Changes in Densities of Basic Constituents during CRMB Production

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***Abstract* -- Along with other polymers, Crumb Rubber (CR) obtained from scrap tyres has also resulted in enhanced performance of the virgin binder to meet the increased traffic density. However, the nature of reaction between the primary components has been reported to be different compared to the other polymer modification resulting in an heterogeneous blend of primary components. Despite the enhanced physical and performance characteristics of CRMB such as improved rutting and fatigue resistance, the product is still not widely accepted; the reason being inadequate and inconsistent observations from different researchers and agencies around the world. This has also led to a gap, where industry and the research community are still trying to understand and control the reaction mechanism between CR and bitumen to obtain consistent results. To understand the reaction mechanism, a total of 27 CRMB blends with different material combinations were prepared in the laboratory. To examine the changes, samples were collected after different intervals of blending and further subjected to laboratory observations for volumetric changes. The collected samples were then put through the separation processes to recover binder residue and swollen CR for further laboratory analysis. Results demonstrated that the aging of bitumen make it harder due to oxidation. Equally CRMB production also contributes to the hardening of base bitumen due to the diffusion of lighter fractions of bitumen into the CR particles. This resulted in changes in volumetric of the blend, bitumen and CR. The main factors responsible for these changes were; proportion of the primary components, CR size, base bitumen grade and volatilisation during production. As a result, a maximum increase of up to 0.42% and 3.9% was observed in the densities of CRMB and bitumen residue respectively. However, a significant reduction of 19% in the densities of the CR phase was observed.**

Keyworkd - Bitumen, Crumb Rubber (CR), Crumb Rubber Modified Bitumen (CRMB), Density, Aging.

1.1 Introduction

A range of polymers has been used in the recent past to enhance the mechanical and rheological properties of the virgin bitumen to meet increased traffic density. Along with other polymers, bitumen has been modified with CR obtained from scrap tyres over the last four decades and has resulted in enhanced properties of the virgin binder [2], [13], [18], [21]. However, the nature of reaction between the primary components has been reported to be different compared to the other polymer modification. This is due to the vulcanised nature of the modifier, which resists the modifier from being completely dissolved into bitumen phase like other polymers. This also results in an inconsistent and heterogeneous blend of primary components with significantly different nature and properties [9], [17].

Despite the published work and experienced advantages of bitumen modified by reclaimed waste tyre rubber, the product is still not widely accepted; the reason being inadequate and inconsistent observations from different researchers and agencies around the world [1], [4], [10], [12], [15]. In addition, limitations such as, lack of chemical reaction during the production and poor storage stability during extended storage are added causes of concern [14], [16]. Therefore, the results presented are always debatable and not broadly accepted. This has also led to a gap, where industry and the research community are still trying to understand and control the reaction mechanism between CR and bitumen to obtain consistent results.

1.2 Materials and Experimental

1.2.1 Materials

The base bitumen 40/60, 70/100 and 100/150 were modified with three different CR sizes and percentages of Ambient CR. The physical properties of base bitumen are presented in Table 1.

TABLE 1 Physical Properties of Base Bitumen

Bitumen Grade	Penetration (dmm)	Softening Point (°C)	Viscosity at 177°C (Pa.s)	Density (g/cc)
40/60	50.5	51.1	0.079	1.0329
70/100	83.2	46.2	0.067	1.0321
100/150	121.5	43.5	0.055	1.0298

Additionally physical characteristics and gradation curves for the different sizes of CR sizes are presented in Table 2 and Fig 1 respectively.

TABLE 2 Physical Properties of Crumb Rubber (CR)

Source	Tyre Type	Processing Technique	Impurities (%)
S. R. C. Ltd. United Kingdom	Truck	Ambient Grinding	0.4 - 1.0

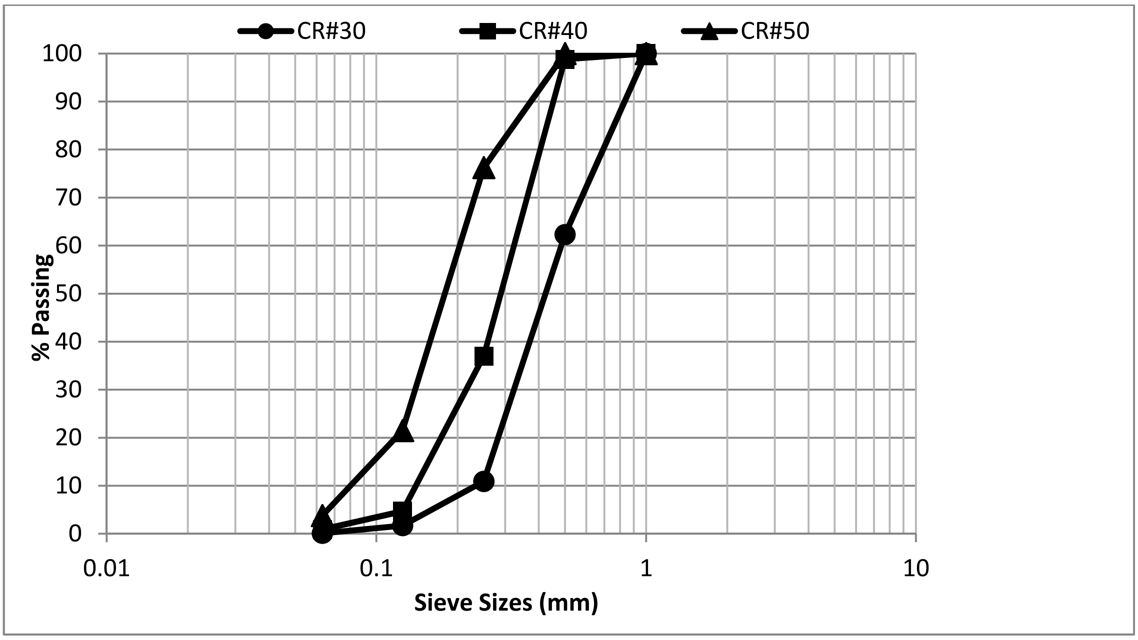

Fig 1 Particle Size Distribution of CR

1.3 CRMB Production and Characterisation

CRMB is mainly assessed by means of the viscosity measurement at certain blending durations to observe the consistency of the blends. Efforts have also been taken to understand the interaction phenomenon by different means such as a diffusion mechanism, swelling characteristics and dimensional changes by different ways. To measure the changes in the properties of basic constituents, researchers have developed several useful methods to separate CR and bitumen after the interaction for further laboratory investigation [1], [7], [11], [19]. However, little effort has been made to understand the interactions between the primary constituents by means of volumetric alterations during the production process. Therefore, an attempt to understand the outcome of the interaction as a function of changes in the densities of the basic components has been made in this part of the research study. In this regard, the laboratory investigation of the changes in the densities of the basic components before and after interaction was carried out using guidance of published work and laboratory experiences. The detailed laboratory production and assessment phase is illustrated in Fig 2.

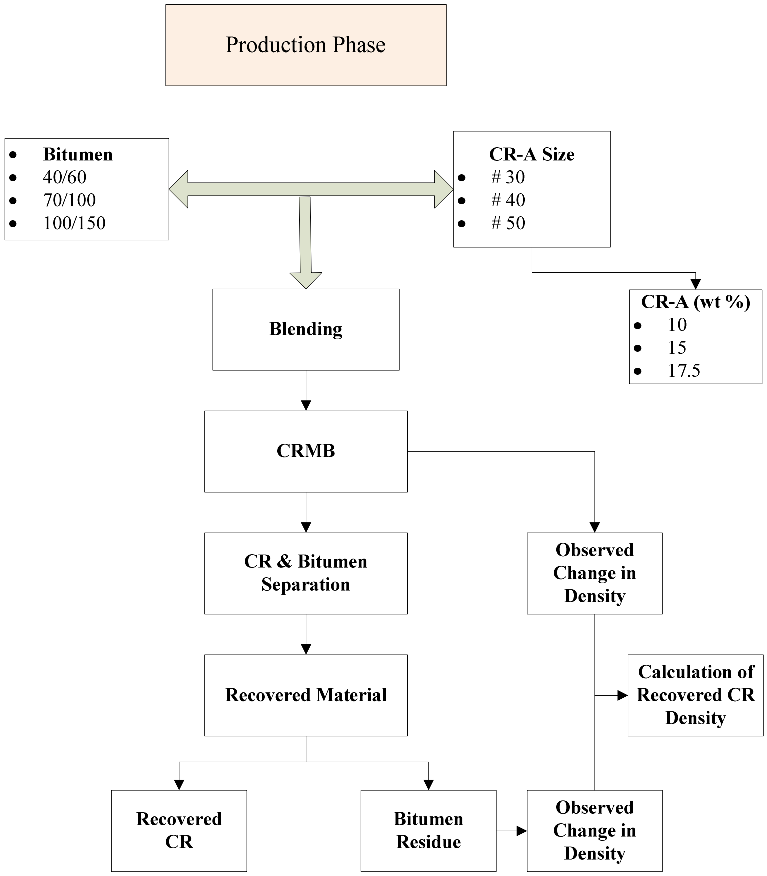

Fig 2 Research methodology to investigate volumetric during production CRMB

1.4 Investigation of Changes in the Densities During the Production Phase

In general, the CR particles used in the wet process are finer and are unlikely to separate from each other after the reaction due to their soft and sticky nature after swelling. To overcome the problem, researchers have used larger CR particles to observe the physical changes in the modifier during interaction with bitumen by means of change in mass and volume [8]. However, results obtained based on coarse particles investigated instead of finer particles are always debatable. This is mainly due to the initial differences in the physical properties (density and surface area) and resultant characteristics (swelling and degradation) of the CR.

1.4.1 Separation of Bitumen and CR after Interaction

From the introduced techniques, the most common methods to separate the primary components are basket drainage [1] and centrifuging at elevated temperature [11], [19]. Based on the limitations exhibited by the two laboratory techniques such as particles size and elevated temperatures, separation of the binder and finer CR after blending was performed by modifying and merging the traditional techniques in this research study. The process of the physical separation of bitumen and CR after interaction was exercised in four different stages; dilution, sieving, centrifuging and distillation.

1.4.2 Dilution of CRMB

The selection of the diluting agent was based on the literature review and laboratory experience. Different diluting agents such as trichloroethylene, DCM and toluene have been used in the past by researchers [6]. However, toluene as a diluting agent was used in this laboratory exercise because it is the most economical and comparatively less harmful compared to the other agents. Moreover, to investigate the effect of toluene, CR particles at different sizes and base bitumen were separately conditioned in the same manner with toluene as they were during the actual laboratory assessment of CRMB. After the condensation and evaporation of the solvent respectively, constituents were subjected to measurement of mass and density to observe the changes in parameters. This revealed no significant difference in the tested properties of the CR and base bitumen. Based on these initial observations, toluene was confirmed as a diluting agent in this study.

1.4.3 Filtration

After dilution of the CRMB samples collected at different intervals during blending, filtration of the binder with solvent was the next step to be performed. Based on the CR gradation, #200 (0.075 mm) sieve was selected to filter most of the CR particles at first stage more easily.

1.4.4 Centrifuging

In general, centrifuging is a process used to separate or concentrate materials suspended in a liquid medium. It is a highly accelerated form of sedimentation, which uses gravity to separate particles heavier than the liquid medium. Centrifugal force accelerates this settling rate in an instrument called a centrifuge. The centrifuge spins the material at high rotation speeds and uses centrifugal force to separate the particulate from the liquid. The centrifugal force can reach many thousand times that of gravity, quickly separating the liquid/solid material [19].

CR particles finer than #200 (0.075 mm) size could have easily passed through due to the sieve size selected for the initial filtration. It was also found impossible to separate those finer CR particles from bitumen with conventional techniques. Therefore, centrifuge equipment was used to separate the two constituents and get pure bitumen without any CR particles for further laboratory investigation.

1.5 Investigation Protocol

The process adopted to investigate CRMB samples at specified blending intervals followed by the recovery and investigation of CR and bitumen residue was carried out in the following order.

- Collection of 5 ± 0.5g of CRMB samples in a density pycnometer after specified blending intervals
- Observation of the density according to the BSEN ISO 3838:2004 standard in the laboratory
- Introduction of the toluene by at least 10 times the amount of CRMB sample in an open headed glass jar at room temperature.
- Submerging of density pycnometer containing CRMB sample into the glass jar filled with toluene
- A maximum of 3 hours were allowed for CRMB sample to be completely dissolved into the suspension to separate its constituents as recovered binder and CR.
- Further washing of CR particles until the resultant solution is free from bitumen (The task was accomplished by emptying the beaker over a No. 200 (0.075 mm sieve. The rubber particles were then removed and placed in another beaker containing toluene and allowed to soak for additional 30 minutes. The particles were removed and washed in the same manner to remove all of the binder from the rubber particles)
- Removal of fine CR particles which might have passed through 0.075 mm sieve (recovered bitumen was further centrifuged at 3000 rpm and 2000 G at room temperature. The resultant solutions were filtered carefully to separate the remaining CR particles and obtain pure recovered binder for further testing).
- Further processing of the diluted recovered binder through evaporation and distillation to obtain pure bitumen without any traces of toluene

1.6 Investigation Criteria

CR after the interaction with bitumen at elevated temperatures becomes soft and sticky. Therefore, direct assessment of changes in the densities of the recovered CR becomes almost impossible. Therefore, indirect measurement of the changes in the CR phase was obtained from the measured values of CRMB blend and bitumen residue. The two phases were subjected to the density measurements according to the BSEN ISO 3838:2004 standard. The calculation of the densities of the CR phase from the known values of the densities of the blends and bitumen residues was performed by using the following Equation (1). Furthermore, samples were investigated at different blending intervals to observe the rate and extent of the changes compared to the theoretical calculations.

$$RCR_d = \frac{CRMB_d - x(RB_d)}{y} \quad \text{(Equation 1)}$$

Where,

RCR_d	=	Density of CR phase
$CRMB_d$	=	Density of CRMB blend
RB_d	=	Density of bitumen residue
x	=	Bitumen proportion in the blend (%)
y	=	CR proportion in the blend (%)

1.7 Results and Discussions

1.7.1 Changes in the Densities of the CRMB Blends

After the initial characterization of the primary components for changes in their densities during conditioning state, CRMB blends with varied proportions of bitumen and CR were prepared. CRMB blends were then subjected to changes in the densities at equilibrium stages. The results are presented in Fig 3 to 5. The terms 'T' and 'M' in the Figs represent theoretical and measured densities of the blends respectively. The theoretical densities are calculated from the initial densities of the material constituents using Equation (1).

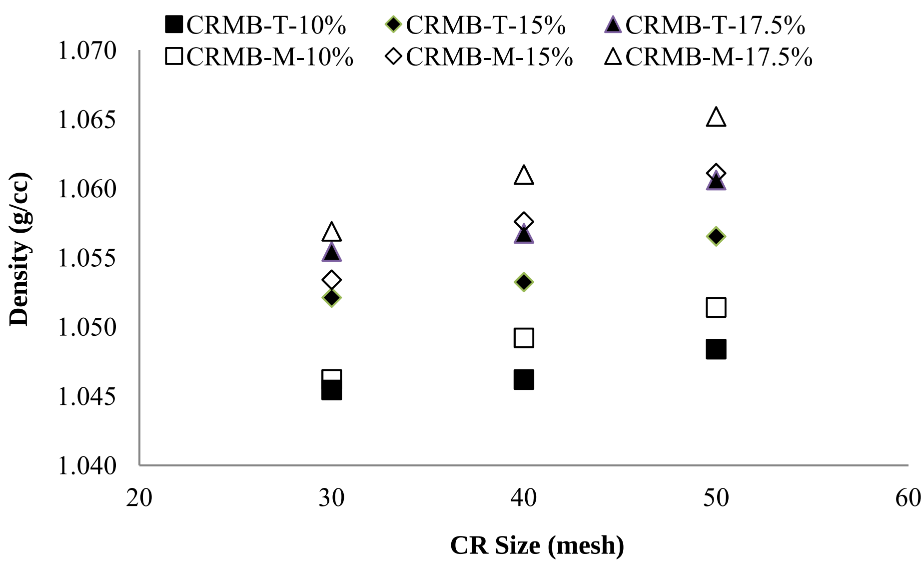

Fig 3 Densities of the 90:10, 85:15 and 82.5:17.5 Bitumen-CR Proportional blend Prepared with 40/60 Base Bitumen

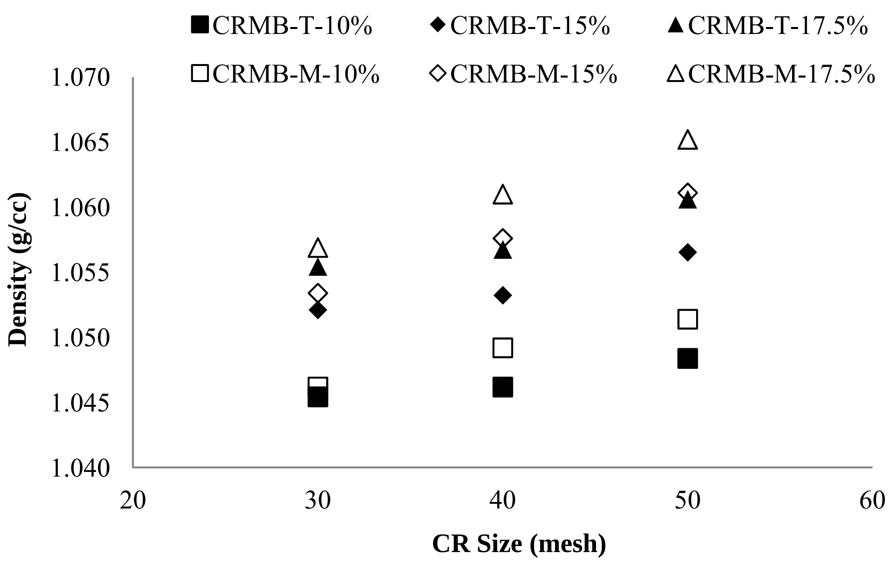

Fig 4 Densities of the 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend prepared with 70/100 base bitumen

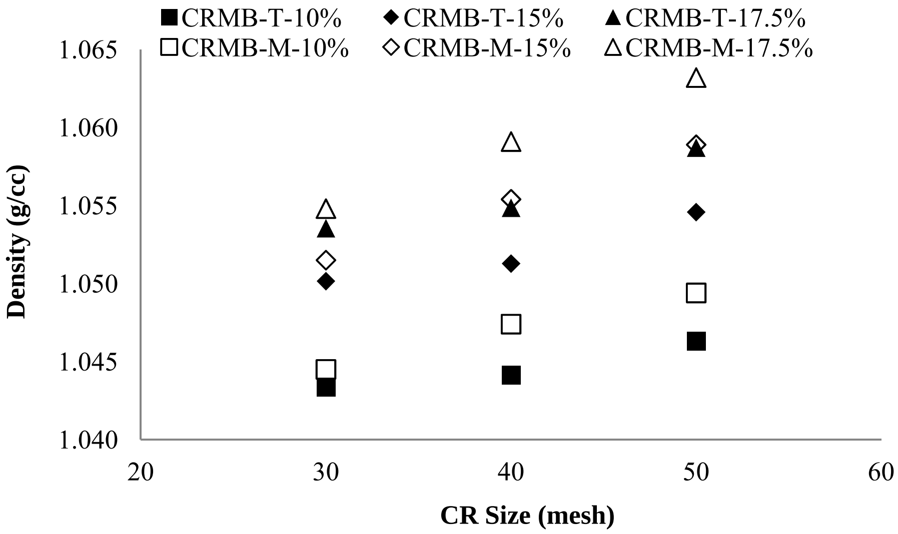

Fig 5 Densities of 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend prepared with 100/150 base bitumen

Furthermore, to get more understanding of the changes in the densities of the CRMB blends at equilibrium states, variation in the densities by means of the changes in the percentages compared to the theoretical values are tabulated in Table 3.

TABLE 3 Percentage variation in the densities of the CRMB blends

Base Bitumen / CR (mesh)	CRMB 10%			CRMB 15%			CRMB 17.5%		
	40/60	70/100	100/150	40/60	70/100	100/150	40/60	70/100	100/150
	Changes in densities of CRMB blends (%)								
30	0.02	0.07	0.11	0.04	0.12	0.13	0.09	0.14	0.12
40	0.15	0.29	0.31	0.20	0.41	0.39	0.24	0.40	0.40
50	0.16	0.29	0.29	0.21	0.43	0.41	0.24	0.43	0.42

In general, when comparing the densities of the blends after specified blending times with the theoretical densities before blending, increasing trends in the densities were observed for all blend combinations. CR obtained from scrap tyre contains moisture (Thodesen et al. 2009), extender oils, sulphur and carbon black (ETRA 2004). Some of these components may dissolve in bitumen phase, when exposed to heat during blending process, which may also contribute to the increase in the densities of the blends. This is also evident from the observations presented in Figs 5-4 to 5-6, where an increasing density trend of the blends with an increase in the CR concentration and decreasing CR size. By doing so, more components that are soluble were exposed, while increasing the surface area of the material thus increased density of the blends. As a result, CRMB blends produced with 40 and 50 mesh CR sizes displayed a significant increase in overall density compared to theoretical values. However, with the introduction of CR size to 30 mesh in the CRMB blend production, there was no significant change in the density of the blend, when compared to the original values. The reported observations were true for all the bitumen-CR proportions. The differences in the variations of the densities of the blends are also compared in Table 3.

1.7.2 Changes in the Densities of Bitumen Residue

The comparison of the observed densities of bitumen residue at the equilibrium state is presented in Figs 6 to 8. The changes in the densities are compared as a function of base bitumen, CR content and CR size. The term 'R-B' in the Figs represents the recovered bitumen residues from CRMB blends.

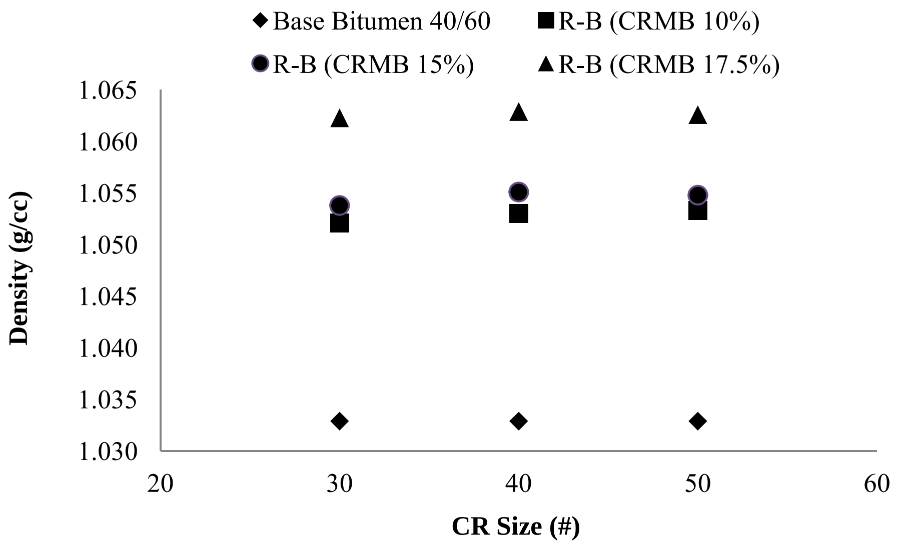

Fig 6 Densities of recovered bitumen residue 40/60 from 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend

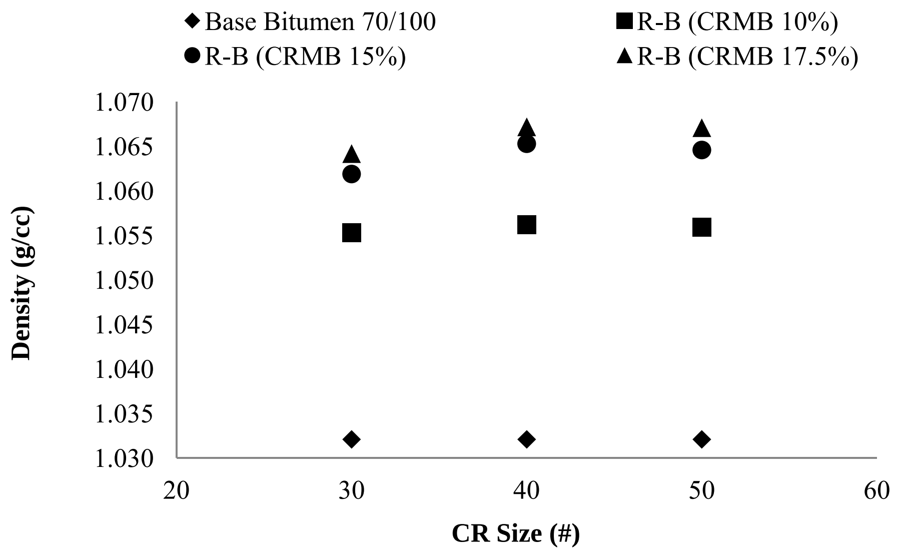

Fig 7 Densities of recovered bitumen residue 70/100 from 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend

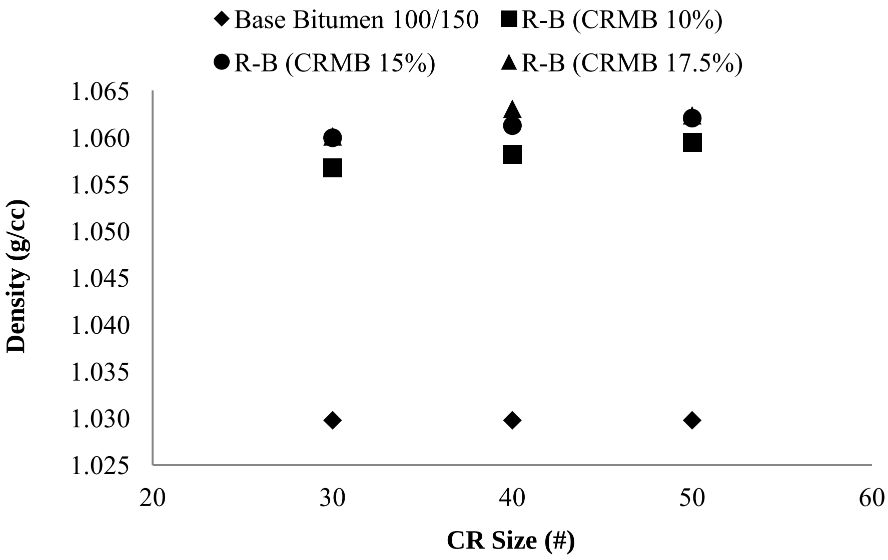

Fig 8 Densities of recovered bitumen residue 100/150 from 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend

In addition, variation in the densities of bitumen residues after the separation from the different proportional CRMB blends at equilibrium states by means of the changes in the percentages compared to the theoretical values are tabulated in Table 4.

TABLE 4 Percentage variation in the densities of bitumen residues

Base Bitumen / CR (mesh)	**CRMB 10%**			**CRMB 15%**			**CRMB 17.5%**		
	40/60	70/100	100/150	40/60	70/100	100/150	40/60	70/100	100/150
	Variation in the densities of bitumen residues (%)								
30	1.9	2.2	2.6	2.0	2.7	3.0	2.2	3.1	3.4
40	1.9	2.4	2.8	2.2	3.1	3.5	2.2	3.4	3.6
50	2.0	2.5	2.9	2.2	3.1	3.6	2.2	3.4	3.6

When CR and bitumen are interacted together at elevated temperatures, bitumen residue gets stiffer and denser after losing lighter fractions to the CR. The process is said to be diffusion of lighter fraction from bitumen to the rubber network. However, the rate and extent of the diffusion is mainly dependant on the nature of base bitumen (Airey et al. 2003; Artamendi and Khalid 2006). In addition to the diffusion of the lighter fraction from bitumen to CR, volatilisation of bitumen at an elevated temperature and dissolution of impurities from CR phase in bitumen may also affect the density of the residual bitumen. Based on the actual observations presented in Figs 6 to 8 and further summarised in Table 4 for the percentage variation in the theoretical and measured values, above mentioned parameters are further discussed in the following sections.

When analysed for the effect of diffusion on the densities of recovered bitumens, densities of bitumen residues were found increasing to different extents irrespective of the variation in material constituents. However, percentage increase was found dependant on the amount of lighter fraction in the base bitumen during the production stage. As a result, base bitumen 40/60 the hardest binder among the selection has shown relatively small increases in the density of bitumen residue compared to the two softer bitumen grades. However, there were no significant differences observed in the densities of the 70/100 and 100/150 recovered binder residues. This could be due to the limited amount of lighter fractions in the 40/60 base bitumen compared to the other two types of base bitumen, which is absorbed by the CR particles and leaves bitumen phase with a higher maximum density.

Volatilisation / oxidation have also been counted as factors to increase the density of bitumen phase, when interacted with CR at elevated temperatures. To see the effect of oxidation, three base binder were separately conditioned to the actual blending conditions without CR. This resulted in an overall increased density of bitumen with some variation (see Fig 5-3). The same phenomenon would have occurred during the blend production and thus contributed to the increase in the overall density of the blend. However, the impact of oxidation could have been lowered in the presence of CR due to the increased viscosity of the blend.

Besides the impact of bitumen composition, the total amount of absorption may also have been controlled by the CR surface area in the blend. The surface area of the CR particles increases with the increase in CR concentration and decrease in size. This could also led to an increase in the density of bitumen residue due to the increased requirement of the lighter fraction for the higher surface area of the CR particles. From the comparison presented in the Table 4, the highest variation in the density of the recovered bitumen was resulted, when blends were produced with 15 and 17.5% CR concentration. In addition, the CR size was also found to be an influential factor to affect the density of the binder phase in the blend. In this regard, CRMB blends produced with fine CR particles, such as 40 and 50 mesh sizes, resulted in more dense recovered binder compared to those recovered from the blends produced with 30 mesh CR size.

In addition, CR contains components such as carbon black and extender oils (ETRA 2004), which may easily dissolve into bitumen at higher temperatures and affect the density of bitumen phase in the blend . From the analysis of results, the density of bitumen recovered from the CRMB may also have affected by the dissolution of some components from the CR phase. An increasing density trend of the recovered bitumen was found and this was directly proportional to the CR concentration and inversely proportional to the size of the CR particles. Therefore, higher the number of CR particles, higher would be the percentage of components to be dissolved in bitumen phase. The dissolution of such components has led to an ultimate increase in the density of bitumen phase.

1.7.3 Changes in the Densities of the CR

The calculated changes in the densities of the recovered CR at the equilibrium state during the blending of CR and bitumen are presented in Figs 5-9 to 11, where the changes are compared as a function of bitumen type, CR content and CR size. The calculation were based on the Equation 1. The term 'R-CR' in Figs represents the recovered CR phases in CRMB blends.

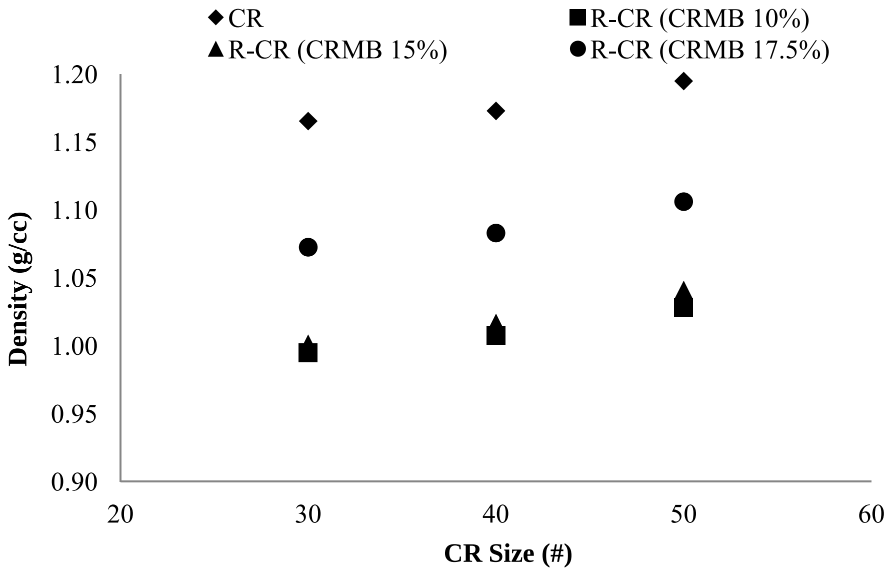

Fig 9 Densities of recovered CR from 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend prepared with base bitumen 40/60

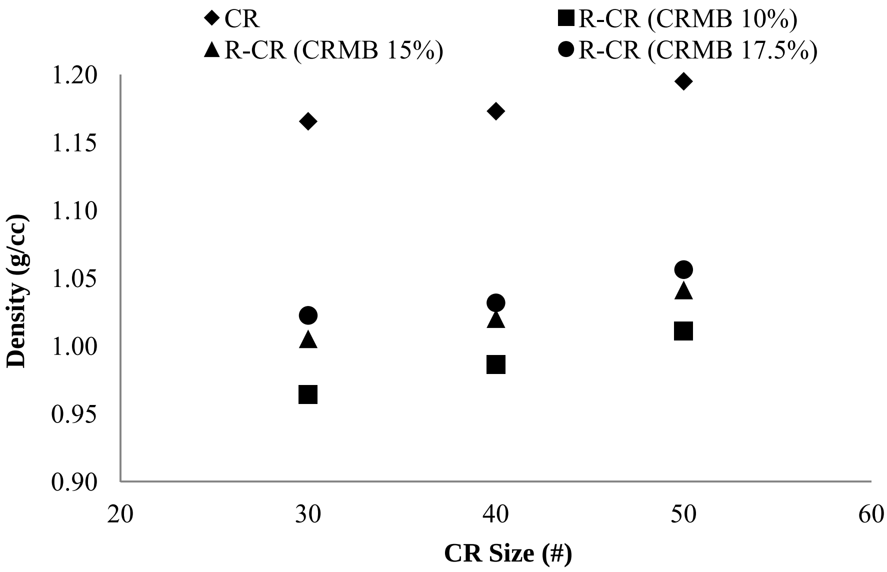

Fig 10 D Densities of recovered CR from 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blends prepared with base bitumen 70/100

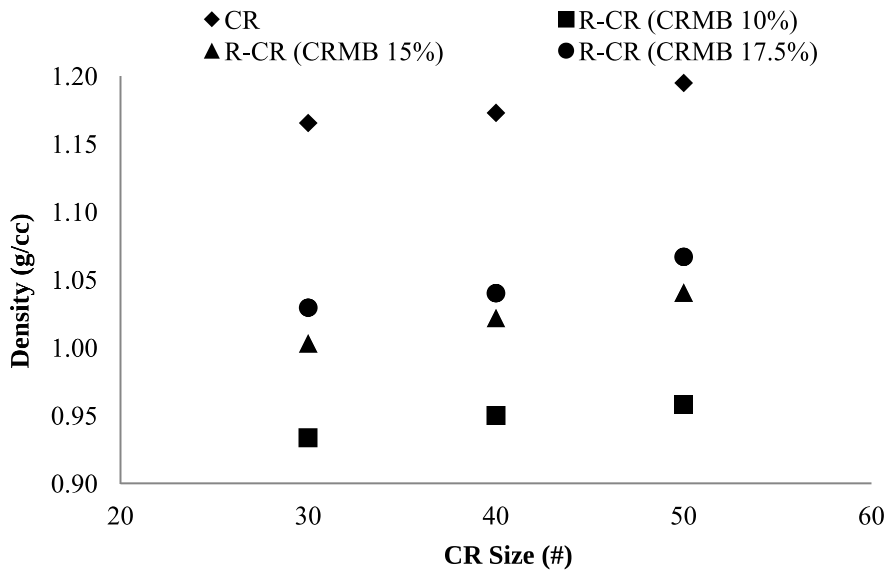

Fig 11 Densities of recovered CR from 90:10, 85:15 and 82.5:17.5 bitumen-CR proportional blend prepared with base bitumen 100/150

In addition, variation in the calculated densities of the CR after separation from the CRMB blends at equilibrium states by means of the changes in the percentages compared to the theoretical values are tabulated in Table 5.

Table 5 Percentage variation in the densities of the CR

Base Bitumen / CR (mesh)	CRMB 10%			CRMB 15%			CRMB 17.5%		
	40/60	70/100	100/150	40/60	70/100	100/150	40/60	70/100	100/150
	Changes in densities of CR phase (%)								
30	-15	-17	-20	-14	-16	-18	-8	-13	-16
40	-14	-16	-19	-13	-15	-18	-8	-13	-16
50	-14	-15	-19	-13	-15	-17	-7	-12	-15

When interacted with bitumen at an elevated temperature, CR known to swell as a result of volume increase. This also leads to a reduction in the density of the modifier, where the amount of variation mainly depends on the extent of swelling of the CR particles. In addition, CR concentration and size may also affect the extent of CR density reduction. When CR is increased in proportion and reduced in size during the production of the blends, an increased lighter fraction may be required by the increased surface area. If provided, highest degrees of reduction in the density of CR phase in the CRMB blend could be observed.

To understand the phenomenon, the calculated results are presented in Figs 9 to 11. Moreover, the variation in the theoretical and the calculated values is presented in Table 5. From the results analysis, recovered CR displayed a significant decrease in the density after the interaction with the bitumen. CR at the 10% concentration with three sizes introduced in base bitumen 40/60, resulted in a 14 to 15% reduction in the density of the modifier at equilibrium state. However, further decrease in the density was observed, when CR at above concentration was interacted with base bitumen 70/100 and 100/150. As a result, the reduction in overall densities of the CR phase reached 15-17% and 19-20%, when recovered from CRMB 70/100 and 100/150 respectively. At this concentration level, the CR size was not observed to be a significant factor to alter the density of the modifier, when interacted with three base bitumen.

When analyzed for the CR introduced at a proportion of 15% with the three sizes, similar trend was observed as with 10% CR concentration. An increasing trend in the reduction of the recovered CR phase was observed with increase in the penetration of the base bitumen. However, lower surface area of the CR particles yielded higher reduction in the density of the recovered phase. As a result, 30 mesh CR observed a relatively higher reduction compared to the other two sizes. The overall reduction in calculated densities of the CR phase after interaction with 40/60, 70/100 and 100/150 at equilibrium state were observed by 13-14, 15-16 and 17-18% respectively.

When CRMB blends were produced with 82.5:17.5 bitumen-CR proportions, a significant variation was observed in the densities of the CR phase. The change was mainly attributed to the CR added with three different sizes. The increased CR content was not found to be compatible with base bitumen 40/60. The combination resulted in the lowest reduction of an approximately 7-8% in the CR density for the three different sizes of the observed in the blends. In contrast, 70/100 and 100/150 base bitumens accommodated increased CR content. The observations were mainly based on the further decrease in the observed overall densities of the CR phase to 12-13 and 15-16%, when observed in CRMB 70/100 and 100/150. The results were true for the three different CR sizes observed in above blends.

1.8 Conclusion and Recommendations

Based on the conclusions derived from this study, CR obtained from scrap tyres cannot be considered as a waste material. It is a valuable commodity, which has proven to be a beneficial additive to virgin bitumen and ultimately to the bituminous mixtures. In other words, the addition of CR in bitumen, results in favourable changes in the mechanical and performance properties of the base bitumen. However, optimisation and the stability of the product at different stages were found to be the main causes of concern. This research study was carried out particularly to address these issues by investigating the interaction of the two material constituents at different stages of production and storage. The conclusions drawn from the observations of the different parameters studied are presented below.

1.8.1 Changes in Volumetric of Material Constituents during Production

Following the production of CRMB blends, the outcome of the interaction of the two basic components as a function of volumetric changes was studied. The task was accomplished by modifying and merging separation techniques used in the past. The new method was found to be quite useful to analyze the changes in the densities of the basic components during the production.

- Increasing trends of the overall density of the CRMB blends were observed for most of the combinations, when comparing the densities of the blends after specified blending times with the theoretical densities before blending. The change was mainly attributed to the presence of components in the CR such as, moisture, extender oils and carbon black and their ultimate dissolution into bitumen phase during blending process. This has affected the density of the blend showing an increasing trend with an increase in the CR percentage and decreasing CR size. This change was attributed to a increased soluble components exposed in the CR, while increasing the surface area of the material.
- The densities of bitumen residues also increased to different extents irrespective of the variation in material constituents. The intensity of the variation has resulted from diffusion, volatilisation and dissolution of some components after the reaction with CR at elevated temperatures for certain durations.
- CR concentration at different percentages in the blend with three different size fractions resulted in significant variation in the overall densities of the CR phase during the interaction process. CRMB blends produced with softer binders and lower CR sizes resulted in improved compatibility by allowing the maximum reduction in the densities of the CR phase in the blend.

1.9 Acknowledgement

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