Investigation of Hot Storage Stability of CRMB using X-Ray Computed Tomography

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Abstract -- **The use of CR in the bitumen industry has significantly evolved in last few decades but has not quite been able to capture the market like other polymers. Despite a reasonable amount of work done in the field of bitumen modification, when CR is used as a bitumen modifier, there are still some areas that are mainly based on assumptions and not completely addressed and validated. One such is the phase separation of the primary components during extended storage of CRMB blends. Due to the different densities of the constituents, inadequate dispersion of CR particles into the bitumen and lack of chemical reaction a significant phase separation of the constituents has been reported in past. Is this study, CRMB blends produced with varied combinations of material constituents were investigated for storage stability. However, evaluation of storage stability and ultimate phase separation of the modified binder using softening point observation may be inadequate. Therefore, observations were mainly based on the differences in visual and analytical observations of top and bottom sections of the storage tubes using X-Ray computed tomography technique. The properties were the softening point results of the similar specimens. Based on the observations, the phase separation was found to be mainly dependant on the density and dispersion of the CR particles in the blend after different storage intervals. On the other hand, CR concentration in the blend has played a key role. Therefore, the variation in the storage stability was also attributed to increased inter-particle distance. The higher the CR concentration, the lower would be the difference in the storage properties of the top and bottom sections. The improved property was related to the reduced movement of the CR particles in CRMB blends with higher CR concentration such as 17.5%. This also resulted in the restriction to excessive settling or floating of CR particles, irrespective of the significant differences in the densities of the two phases. However, the idea of increased CR concentration in the CRMB blend to achieve high storage stability may not be useful due to a poor workability of the blend, while pumping and mixing with aggregates.**

Keywords - Bitumen, Crumb Rubber (CR), Crumb Rubber Modified Bitumen (CRMB), Density, X-Ray Computed Tomography (X-Ray CT) and softening point.

1.1 Introduction

Bitumen and CR are significantly different in nature and properties. When the two interact with each other, the reaction between the two is assumed to be non-chemical in nature unlike most of the other polymer modified binders [9]. This reaction is rather reported as a diffusion process, where bitumen residue loses its aromatic components, becomes stiffer which may lead to altered properties compared to the unmodified binder [1], [3], [18] On the other hand, CR after absorbing lighter fractions from the bitumen phase results in swelled CR particles [6]. The alteration in the properties of bitumen residue and CR may lead to further differences in their properties. This may also result in significant phase separation when the two components are not chemically bonded during the production stage.

1.2 Phase Separation during Hot Storage

The storage stability has been recognized as an important criterion of production and usage of CRMB. The presence of non-dissolved CR particles in bitumen and a poor compatibility between the two has led to significant phase separation, when CRMB blends are stored at high temperatures [2], [5], [8], [14]. As a result, the swollen CR particles are considered to settle down quickly due to an initial higher density than the bitumen phase [14]. On the other hand, migration of the CR particles to the top of the storage container due to a reduced density after swelling has also been reported in some research studies [8], [11], [16], [17], [19]. Despite the differences in the two approaches, the modified binder leads to an unstable condition in both cases and results in varied properties in different regions of the modified binder. In addition, typical designs of storage tanks used in the field indicate that CRMB is stored with continuous agitation to obtain uniform temperature and homogeneity in the material. However, this has also resulted in a significant phase separation of the primary components and an ultimate increase in overall cost. Therefore, an improvement in the storage stability would result in a significant cost reduction and wider acceptance of the material in the bitumen industry.

1.3 Storage Stability Investigation Techniques

During storage stability, ultimate phase separation of the modified binder is generally assessed with conventional techniques such as penetration and the softening point. The difference in the observations of top and bottom section is then used to understand the storage stability of the binder. However these techniques may be inadequate due to the amount and nature of testing [10]. Therefore, researchers have also performed Polymer Dispersion Analysis (PDA) using fluorescence microscopy to investigate the dispersion of the un-dissolved polymer phase in bitumen after the storage at certain durations [7], [17]. In this process, a small amount of representative sample is collected from the top and bottom thirds of the tube to capture images at certain magnifications using fluorescence microscopy. The objective is to observe the presence of polymer in the bitumen matrix after different storage periods. However, the results presented may also not be a true representation of the dispersion of CR particle in the blend due to there being a limited amount of the material observed during the test. Furthermore, the nature of the method is understood to be destructive, where actual dispersion of the CR phase in the bitumen matrix may be disturbed due the nature of testing. Therefore, to get a proper understanding of the phase separation during hot storage, a non-destructive technique needs to be adopted to visualize and analyze the true dispersion of the CR particles in the blend such as X-Ray CT imaging.

1.4 Functionality of X-Ray computed tomography

X-Ray Computed Tomography is a modern and powerful technique for non-destructive testing by visualizing inner characteristics within a solid object. In other words, this technique is used for digitally slicing a specimen using X-Rays. The specimen is scanned by X-Rays at the desired location resulting in a series of linear attenuation coefficients and, ultimately rebuilding of a cross-sectional image of the specimen. An obtained CT image is called a slice, which represents what the object being scanned would look like if it were sliced open along a plane. The gray levels in a CT slice image correspond to X-Ray attenuation, which reflects the proportion of X-Rays scattered or absorbed as they pass through each volume element. X-Ray attenuation is primarily a function of X-Ray energy based on the density and composition of the material being imaged.

X-Rays are electromagnetic radiation and are classified by their energy as opposed to their frequency or wavelength. X-Rays are generated in a hot cathode ray tube composed of a glass envelope containing a vacuum. A Tungsten filament creates a current, which is passed through a tungsten filament cathode to emit electrons. The large potential difference between the anode and cathode causes acceleration of the electrons towards the anode where a small amount of the electron energy is transformed into X-Rays, which then exit the vacuum. The rest of the energy is turned into heat, which is why a water-cooling system is used to prevent overheating. A Tungsten cathode is used due to its high melting point and good conductive properties. The schematic of the system is illustrated in Fig 1.

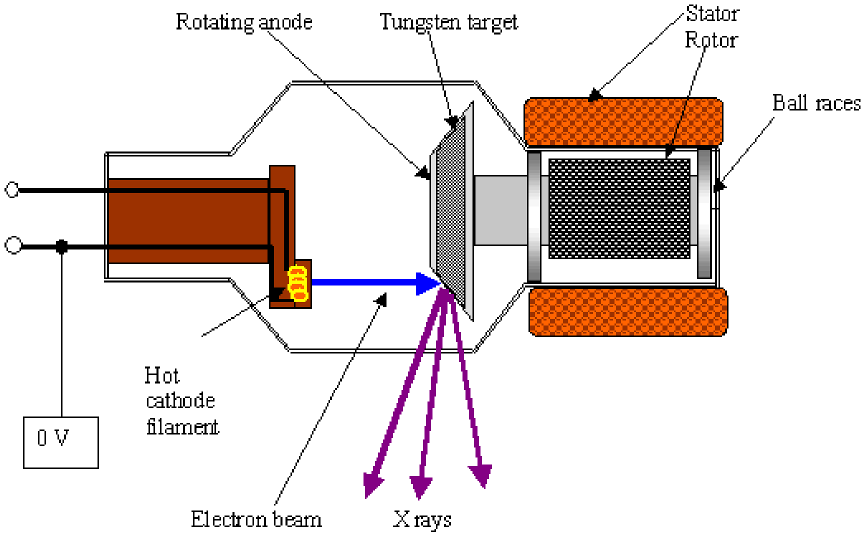

Fig 1 Schematic diagram of X-Ray computed tomography

1.5 Materials and Experimental

1.5.1 Materials

The base bitumen 40/60, 70/100 and 100/150 were modified with CR (40 mesh) and with three different variations in the 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 2 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

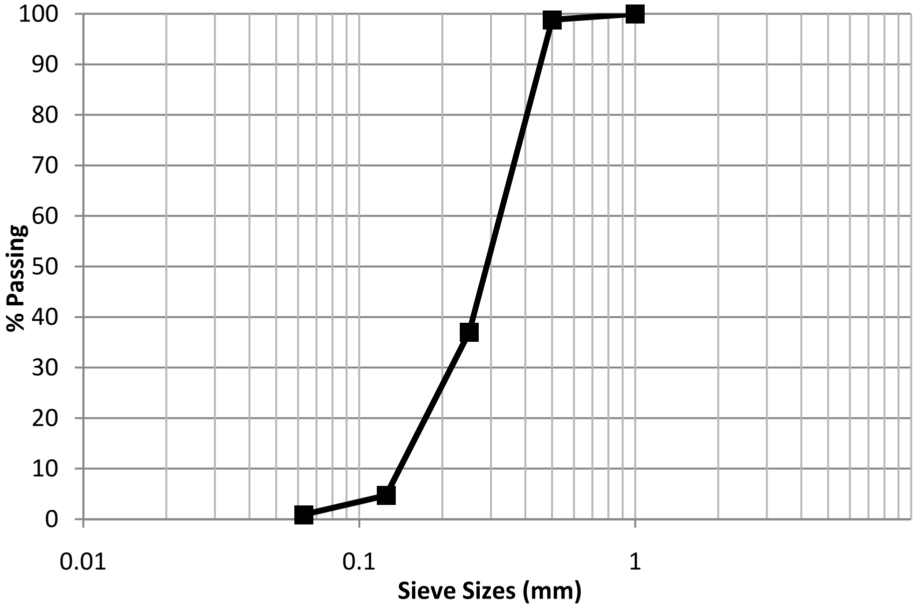

Fig 2 Particle Size Distribution of CR (40mesh)

1.6 Storage Stability Investigation of CRMB Blends

The properties of the CRMB blends are mainly governed by the presence of CR particles in the blend. In addition, factors such as stiffening of the bitumen phase during the production and extent of proper dispersion of the CR particles in the bitumen matrix may also alter the properties of the modified blend.

1.6.1 X-Ray Computed Tomography Image Observation

After different storage periods, tubes were then taken out from the oven and were cooled down to ambient (room) temperature before placing into a freezer at -20°C. The frozen samples were taken to the X-Ray CT machine to capture images at required sections of the storage tube. In this case, image slices from the top third and bottom third of the tube were of the interest. The objective was to understand and correlate softening point observations at these sections of the tube using polymer dispersion analysis. The frozen samples were then cut into thirds and the top and bottom sections saved for further analysis. Effect of the storage was analyzed by comparing the differences in properties of top and bottom third sections by means of X-Ray CT image and softening point analysis. To capture images using X-Ray machine, protocol is mentioned in following section.

- ***Parameter Set-Up***

The parameters directly relating to the level of penetration achieved are those of current, voltage, exposure time and the use of filters. The proper selection of these parameters was achieved with the laboratory experience and guidance of the equipment manual. The adopted parameters for optimal penetration and resolution for a 25mm diameter CRMB specimen are listed below;

Voltage:	310kV
Magnification	50x
Current:	2.1mA
Exposure:	90ms
Source Filter:	2mm Copper
Back Filter:	25mm Aluminium

- ***Image Processing***

A minimum of two slices through the specimen were received from the scanning procedure as 16 bit digital images. The Image J programme was used to separate bitumen and CR phases in the captured image based on the density and material properties.

- ***Thresholding of the Captured X-Ray Image***

The Image J programme was used as a tool to threshold and analyze the digital image. By this technique, specific ranges of pixel grayscales were isolated for analysis. The images used are 16-bit meaning that they have 65539 (2^{16}) different shades of grey. Each shade of grey from an X-Ray image represents a different density because it represents its specific attenuation properties. Different densities relate to different materials, which can therefore be isolated and quantified in the digital image by isolating its specific grayscale ranges through threshold. Examples of directly captured and threshold images are presented in Fig 3.

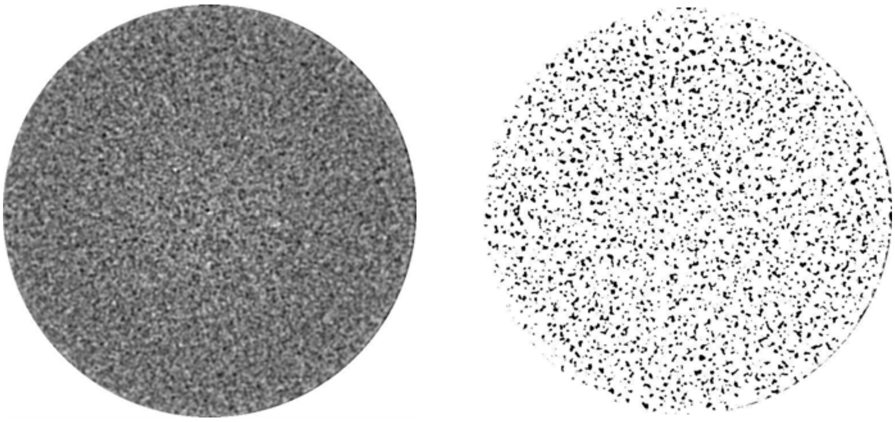

Fig 3 X-Ray CT image (left), threshold image for CR particles dispersion at 50x magnification (right)

1.6.2 Ring and Ball Softening Point Observation

Another fundamental requirement to evaluate the stored CRMB samples hot storage stability is softening point. Therefore, CRMB blends were further investigated in the laboratory for the variation in softening points of top and bottom section at different storage intervals using ring and ball softening point test based on the BSEN 13399 standard. To accomplish the task, aluminum toothpaste tubes were used to fill CRMB samples straight after production at 180°C. Filled tubes were then sealed and subjected to a hot storage stability test by placing them in a vertical orientation in the oven at 180°C. The assembling of the toothpaste tubes to be placed in a pre-heated oven to required temperature is illustrated in Fig 4. Moreover, the illustration of the adopted methodology is shown in Fig 5

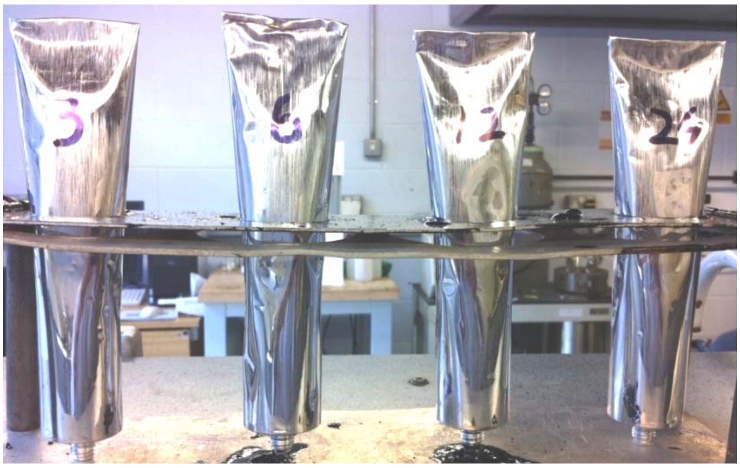

Fig 4 Arrangement of toothpaste tubes in vertical orientation

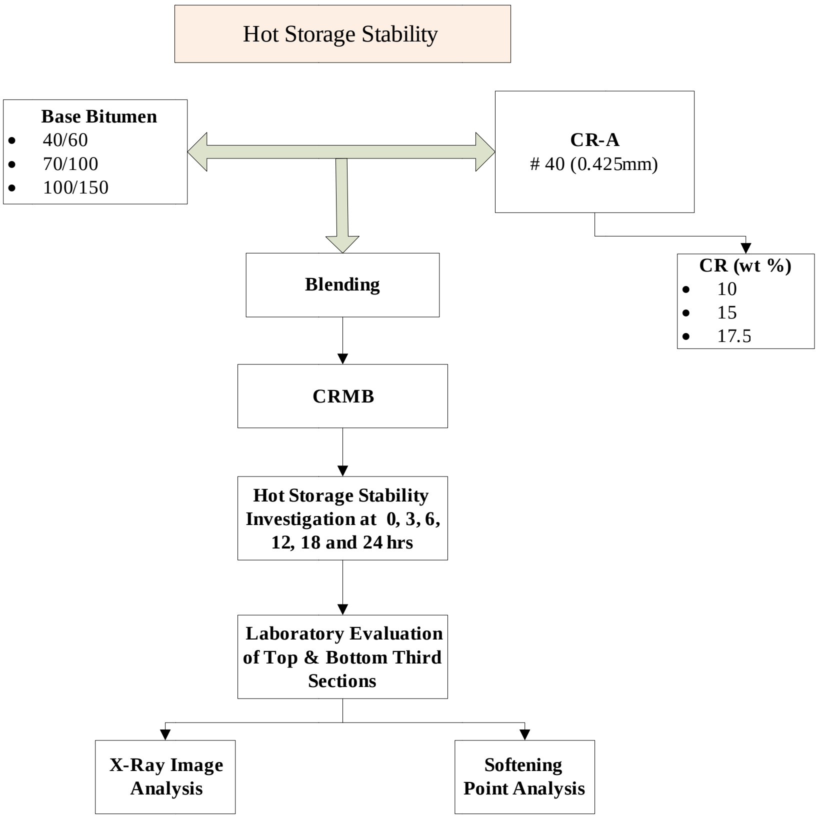

Fig 5 Methodology adopted to examine storage stability of CRMB blends

1.7 Results and Discussions

1.7.1 CR Dispersion after Storage Stability

Before carrying out the conventional investigation of the material by means of the ring and ball softening point test, un-disturbed frozen samples were examined using X-Ray imaging. From the observations, similar trends of polymer dispersion in the bitumen matrix were observed, when comparing the blends prepared with the three bitumen types. However, dispersion was found to be governed by the CR concentration and the particle densities. Therefore, X-Ray images of CRMB 70/100 blends with three CR concentrations (10, 15 and 17.5%) and one CR size (40 mesh) before and after storage are presented in Fig 6 to 8. The images are representative sections from the actual X-Ray images at 100x magnification to see the clear distribution of the CR particle sizes in different sections after hot storage.

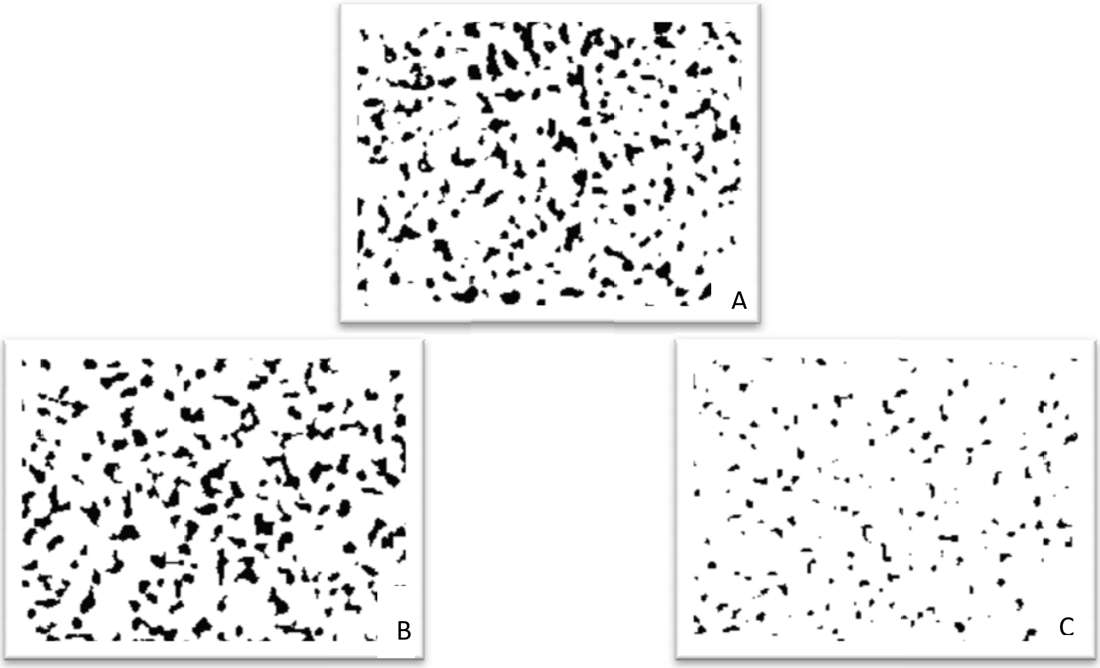

Fig 6 X-Ray CT images of A) CRMB 70/100 (90:10), B) top after storage and C) bottom after storage at 200x magnification

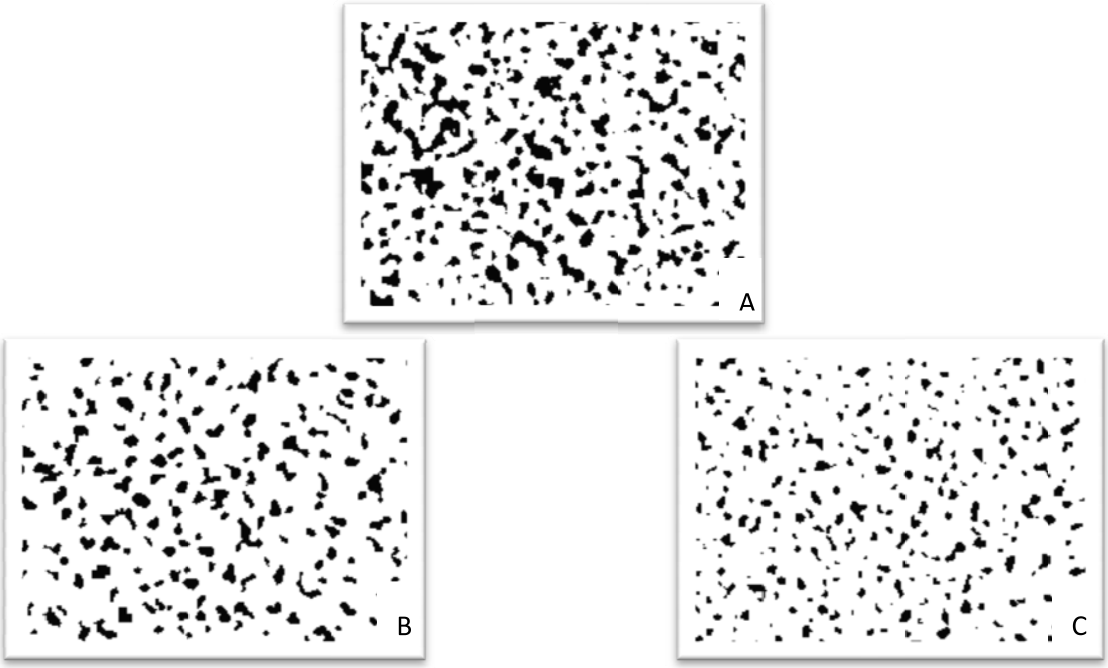

Fig 7 X-Ray CT images of A) CRMB 70/100 (85:15.5), B) top after storage and C) bottom after storage at 200x magnification

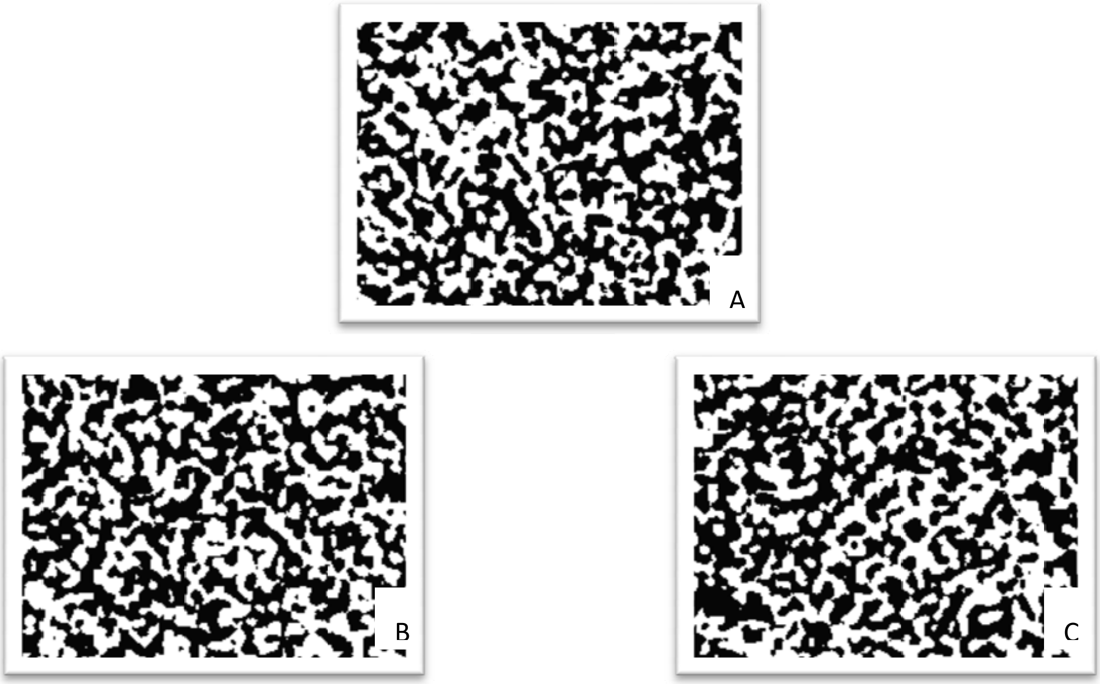

Fig 8 X-Ray CT images of A) CRMB 70/100 (82.5:17.5), B) top after storage and C) bottom after storage at 200x magnification

Fig 6 to 8 present the images obtained using the X-Ray machine at required locations of the specimen. These illustrate the dispersion of the CR particles in the bitumen matrix after the production phase and top and bottom sections after 24 hours of storage stability investigation. The X-Ray images indicate that the CR particles are evenly distributed in the bitumen phase straight after production. However, a significant phase separation of the two primary components after prolonged hot storage can be observed, when compared to the unconditioned sample. In general, the CR with a higher density (fine particles) compared to bitumen residue has settled in the bottom third and the CR particles having a lower density (coarse particles) had migrated to the top. In addition, the variation of CR concentration in the blend had also affected the migration of CR particles during the storage period.

CR and bitumen were examined separately for their physical properties before interacting with each other. Furthermore, interaction of CR with bitumen at elevated temperatures has resulted in an overall decrease in the density values, when compared to the initial values [13]. Based on the initial characterization of the CR before addition to bitumen and changes that occurred during the blending process, the overall CR phase has significantly changed its properties. However, there is definite need to understand the effect of various CR sizes with different densities present in a gradation on the overall distribution in the bitumen matrix after storage. Based on the initial density values of the CR, large particles with an initial density of 1.17 g/cc would have reached 0.99 g/cc approximately after a reduction of 16% during the production process. Similarly, fine material with an initial value of 1.37 g/cc would have resulted in 1.12 g/cc approximately at equilibrium stage with the same amount of reduction in density. The two extreme sizes in the gradation have resulted in lower and higher values compared to the bitumen phase respectively. As a result, a clear presence of fine and medium coarse particles can be seen in the bottom section. However, coarse CR particles have moved to the top of the storage tube due to lower density than the bitumen. The trend can be observed in Fig 6 to 8.

The observations were true, when polymer dispersion was assessed for the CRMB blends produced with 10 and 15% CR concentration. However, CRMB blends produced with 17.5% CR concentrations have revealed almost similar CR dispersion before and after storage. The improved dispersion could be related to the increased interlocking of the CR particles due to the higher number of the particles in the blends which were thus less prone to separation, which can be clearly seen in Fig 6-7.

Moreover, to examine the amount of CR particles present in the top and bottom, the two sections were assessed for the measurement of the areas of CR particles separately. The particle area measurement was performed using the original X-Ray images at 50 times magnification using Image J software. The obtained differences in the values were also useful in understanding the differences in the softening points of the two sections after storage. The calculated areas are presented in Fig 9.

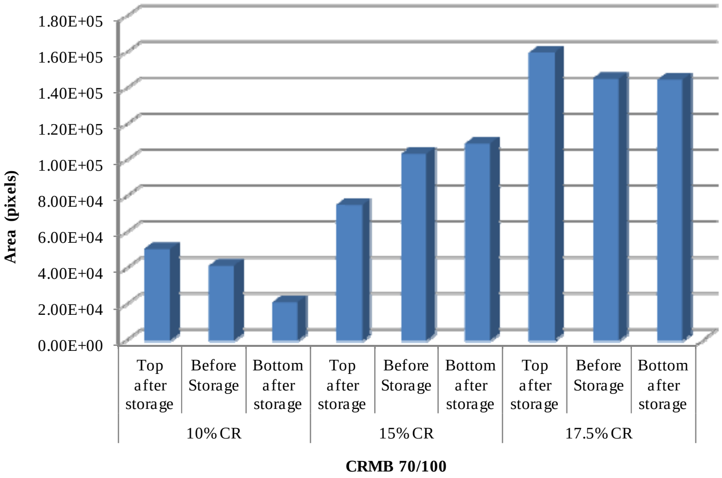

Fig 9 Area of CR particles in different sections of the storage tube before and after storage

Following the visual observations, the areas of CR particles were also calculated at required sections of the storage tubes before and after storage. The objective was to confirm the visual analysis of CR particles by analytical means. The area measurements of the CR particles in particular sections are presented in Fig 9. The results reveal clear differences in the areas of CR particles in the top and bottom sections after storage. In addition, an apparent variation can be observed between the values calculated before and after storage. The difference can clearly be attributed to the amount and size of CR particles in different sections.

Analyzing the CRMB blend with 10% CR concentration, the top section after storage has resulted in a higher area compared to the blend before storage and the bottom after storage. This also confirmed the visual particle distribution before and after storage, where the top section mostly comprised of higher numbers of coarse particles compared to the other two arrangements. Blends with 15% have shown reversed trends, where the bottom part has a higher area compared to the original state and the top after storage. This also represented the polymer dispersion in the three arrangements. However, no significant difference in the areas of the three states was observed, when blends with 17.5% CR concentration were assessed for particle's area measurements. The trend was attributed to the interlocking of CR particles, which resisted the free movement in the bitumen matrix and thus gave relatively similar results.

1.7.2 Effect of CR Dispersion on the Softening Points after storage

After a prolonged storage at elevated temperature, the modified binder is said to be stable, if the difference in the softening point of the top and bottom third sections is less than 2.5 °C [12]. To investigate the differences in the softening points and ultimate stability of the blend, samples obtained from top and bottom thirds were further evaluated by the ring and ball test. The tests were conducted in accordance with ASTM D5976 / ASTM D36 (95) and the observed values of the softening point temperature for the top and bottom thirds were compared based on the difference in the softening point temperature of the two. The objective was to get a proper understanding of the phase separation phenomenon in the light of the polymer dispersion in the bitumen phase.

From the laboratory investigation of the hot storage stability of modified blends with varied combinations, the results of the softening points of top and bottom thirds at different storage periods are compared in Tables 3 to 5. On each occasion, the test was repeated twice and samples were tested for softening point variation. The results were found very close in most cases. The average of the two has been tabulated.

TABLE 3 Softening points of CRMB 40/60 blends before and after storage

Hot storage stability (hrs)	CRMB 10%		CRMB 15%		CRMB 17.5%	
	Top	Bottom	Top	Bottom	Top	Bottom
	°C		°C		°C	
0	63.3	63.3	73.2	73.2	77.8	77.8
3	65.6	61.9	71.5	74.2	78.1	77.2
6	68.3	58.3	71.8	74.7	77.9	76.5
12	69.1	58.5	71.1	73.6	78.6	76.7
18	69.3	59.0	71.1	74.6	79.0	76.9
24	69.5	59.3	72.0	74.7	79.1	76.8

TABLE 4 Softening points of CRMB 70/100 blends before and after storage

Hot storage stability (hrs)	CRMB 10%		CRMB 15%		CRMB 17.5%	
	Top	Bottom	Top	Bottom	Top	Bottom
	°C		°C		°C	
0	61.8	61.8	67.7	67.7	75.0	75.0
3	65.7	59.2	66.3	67.5	75.6	75.0
6	65.9	55.1	66.0	67.1	76.3	74.5
12	65.7	54.4	65.1	68.2	77.3	73.9
18	65.9	55.1	64.7	67.6	77.8	74.0
24	66.2	54.4	65.2	68.3	77.5	73.8

TABLE 5 Softening points of CRMB 100/150 blends before and after storage

Hot storage stability (hrs)	CRMB 10%		CRMB 15%		CRMB 17.5%	
	Top	Bottom	Top	Bottom	Top	Bottom
	°C		°C		°C	
0	58.2	58.2	61.4	61.4	64.9	64.9
3	64.8	54.9	61.7	61.1	65.1	63.5
6	66.5	53.2	60.9	61.7	66.6	63.0
12	66.2	54.1	60.0	62.8	67.1	62.8
18	66.9	53.6	58.9	63.1	67.3	62.9
24	67.5	53.9	59.1	63.6	67.4	63.0

Moreover, the maximum difference in softening point temperatures of the top and bottom section of CRMB blends at the same CR concentration but different base bitumen are compared in Figs 10 to 12.

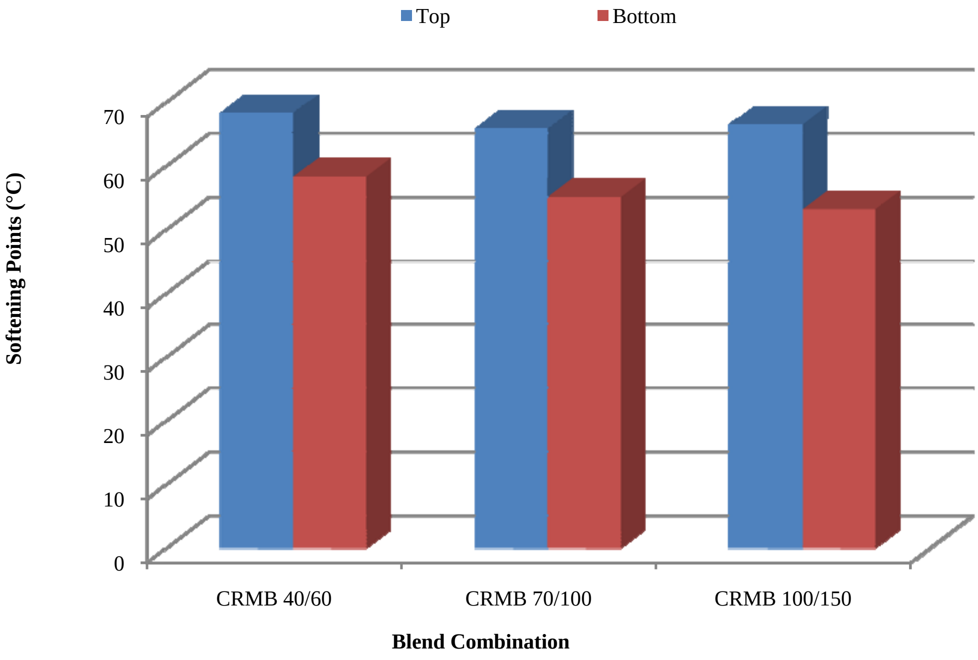

Fig 10 Maximum softening point variation in the CRMB with 10% CR concentration after storage

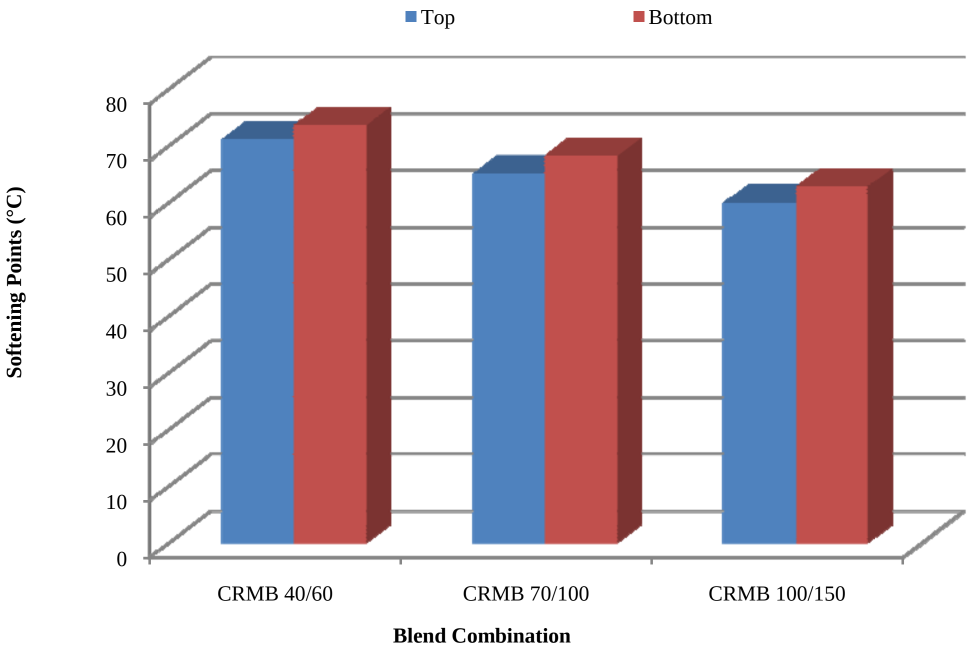

Fig 11 Maximum softening point variation in the CRMB with 15% CR concentration after storage

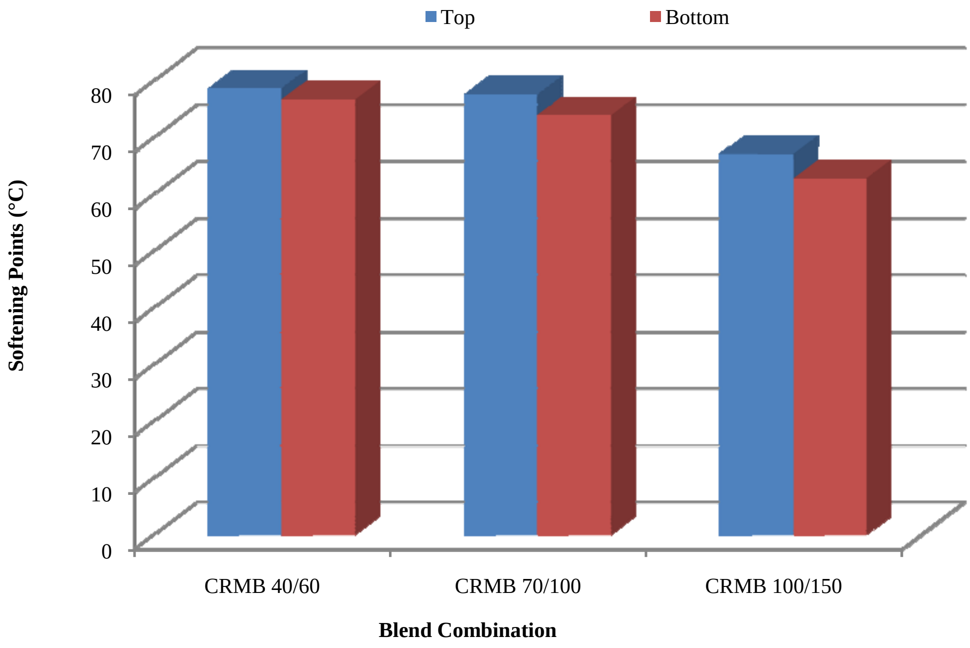

Fig 12 Maximum softening point variation in the CRMB with 17.5% CR concentration after storage

Phase separation between the CR and bitumen is a common problem mentioned in several studies. However, no quantitative theoretical understanding could be developed because of the wide particle size distribution, non-homogeneity of particle shape and curing processes during storage that modify both the continuous and particulate medium. Therefore, an attempt to understand the phase separation phenomenon during hot storage on the basis of density variations in the CR particles is made in this section. The effect of phase separation on the softening point of the two sections is further discussed in the light of the X-Ray image analysis in the following sections.

CR 40 mesh with three different proportions was added into the three base bitumen for the production of CRMB blends. The softening point results were also found to be in correlation with the results drawn from X-Ray CT image observations. This has also helped to find the facts, which actually govern the variation in the softening points of the binder in the two sections after the test. In general, the softening point of the material is mainly governed by the amount and size of the CR particles present in the blends. This was confirmed by the increasing trend of softening points with increase in the CR content in the blend. Moreover, the base bitumen type and storage duration has also affected the properties to some extent. To understand the phenomenon, CRMB blends were further analyzed for the difference in the softening point temperature after hot storage as a function of the base bitumen, CR content and storage duration.

1.8 Conclusion

1.8.1 Effect of Base Bitumen on the CRMB blends during Storage

From the observations, the penetration value of the base bitumen was found to be an influential factor to govern the separation of the two phases during the storage stability test. CRMB blends produced with base bitumen 100/150, which was the softest in the selection, revealed the highest difference in softening points between top and bottom sections of the storage tube. On the other hand, storage stability of the CRMB blend was found to increase with decreasing penetration value of the base bitumen used during the production of modified binder. As a result, CRMB blends produced with base bitumen 40/60 proved to be most stable among the selection.

1.8.2 Effect of CR Concentration on the CRMB blends during Storage

According to the softening point observations after storage, CRMB blends produced with 10% CR concentration with the three bitumen types have resulted in the most unstable binder. The differences in the results of softening points of the top and bottom sections are compared in Table 3 to 5. The observations were further confirmed by the dispersion of the CR particles in the bitumen matrix presented in Figure 6 to 8. Due to the limited amount of CR particles in the blend during the production, the highest possible decrease in the density could have been achieved as a result of swelling. Moreover, the presence of fewer CR particles could not affect the movement of the particles even after swelling. This has resulted in easy migration of the fines with higher density compared to bitumen residue to the bottom and swelled and less dense CR particles to the top. This distribution of CR particles has also resulted in higher softening points with larger CR particles in the top section compared to the lower softening point of the bottom containing only fewer fine CR particles.

CRMB blends produced with the CR at 15% concentration were found to be relatively stable compared to the above selection. The results are presented Fig 11, where the maximum differences in softening points between the two sections after storage are compared. To understand the findings, X-Ray images from the top and bottom sections of the storage stability specimens presented in Fig 7 were referred to. From the analysis, it was found that the observed bitumen-CR proportion in the blend provided an ample amount of CR particles in the bitumen matrix. In this scenario, the resulting swelling of the CR particles would be less compared to the CR at 10% concentration in the blend and could not achieve the highest swelling and ultimate reduction in density. Therefore, fine with more medium coarse CR particles were observed in the bottom and a lower number of coarse CR particles were observed at the top. Such a distribution of CR particles has resulted in a higher softening point value in bottom of the storage tube compared to the top. In addition, the increased number of CR particles could have also resulted in reduced particle movement and thus affected transportation of the CR particles to the desired sections.

Increasing the CR concentration to 17.5% resulted in an increased CR interlock. Based on the softening point observations presented in Fig 12, CRMB blends produced with a higher CR content such as 17.5% were found to be relatively stable with a further reduced difference in softening points of top and bottom third sections. In addition, from the image analysis presented in Fig 8, less movement of the CR particles was observed due to the reduced inter-particle distance and therefore the material was less prone to phase separation.

1.8.3 Effect of Storage Duration on the Properties of CRMB Blends

CR transportation in the bitumen matrix during storage was also affected by the duration of the storage test. CRMB blends comprised of 10% CR and softer base binders were more prone to early separation compared to the blends produced with increased CR concentration and binder hardness. In this regard, earlier combinations have shown maximum separation in the first 6 hours compared to the 12 hours of hot storage required by the later arrangement. No significant changes in the softening point value were observed after 12 hours of hot storage. In general, increasing CR content and decreasing penetration grade in the blend have resulted in delayed phase separation.

1.9 Acknowledgement

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