contents and blending durations. This means the blends produced with low shear mixer speed (i.e., 700 rpm) and a mixing duration between 30 and 60 min have better rheological properties.

The effect of CR concentration is quite significant. In particular, the elastic response (i.e., reduction of phase angle) is significantly improved with increasing CR content, especially at high temperature. Additionally, the viscous response at low temperature (i.e., complex viscosity at 5 °C) is also enhanced by the addition of a greater quantity of CR. Both the SHRP rutting and fatigue parameters are improved by greater CR contents; however, it can be noticed that betterments in rutting resistance due to CR are more pronounced that those in fatigue resistance. Moreover, the MSCR test confirms the previous findings about rutting improvements; incremental benefits can be seen when adding more CR in terms of decreased J_{nr} and increased %Recovery at all stress levels (i.e., from 0.1 to 6.4 kPa) compared to standard bitumen. The benefits associated with the addition of CR also suggests that the low content CR asphalt mixes have the potential to be largely used in local roads, hence reducing the amount of EOLTs produced annually. The experimental outcomes in this study can be used to further refine CR specifications.

5 Highlights

- Recycling end-of-life tyres can be further promoted by more accurate specifications.
- Blending conditions affected the chemo-rheological properties of modified bitumen.
- High mixing speed induces aging in low-content rubber-modified bitumen.
- Bitumen–rubber reaction time should vary depending on concentration and blending speed.

Author Contributions MJ and FG: Conceptualization; MJ and FG: Methodology; MJ: Investigation and testing; MJ and FG: Data analysis; MJ: Writing—original draft; MJ, FG: Writing—review and editing, FG: Project administration, funding and supervision.

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Declarations

Conflict of interest The authors declare not to have any conflict of interest.

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Muhammad Jamal completed his master's and bachelor's degrees in Civil Engineering from Pakistan and is now enrolled in the Ph.D. program at RMIT University since 2018. He has contributed to road motorway and airport runway construction projects in Pakistan. During his master's degree research work, he proposed a feasible solution to recycle the waste material generated from the rehabilitation of 357-km-long three-lane dual carriageway motorway M-2, Pakistan. Currently, he is working on the development of 'Crumb Rubber Asphalt Binder Characterization and Specification for Road Pavement Applications' under the supervision of Associate Professor Filippo Giustozzi at RMIT University.

Associate Professor Filippo Giustozzi is an expert in road and airport pavement materials. He completed his second Ph.D. at Virginia Tech University (USA) and is now the Chair of the Technical Committee on Sustainable and Resilient Pavements at the Transportation Research Board of the National Academies of Sciences and Engineering in the USA. He has participated in several major road and airport construction projects since 2008. Dr. Giustozzi is the Lead Investigator of the national Austroads project APT6305 on Road-grade recycled plastics for sustainable asphalt pavements, approved by the Transport and Infrastructure Council that brings together Commonwealth, State, Territory and New Zealand Ministers. He also collaborates with the Australian Flexible Pavements Association on crumb rubber projects and with several national and international road contractors and bitumen suppliers on a variety of research and field projects, mainly on polymer-modified bitumen and recycled materials for road applications. At RMIT University, he is leading the Intelligent Materials for Road and Airport Pavements research group and has developed the only university-based fully equipped asphalt and bitumen laboratory in Australia, which currently employs more than 20 researchers.