01 Jan 2013

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Recommended Citation

A. Ghavibazoo et al., "Effect of Crumb Rubber Modifier Dissolution on Storage Stability of Crumb Rubber-Modified Asphalt," *Transportation Research Record*, no. 2370, pp. 109-115, National Research Council (U.S.), Jan 2013.
The definitive version is available at https://doi.org/10.3141/2370-14

Effect of Crumb Rubber Modifier Dissolution on Storage Stability of Crumb Rubber–Modified Asphalt

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Crumb rubber–modified asphalt (CRMA) extensively suffers from high-temperature storage instability. Because of the higher density of the crumb rubber modifier (CRM) particles, they settle down to the bottom of storage tanks and cause variation between physical properties of the top and bottom samples. The storage stability of the CRMA changes under different interaction parameters and consequently different mechanisms of interaction. In this research the mechanism of separation under different levels of CRM dissolution was investigated by using Stokes' law. The extent and mechanism of CRM dissolution in asphalt was controlled through regulating interaction temperature and interaction mixing speed. A standard storage stability test was conducted on CRMA samples to measure the corresponding storage stability index. Viscosity of the liquid phase of CRMA was measured at the same temperature as that of the storage stability test (163°C), with the use of a rotational viscometer, to study the effect of viscosity development of the liquid phase on the storage stability of CRMA. Composition analysis was conducted by using thermogravimetric analysis to investigate the effect of changes in CRM composition on the storage stability of CRMA. Results reveal that, in addition to the CRM particle size reduction resulting from CRM dissolution, the viscosity development of the liquid phase affects the storage stability of CRMA with the interaction temperature of 190°C. But increasing the interaction temperature to 220°C changes the mechanism of dissolution and the nature of the residual CRM particles and consequently leads to a different mechanism of separation.

Modification of an asphalt binder is a common practice to improve its physical properties and performance. Modification of asphalt decreases its temperature susceptibility and this enables asphalt to withstand more load and more severe environments (*1, 2*). Asphalt modification also changes the composition of asphalt and its homogeneity, depending on the type of modifier (*3*).

In practice, asphalt binders are stored at high temperatures to facilitate handling and the mix production process (*2, 4*). Therefore, it is important for asphalt to remain stable during its storage time. However, in the case of most modified asphalts, severe phase separation occurs during storage at high temperatures. The mechanism of separation significantly depends on the type of modifier and its interaction with asphalt (*4*).

Because the modified asphalts consist of two distinct phases, its phase separation can be theorized based on Stokes' law. According to this theory, the phase separation in modified asphalt is governed by the terminal velocity of the dispersed phase in Newtonian liquid. The terminal velocity is the velocity of the displacement of the particles when the gravity force on the particles equals the drag force on the particles in a Newtonian liquid and can be quantified by the following equation (*5*):

$$v_t = \frac{2a^2 \Delta\rho g}{9\eta} \tag{1}$$

where

a = radius of dispersed particle,
$\Delta\rho$ = difference of density between particle and Newtonian liquid medium,
g = gravitational acceleration, and
η = viscosity of liquid medium.

According to this theory, terminal velocity of the particles is directly related to the square of the radius of the particles and inversely related to the viscosity of the medium. Decreasing the terminal velocity in the system will decrease the displacement velocity of the particles. This theory has been used by several researchers to understand the mechanism of separation in modified asphalts, containing different modifiers, and the effective parameters that can prevent the separation from happening (*2, 3, 6–8*).

The same concept can be considered about the crumb rubber–modified asphalt (CRMA) in which there are crumb rubber modifier (CRM) particles with various sizes suspended in liquid asphalt at high temperatures (*2*). CRM particles have a relatively higher density (1.15 ± 0.05 g/cm^3) than asphalt (1.02 g/cm^3) and consequently have the tendency to descend because of the gravitational forces, which depends on the volume of the CRM particles and difference between the density of the particles and asphalt (*9*). Conversely, asphalt at very high temperatures shows Newtonian liquid behavior with a finite viscosity and therefore shows resistance to displacement of CRM particles by applying the drag force, which depends on the volume of the CRM particles and the viscosity of the liquid. Manipulating these forces enables one to reduce the terminal velocity of the particles (displacement velocity of particles when the two forces are equal) and consequently reduce the sedimentation rate of CRM particles. To do this it is necessary to understand the changes in the size and density of the CRM and the viscosity of the liquid phase of the CRMA and their effect on storage stability of the CRMA. In CRMA, terminal velocity defines the sedimentation velocity of the CRM particles

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Transportation Research Record: Journal of the Transportation Research Board, No. 2370, Transportation Research Board of the National Academies, Washington, D.C., 2013, pp. 109–115.
DOI: 10.3141/2370-14

and, therefore, it is denoted as sedimentation velocity throughout this paper.

Storage stability of CRMA has been investigated in several studies considering different parameters, including CRM particle size, asphalt type, and interaction conditions (*2, 10–12*). It is stated that decreasing the CRM particle size, increasing the interaction mixing speed, or extending interaction time improves the storage stability of asphalt (*2, 11, 12*). Compatible asphalts have also shown better storage stability in the case of modification. In this study the effect of the dissolution of CRM on the storage stability of CRMA was investigated by using Stokes' law. The main objective was to investigate the possibility of improving the storage stability of CRMA only through controlling the dissolution of CRM and finding an optimum level of dissolution. Accordingly, the dissolution of CRM in asphalt was controlled through regulating the interaction parameters (interaction temperature, interaction mixing speed, and time) and CRM concentration. The effect of dissolution on the viscosity development of the liquid phase of the CRMA and the density of the CRM particles were studied by using a rotational viscometer and thermogravimetric analysis (TGA), respectively. Finally, the contribution of each of these factors to storage stability of the CRMA was investigated through storage stability tests.

MATERIALS AND METHODS

Interactions and Materials

One PG 58-28 performance-graded asphalt (NF) and one ambient processed CRM from passenger car tire sources (WTG) were used in this study. Asphalt was provided by Flint Hill Resources (Fargo, North Dakota) and the CRM was provided by Liberty Tire Recycling. The CRM particle size, in all interactions, was controlled to be smaller than the mesh No. 30 (0.55 mm) and larger than the mesh No. 40 (0.4 mm), according to the U.S. standard system. Seventeen interactions were conducted, considering different interaction parameters, listed in Table 1. As illustrated in Table 1, the varying parameters were CRM concentration, interaction temperature, interaction mixing speed, and time. All interactions were conducted by putting 1,200 ± 100 g of asphalt in 1-gal paint cans. The temperature was controlled by using an aluminum heating mantle (100B TM634) attached to a bench-type temperature controller and a J-type thermocouple. The CRM concentration was calculated as a percentage of the asphalt weight and was added to the asphalt after reaching the objective temperature. The mix was conducted by using a high shear mixer (HSM-100LM-2). Nitrogen gas was applied on top of all interactions to prevent any oxidation. The asphalt type, CRM type, and CRM size were not considered in this paper because of the scope of this work; however, they all have been considered in the research program of this research group and some of these results have been reported in previous studies (*12*).

Storage Stability Test

The storage stability test, also known as the separation test, was conducted on CRMA samples following ASTM D7173. In this test, a sample (50 ± 0.5 g) was poured into the specific tubes with standard dimensions, based on the ASTM standard, and kept at 163°C ± 5°C for 48 ± 1 h. Then, the tubes were cooled to −10°C ± 2°C. After cooling, the tubes were cut into three equal parts and the samples from the top and bottom parts of the tube (denoted top and bottom, respectively) were collected and stored for further testing.

To evaluate the storage stability of the samples, a formula was adapted from the old standard ASTM D5892-00 and other studies to develop the separation index (SI) (*2, 4, 11, 12*). In this study the storage stability of all samples was evaluated by conducting a dynamic shear rheometer analysis on the top and bottom samples to

TABLE 1 Interactions and Corresponding Code and Parameters

Interaction Name	Time (min)	CRM Concentration (%)	Mixing Speed (Hz)	Temp (°C)	Dissolution (%)	SI
NF-WTG-10%-10Hz160C-240min	240	10	10	160	13.8	41.6
NF-WTG-10%-30Hz160C-240min	240	10	30	160	18.8	30.7
NF-WTG-10%-10Hz190C-240min	240	10	10	190	23.1	33.3
NF-WTG-10%-30Hz190C-60min	60	10	30	190	26.5	32.9
NF-WTG-10%-30Hz190C-120min	120	10	30	190	30.7	37.3
NF-WTG-10%-30Hz190C-240min	240	10	30	190	41.2	34.7
NF-WTG-10%-30Hz190C-480min	480	10	30	190	55.0	26.6
NF-WTG-15%-30Hz190C-240min	240	15	30	190	41.2	21.9
NF-WTG-20%-30Hz190C-240min	240	20	30	190	35.1	NA
NF-WTG-10%-50Hz190C-240min	240	10	50	190	69.6	16.5
NF-WTG-10%-10Hz220C-120min	120	10	10	220	53.0	29.5
NF-WTG-10%-10Hz220C-240min	240	10	10	220	71.7	20.2
NF-WTG-20%-10Hz220C-120min	120	20	10	220	51.8	28.1
NF-WTG-20%-10Hz220C-240min	240	20	10	220	68.1	30.6
NF-WTG-10%-50Hz220C-240min	240	10	50	220	83.5	12.2
NF-WTG-15%-50Hz220C-240min	240	15	50	220	80.1	28.3
NF-WTG-20%-50Hz220C-240min	240	20	50	220	82.5	22.2

NOTE: Temp = temperature; SI = separation index; NA = not available.

measure their complex modulus. The complex modulus of the samples was used in the following equation to calculate the SI, which is a unitless index, of each sample.

$$\mathrm{SI} = \left(\frac{\max\left(G^*_{\mathrm{top}}, G^*_{\mathrm{bottom}}\right) - G^*_{\mathrm{avg}}}{G^*_{\mathrm{avg}}} \right) * 100 \tag{2}$$

where

G^* = complex modulus of the sample,
top and bottom = relative positions of samples in tube, and
avg = parameter averaged between top and bottom samples.

Viscoelastic Analysis of Top and Bottom Samples

A dynamic shear rheometer from Bohlin Instruments CVO (Worcestershire, United Kingdom) was used for viscoelastic analysis of the top and bottom samples. All tests were performed with 25-mm-diameter parallel plates at 58°C and 1.59 Hz. The gap between plates was selected to be 2 mm. To avoid the nonlinear region of viscoelasticity, all tests were performed in a strain control mode with strain less than 1%.

Rotational Viscosity Test

To investigate the effect of viscosity on storage stability of CRMA samples, the high-temperature viscosity of the liquid phase of the CRMA was measured with a Brookfield (DV-II) rotational viscometer. This test procedure was performed following the ASTM D4402-06 standard. A preheated sample at 163°C was poured into the preheated sample holder and kept in the chamber at 163°C for 15 min to stabilize the temperature. All measurements were conducted at 163°C and 20 rpm, with spindle #27.

Dissolution Test

It is known that CRM swells and dissolves into the asphalt matrix and consequently changes asphalt properties (*10, 13–15*). To investigate the effect of dissolution on the storage stability of the modified asphalt, the dissolution test, used by this research group in previous works (*16*) and other researchers (*17*), was conducted on the CRMA samples. In this method 10 ± 2 g of modified asphalt was diluted in trichloroethylene and drained through a mesh No. 200 (75 μm). The retained particles were washed with extra trichloroethylene, until the filtrate became colorless, and dried in the oven at 60°C to assure removal of all solvent residues.

Comparing the weight of the dried extracted CRM to the expected weight of the original CRM in the collected amount of the CRMA sample revealed the amount of dissolved CRM in the asphalt matrix. As it is clear in the test method, the portion of the CRM that passed through mesh No. 200 was considered as the dissolved portion of the CRM.

Extraction of Liquid Phase

The liquid phase of the CRMA was extracted by removing the non-dissolved CRM particles from the CRMA matrix, to measure the viscosity of the liquid phase of the CRMA. In this regard, the required amount of the CRMA sample was heated to 165°C and drained through mesh No. 200 (75 μm) in the oven at 165°C for 25 min. The extracted liquid phase was stored at −12°C immediately to prevent any unwanted aging or other unknown reactions.

TGA Method

TGA is a simple and valuable method in composition analysis of multicomponent materials (*18*). This method has been used by several researchers to investigate the composition of multicomponent polymeric materials including CRM (*19–22*). The TGA also has been used by this research group in previous works to monitor the changes in CRM composition after partial dissolution in asphalt (*23*). The studies showed that the CRM composition changes through interaction with asphalt. Therefore, in this study the TGA method was used to monitor the changes in CRM composition and their effect on the storage stability of the corresponding CRMA.

A TA Instruments Q500 TGA was used to carry out the stepwise isothermal thermogravimetric (SITG) analysis in this work. This method prevents overlapping of the decomposition temperatures of the different constituents in the sample and has higher accuracy in comparison with the conventional method, in which a constant heating rate is applied to the sample and the mass loss is measured as a function of temperature, and therefore a significant overlap could occur between decomposition temperatures of different constituents (*24*). In the SITG method, 20 to 25 mg of sample was heated with a constant heating rate (20°C/min) until the mass loss rate of the sample, measured automatically with the instrument, reached above a predefined constant (1%/min in this study). At this point the temperature of the sample was kept constant until the mass loss rate of the sample reached below a predefined constant (0.5%/min in this study). The sample was heated again, with the predefined heating rate, to the new temperature, where the mass loss criterion, mentioned earlier, was satisfied again. This process was repeated until the temperature of the sample reached a predefined temperature (420°C in this study). The type and concentration of each component in the sample, in this method, was defined on the basis of its specific decomposition temperature and the amount of decomposing mass at that temperature, respectively (*24*).

RESULTS AND DISCUSSION

Storage Stability of the Samples

The results obtained from the storage stability test of different CRMA samples are presented in Table 1. Storage stability of samples improves by increasing the amount of energy applied to the interaction, through higher temperature, mixing speed, and extended time. It can be seen that increasing the mixing speed at 160°C and 190°C decreases the amount of SI. Also, extending the interaction time from 60 min to 480 min under the same interaction conditions (190°C and 30 Hz) improves the storage stability (decreases the SI) of the CRMA, which is in agreement with the results of other researchers (*2, 10, 11*).

Moreover, the results show that increasing the CRM concentration under the intermediate interaction conditions, 190°C and 30 Hz, improves the storage stability (decreases the SI) of the CRMA (*11*). However, at a higher interaction temperature (220°C), increasing the CRM concentration degrades the storage stability of the asphalt, which can be attributed to different mechanisms of separation under these two conditions, as explained further in the next sections.

Dissolution of CRM in Asphalt

Dissolution tests on CRMA samples reveal the amount of mass lost by CRM particles during interaction, which can imply the amount of reduction in the volume of the CRM particles, assuming a constant density of CRM throughout the interaction. Dissolution tests also reveal the amount of CRM components released into the asphalt matrix, which can contribute to the viscosity development of the liquid phase of the CRMA. The dissolution test results are presented in Table 1.

Results from previous studies of this research group showed that less than 20% dissolution of CRM in asphalt, which occurs at a low interaction temperature (i.e., 160°C) or in the early stages of an intermediate interaction temperature (i.e., 15 min at 190°C), mainly corresponded to the release of the oily phase of the CRM into the asphalt matrix (*16*). Therefore, on the basis of previous studies and the results in this study, it can be stated that at a low interaction temperature (i.e., 160°C) or in the early stages of an intermediate temperature (i.e., 15 min at 190°C), the swelling of CRM is the dominant activity of CRM and therefore the size of CRM particles is increasing relative to the original CRM size. However, when more interaction energy is applied (through increasing the temperature, mixing speed, or interaction time), CRM particles start releasing their polymeric components into the asphalt matrix. These components define the volume of the CRM particles and their release results in size reduction of the CRM particles.

Viscosity of Liquid Phase of CRMA

Viscosity of the liquid phase of the CRMA was measured at the same temperature of the storage stability test (163°C) to investigate its effect on storage stability of the corresponding CRMA. The trend of viscosity development of the binder as a function of the dissolved percentage of CRM by the weight of asphalt is presented in Figure 1. Regression analysis on these results and related statistical parameters, including the R^2 and significance *F,* is presented below:

	Equation	R^2	*Significance* F
Interactions at 190°C	$y = 28.5x + 27.2$	.89	.99999
Interactions at 220°C	$y = 14.5x + 94.7$	.82	.99549

In Figure 1, the viscosity development of the liquid phase of the CRMA is presented under two different interaction temperatures. Temperature is known to be the main parameter that defines the mechanism of CRM activities in the binder (*15*). As the results in Figure 1 and the related statistical analysis in the table above show, viscosity development of the liquid phase shows a good correlation, high R^2 value and high significance F, with the amount of dissolved portion of the CRM in asphalt. However, the slope of the viscosity development at 190°C is slightly higher than the one at 220°C. This can be attributed to differences in the mechanism of dissolution of the CRM and releasing components into the asphalt matrix, which have been investigated and reported by other researchers (*14, 15, 25*).

Effective Parameters on Progression of Storage Stability

As mentioned before, the separation in the CRMA can be theorized by Stokes' law. Based on this theory, the sedimentation velocity (terminal velocity) of the CRM particles during high-temperature storage of the CRMA is a function of the radius of CRM particles and the viscosity of the liquid phase of the CRMA. Based on Equation 1, decreasing the radius of the particles or increasing the viscosity of the liquid phase decreases the sedimentation velocity of the particles in the liquid medium of asphalt and consequently results in better storage stability of the CRMA. To study the specific effect of each of these parameters on the experimental storage stability of the CRMA, the effect of the dissolution of CRM particles, representative of the reduction in the size of CRM particles, and the effect of the viscosity of the liquid phase of the CRMA, dispersing medium, on the final storage stability of the CRMA samples were investigated through regression analysis as shown in Figure 2 and Table 2.

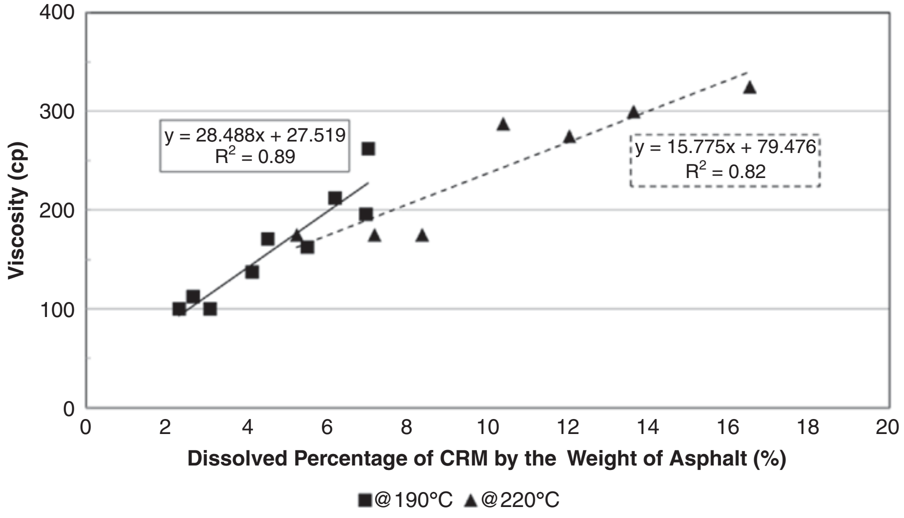

FIGURE 1 Viscosity development of the liquid phase of CRMA as a function of dissolved percentage of CRM by the weight of asphalt (cp = centipoise).

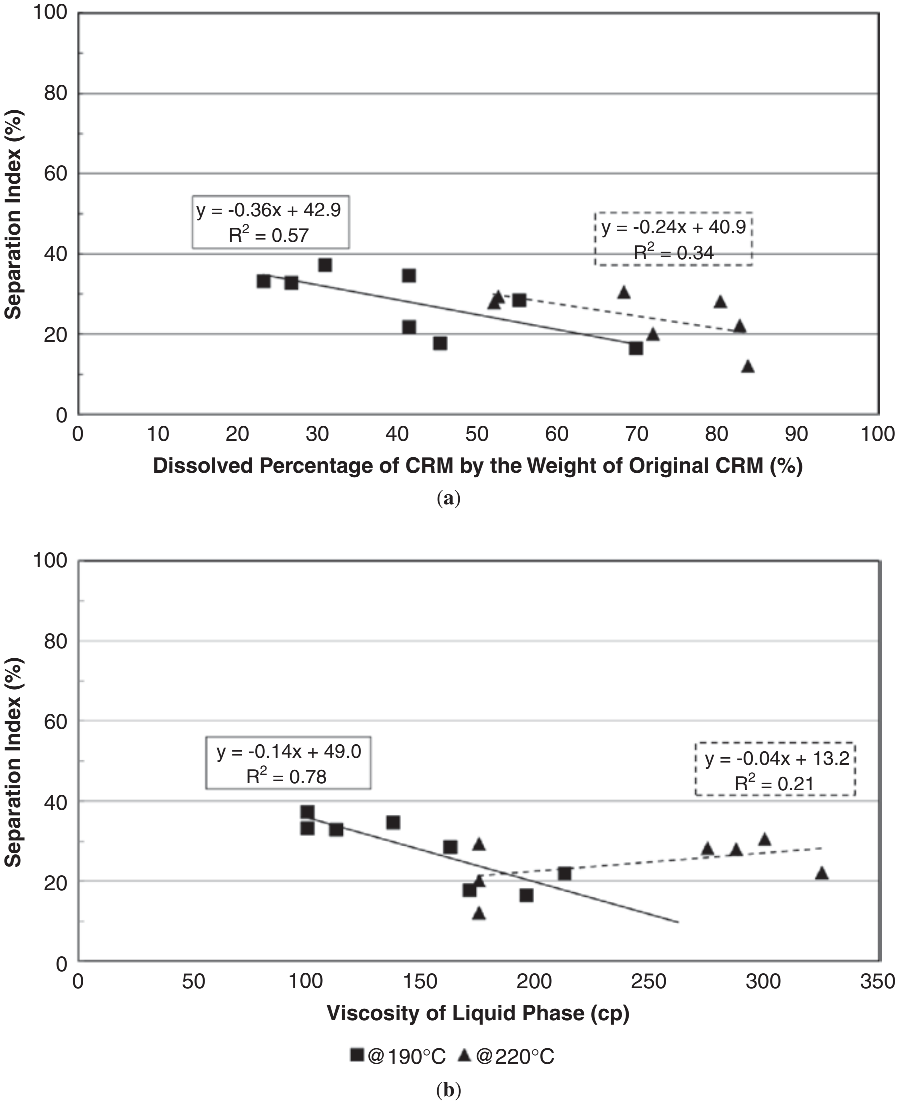

FIGURE 2 Separation index of CRMA samples as a function of (*a*) dissolved percentage of CRM and (*b*) viscosity of the liquid phase of CRMA.

Figure 2*a* represents the effect of the dissolved percentage of CRM on the storage stability of CRMA under two different interaction temperatures. Regression analysis on these results in Table 2 indicates that for interactions at 190°C, the correlation between these two parameters does not show a strong R^2 (.57), but it is a significant correlation because the significance F factor is considerably high (.9889). Therefore, the correlation between these two parameters is meaningful for interactions at 190°C by a confidence interval of 95%. For the interactions at 220°C, the R^2 is .34 and the significance F factor is .8381, which is less than .95 and indicates that correlation at this condition is not meaningful.

These results illustrate that for interactions at 190°C, dissolution of CRM improves the storage stability of the CRMA, which can be attributed to the reduction in the particle size of CRM and accordingly

TABLE 2 Regression Analysis of Effect of Viscosity of Liquid Phase of CRMA and Dissolution of CRM on Storage Stability of CRMA with 95% Confidence Interval

	Effect of Dissolved CRM Percentage on Storage Stability of CRMA			Effect of Viscosity of Liquid Phase on Storage Stability of CRMA		
Interaction Temperature	Equation	R^2	Significance F	Equation	R^2	Significance F
190°C	$y = -0.36x + 42.9$	.57	.98898	$y = -0.14x + 49.0$	.78	.99930
220°C	$y = -0.24x + 40.9$	.34	.838101	$y = 0.04x + 13.2$	.21	.705121

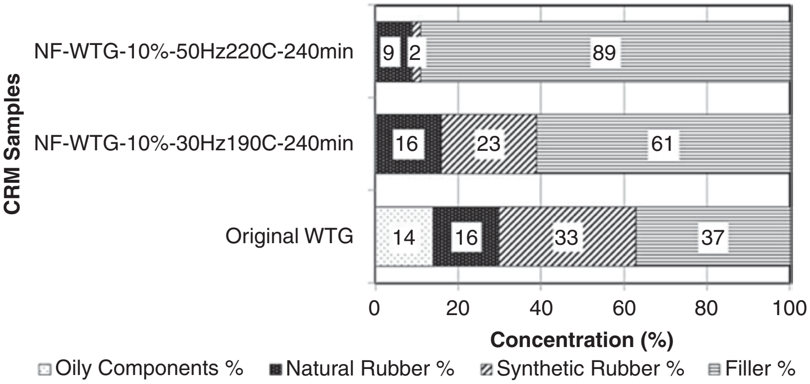

FIGURE 3 Composition of original CRM sample and extracted CRM samples from two different interactions after 240 min.

reduction in the terminal velocity based on Equation 1. However, for interactions at 220°C, there is no meaningful correlation between the dissolution percentage and the storage stability of CRMA in spite of extensive dissolution of CRM and consequently considerable reduction in particle size. This can be related to the changes in the nature of CRM particles, explained in the next sections.

Figure 2*b* represents the storage stability of the CRMA as a function of viscosity of its liquid phase at 163°C (the same temperature as the temperature of the storage stability test) for two sets of interactions at two different interaction temperatures, 190°C and 220°C. Regression analysis on these results, presented in Table 2, shows that at 190°C the storage stability of the CRMA has a meaningful correlation with the viscosity of the liquid phase of the CRMA (significance $F = .9993$) in the confidence interval of 95%. However, the correlation of these parameters for the interactions at 220°C is not meaningful (significance $F = .7051$) in the same confidence interval. This illustrates that increasing the viscosity of the liquid phase of the CRMA samples, interacted at 220°C, does not affect its storage stability, which again proves the different mechanisms of separation in these CRMA samples.

The results in Figure 2 and Table 2 illustrate that for interactions at 190°C, two parameters that were mentioned in the sedimentation velocity equation based on Stokes' law control the dispersion of CRM particles in the matrix and can help to improve the storage stability of the samples. However, the mechanism of separation in interactions at 220°C does not show the same trend and the two factors, viscosity of the liquid phase and dissolution percentage of the CRM, have no effect on the storage stability of the CRMA samples. These results explain the reason behind the different effect of CRM concentration on storage stability of CRMA under different interaction conditions. Increasing the CRM concentration for the interactions at 190°C improves the storage stability of CRMA, but for interactions at 220°C this has the reverse effect according to the results in Table 1. To investigate the reason behind the different separation mechanisms on CRMA samples with two different interaction temperatures, 190°C and 220°C, TGA analysis was conducted and is presented in the next section.

TGA Results

In this research the TGA analysis was conducted on original and extracted CRM samples to investigate the changes in composition of CRM and their possible effect on the storage stability of the CRMA. Figure 3 shows the composition of the original CRM sample and two extracted CRM samples from two different CRMAs.

The results show that after 240 min of interaction with asphalt at 190°C and 30 Hz, CRM still contains a considerable amount of polymeric components and the overall composition is not changed significantly relative to the original CRM. However, after 240 min of interaction at 220°C and 50 Hz, it can be seen that CRM particles have lost all their polymeric components and basically the remaining particles mainly contain fillers, such as carbon black.

The density of the CRM depends on the concentration of each of its main components. About 80% of the constituents of the extracted CRM from interaction at 220°C and 50 Hz after 240 min is carbon black, which is the main density builder in CRM because of its higher density (1.8 to 2.1 g/cm^3) relative to the density of asphalt (1.02 g/cm^3) and CRM (1.15 ± 0.05 g/cm^3) (*26*). The higher density of the carbon black and its high concentration in the nondissolved CRM particles, extracted from the CRMA sample with interaction conditions of 220°C and 50 Hz, significantly increases the density of the remaining particles. This increase leads to a higher difference between the density of the CRM particles and the asphalt ($\Delta\rho$) in Equation 1 and consequently a higher sedimentation velocity. Therefore, CRM particles sediment faster in this situation and corresponding CRMA shows lower storage stability.

The TGA results shown in Figure 3 show that the nature of the remaining CRM particles in the CRMA matrix is different between interactions at 190°C and 220°C and, therefore, the mechanisms of separation of these particles are totally different, which is shown in the earlier section.

CONCLUSION

In this research the possibility of improving the storage stability of asphalt through controlling the dissolution of CRM in asphalt is investigated by conducting storage stability tests, high-temperature rotational viscosity tests, and TGA analysis.

Regression analysis on the results showed that at an intermediate interaction temperature (190°C), dissolution of CRM in asphalt can improve the storage stability of the CRMA samples. This improvement occurs as a result of the increase of the viscosity of the liquid phase of CRMA and the decrease of the CRM particle size, which are the two main parameters that define the sedimentation velocity of the particles in any medium based on Stokes' law. Under this condition of interaction, increasing the CRM concentration leads to improvement of the storage stability of the CRMA samples. This improvement also can be attributed to the increasing of viscosity of the liquid phase of CRMA and its effect on the sedimentation velocity of the CRM particles.

However, at a higher interaction temperature (220°C) at which extensive dissolution of CRM happens, the separation of the remaining CRM particles does not follow the same trend as the interaction at the lower temperature (190°C). Under this condition of interaction, remaining CRM particles are mainly composed of carbon black, based on TGA analysis, which has a much higher density (1.8 to 2.1 g/cm^3) compared with the density of asphalt (1.02 g/cm^3) and the original CRM (1.15 ± 0.05 g/cm^3). Therefore, at this level of interaction, the high density of particles affects its sedimentation velocity and the particles' sedimentation occurs independent of the viscosity of the liquid phase of CRMA. Nevertheless, at this stage, remaining CRM particles have less modifying effect on the total CRMA matrix and therefore, in general, samples of interactions with higher interaction temperature (220°C) show higher storage stability. But the storage stability degrades by increasing the CRM concentration in asphalt as the concentration of the remaining particles increases.

ACKNOWLEDGMENT

This material is based on work supported by the National Science Foundation.

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Any opinions, findings, and conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the National Science Foundation.

The Characteristics of Asphalt Materials Committee peer-reviewed this paper.