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Controlling Performance of Crumb Rubber–Modified Binders Through Addition of Polymer Modifiers

Magdy Abdelrahman

The use of tire rubber as a modifier to enhance the properties of asphalt mixes through the wet process has proved to be a successful procedure, but full control of the properties of crumb rubber–modified binders has been a challenge. The literature indicates a wide variation on adding rubber to asphalt and that the degree of success of rubber modification in AC mixes depends on several factors, the majority of which relate to the method of mixing, storing, and transporting and to construction technologies. Understanding the nature of the interaction process between asphalt cement and crumb rubber–modifier (CRM) helps explain the development of binder properties. This paper covers the wet process, a relatively different technology in the application of CRM in asphalt with virgin polymers used to control and enhance the performance properties of CRM binders. The paper provides some clear insights into the mechanisms by which the interaction, with and without the existence of polymer modifiers, takes place. Effects of the interaction process variables, time and temperature, are explained. Results of this research are based on monitoring the changes in the rheological parameters of the developed binder.

This paper investigates a relatively different technique in the application of crumb rubber–modifier (CRM) in asphalt by the addition of virgin polymer modifiers to the asphalt–rubber mix. This technique offers a potential alternative to the straight addition of virgin polymer to neat or blended asphalts. The main objective of this research is to investigate the effectiveness of adding polymer modifiers to the asphalt–CRM interaction to enhance and improve the binder performance and storage properties. The paper presents some differences in the mechanism of property development through the interaction between asphalt and CRM, depending on whether virgin polymers are present or not.

NATURE OF ASPHALT–RUBBER INTERACTION

The nature of the mechanism by which the interaction between asphalt cement and CRM takes place has been reported in the literature as two main mechanisms: particle swelling and degradation, which includes devulcanization, depolymerization, or both (*1, 2*). These activities occur as the binder is subjected to different combinations of interaction time and temperature. The "wet process" as developed by McDonald in the late 1960s involves about 20% by weight ground tire rubber (#8 to #20 mesh size) interacted with asphalt at elevated temperature. The reaction of the wet process, as reported by Heitzman, is not a chemical reaction (*3*). It is the absorption of aromatic oils from the asphalt cement into the polymer chains, which are the key components in CRM. Heitzman reported that the reaction should not result in a melting of the CRM into the asphalt cement. Rather, rubber particles are swollen to two to three times their original volume by absorption of the asphalt's oily phase at high temperatures, 160°C to 200°C, to form a gel-like material. The change in rubber particle sizes and formation of gel structures result in a reduction in the interparticle distance between rubber particles and the presence of a modified material, which may produce a viscosity increase by up to a factor of 10 (*3, 4*). Rubber swells in a time- and temperature-dependent manner. Figure 1 illustrates the relation between binder viscosity and particle size changes. Figure 1*a* shows a typical viscosity progression over time. Figure 1*b* shows the changes that occur in a typical rubber particle as the interaction process progresses. Figure 1*c* shows the development of the binder matrix as rubber swells. After rubber reaches maximum swelling in the asphalt (Case II in Figure 1*c*), if the temperature is too high or the time is too long, dispersion into the asphalt begins as the rubber experiences depolymerization because of long exposure to the high temperatures—an undesirable occurrence (*3, 5*). This may cause a gradual reduction in viscosity (Case III in Figure 1*c*). If the rubber is kept at very high temperature for an extended time, full depolymerization can occur (Case IV in Figure 1*c*), as the rubber experiences full dispersion in the asphalt and the binder loses most of its modified properties.

The gradual change in the viscosity of the binder has been used to indicate the progress of the interaction between asphalt and rubber. Green and Tolonen emphasize the importance of controlling the swelling processes through controlling the interaction time and temperature and concluded that temperature has two effects on the interaction process (*6*). The first effect is on the rate of swelling of rubber particles. As the temperature increases, the rate of swelling increases. The second effect is on the extent of the swelling. As the temperature increases, the extent of swelling decreases. Particle size controls the swelling mechanism over time and affects the binder matrix. Buckley and Berger show that the time required for swelling increases with the particle radius squared (*5*). Abdelrahman and Carpenter compared property development of fine rubber versus coarse rubber in asphalt interactions (*2, 7*). They concluded that fine rubber swells faster and depolymerizes faster, affecting the liquid phase more than the matrix of the binder, and that coarse rubber has more effect on the binder matrix but has less effect on the liquid phase than does the fine rubber. Liquid-phase modifications are more stable than matrix modifications.

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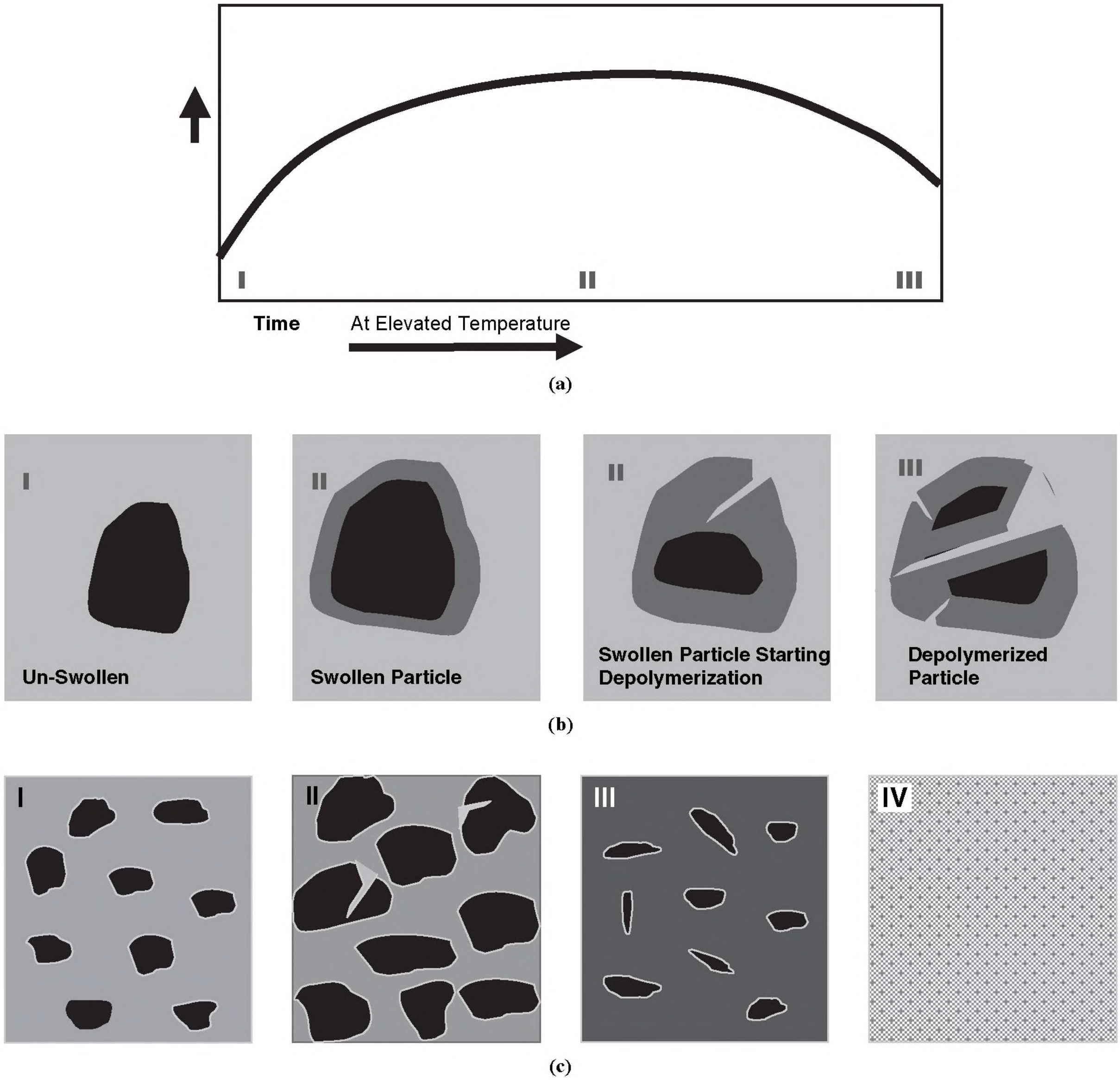


FIGURE 1 Progression of asphalt–rubber interaction at elevated temperature: (*a*) change in binder viscosity over time at elevated temperature, (*b*) change in particle size over time at elevated temperature, and (*c*) change in binder matrix over time at elevated temperature.

The partial dispersion of CRM in asphalt releases components to the liquid phase of the binder and affects the binder properties. It is explained in the literature as either depolymerization or devulcanization (*1, 4*). Both are chemical reactions that reduce the molecular weight of the rubber by breaking chemical bonds. Devulcanization breaks sulfur–sulfur or carbon–sulfur bonds that are formed by the vulcanization process during tire production. The literature on the asphalt–rubber interaction process does not clearly distinguish between the two concepts, particularly at temperatures below 240°C (*1, 8*). Research by the Western Research Institute suggests that devulcanization can occur at high temperatures (*9*). Bahia and Davies claim that the increase in binder viscosity cannot be accounted for only by the existence of the rubber swelling particles (*4*). They examined theories commonly used for particulate-filled composite materials to calculate the increase in viscosity of CRM binders and concluded that these theories underestimate the increase in binder viscosity by a large margin. There has to be some type of interaction phenomenon that not only increases the effective volume of the rubber particles, but also changes the nature of the liquid phase. Changes in the properties of the liquid phase of the binder are related to the degree of cross-linking in the material, which in turn gives the material its elastic characteristic, as can be measured by the values of the elastic component (*10*). The change in magnitude of the phase angle of the binder during material processing can be an indication of the primary mechanism involved.

EXPERIMENTAL CONSIDERATIONS

Using high CRM content through the wet process enhances the performance of the binder but sets limitations concerning compliance with the Superpave® testing and the workability of the binder, particularly in spray applications. Milling and further processing of high CRM content helps. However, viscosity remains high enough to diminish workability in mixing or spray applications. Using a lower CRM content with added polymers has been an option to improve workability. In addition to the traditional wet process application, this paper compares two approaches of adding polymer modifiers to CRM in asphalt applications. The first is the straight addition of polymer to the asphalt–rubber interaction. The second is treating CRM with virgin polymer before interacting with asphalt. In this paper that approach will be referred to as the proprietary technology. This paper presents testing results of two experiments: the first, which is AC-10 asphalt cement and nontreated CRM product made of truck tires, is a combination of both natural and synthetic rubber sources at 10% of the asphalt weight. CRM particle size is controlled as passing Sieve #30 and retained on Sieve #40, according to the U.S. standard system. Rouse Polymerics International, Inc., Vicksburg, Mississippi, supplied the CRM product. Emulsico at Urbana, Illinois, supplied the AC-10 asphalt cement. The interaction temperature is controlled at three levels: 160°C, 200°C, and 240°C. Individual binder formulations are prepared in 500-g batches in an oil bath. No mechanical shear is used in the interaction process. A BARNANT Mixer Model 750-0200 with a three-blade propeller is used in mixing the binder during the interaction process. Mixing at 200 rpm is applied for the initial 3 min, after which interaction time begins. After mixing, the speed is lowered to 80 rpm for the rest of the interaction period. Interaction time starts immediately after mixing up to 3 h. The detailed procedures of this experiment can be found in the literature (*2, 7*). Material testing is conducted at 52°C and 10 radians/s with the dynamic shear rheometer device, a PAAR PHYSICA Model RHEOLAB MC100. Limited replicate testing indicates that the presented data are valid (*2, 7*).

The second experiment was conducted with a Citgo PG 64-22 asphalt and a proprietary modified CRM product with styrene-butadiene-styrene (SBS) polymers in the preprocessing of the CRM. More than one type of SBS modifier was used in this study. Full details on the proprietary products can be found in the literature (Rouse, Deeb, White, and Abdelrahman, U.S. Patent 6,815,510,2004). A ROSS Mixer Model PVM2 was used in interacting asphalt with modifiers. The mixer has three independent blades: a shearing blade, mixing blade, and stirring blade. Mixing, stirring, and shearing speeds can be controlled through independent controllers. Temperature control is provided through an attached heating oil system that ensures accurate temperature control. Binders were mixed, sheared, or both, depending on experimental settings. In cases of both mixing and shearing processing, asphalt is heated to the appropriate temperature. The modifier is then added to the asphalt and is mixed for 10 min or until the temperature stabilizes. The binder is mixed for 1 h at a speed of 3,500 rpm. If shearing is to be applied, it is done at a speed of 7,000 rpm for 2 h, simulating an ultra-high-speed shearing mill in an asphalt terminal. The rest of the designated reaction time is used for mixing only at a speed of 3,500 rpm. In the case of mixing-only experiments, mixing at 3,500 rpm for the total interaction time is applied. Selected binders were evaluated by separation testing. The separation test, also known as the cigar-tube test, is a measure of asphalt–modifier compatibility. It is commonly used in approving terminal blending for asphalt binders. Detailed procedures for sample preparation are provided in ASTM D-5976. In the cigar-tube test, an aluminum tube filled with asphalt is sealed and kept undisturbed for 48 h at 163°C. The tube is then frozen and cut into three parts. Both top and bottom parts are prepared and tested in accordance with the Superpave dynamic shear rheometer (DSR) test. A TA-Instrument AR-1000 device was used in this experiment. Separation (%) is calculated with the following equation:

$$\text{separation } (\%) = 100 * \frac{\left[(G^*/\sin\,\delta)_{\max} - (G^*/\sin\,\delta)_{\text{avg}}\right]}{(G^*/\sin\,\delta)_{\text{avg}}} \tag{1}$$

where

G^* = shear modulus (measured in DSR test),
δ = phase angle (measured in DSR test),
$(G^*/\sin\,\delta)_{\max}$ = higher value of either the top or the bottom portion of the tube, and
$(G^*/\sin\,\delta)_{\text{avg}}$ = average value of the two portions.

Fluorescent microscopy images were taken with a NOVA EPI-Fluorescent Microscope Model 982ES in the labs of Citgo Asphalt Refining Company in Savannah, Georgia. A nonmodified CRM product was used in this experiment for comparisons, GF-40. Table 1 lists the components of the modified and nonmodified CRM materials as tested in this study. For example, modified CRM1 is a preprocessed CRM using SBS Type 1. CRM + SBS1 contains 75% CRM + 25% SBS Type 1 by weight and was used as 4% of the asphalt content. CRM + SBS2 contains 66% CRM + 33% SBS Type 2 by weight. The two components were added separately to the hot asphalt with no preprocessing of CRM. All DSR testing of this experiment was conducted at 76°C with parallel plate geometry. That was justified because the developed proprietary binder is not a filled system. Solubility testing on the proprietary binder confirms that observation. Preprocessing of CRM along with high-speed shearing reduces the CRM particle sizes significantly.

TABLE 1 Components of Crumb Rubber Modifiers

Modifier	Content % of Asphalt Weight	CRM Average Particle Size	Content % of CRM/Polymer by Total Weight	Technology
Proprietary modified CRM	3.8	80 mesh	66/33	Proprietary
CRM + SBS1	4	80 mesh	75/25	Added separately
CRM + SBS2	4	80 mesh	66/33	Added separately
Modified CRM1	4	80 mesh	75/25	Proprietary
Modified CRM2	4	80 mesh	66/33	Proprietary
GF40	3.8	40 mesh	100	NA
CRM 30-40	10	30–40 mesh	100	NA

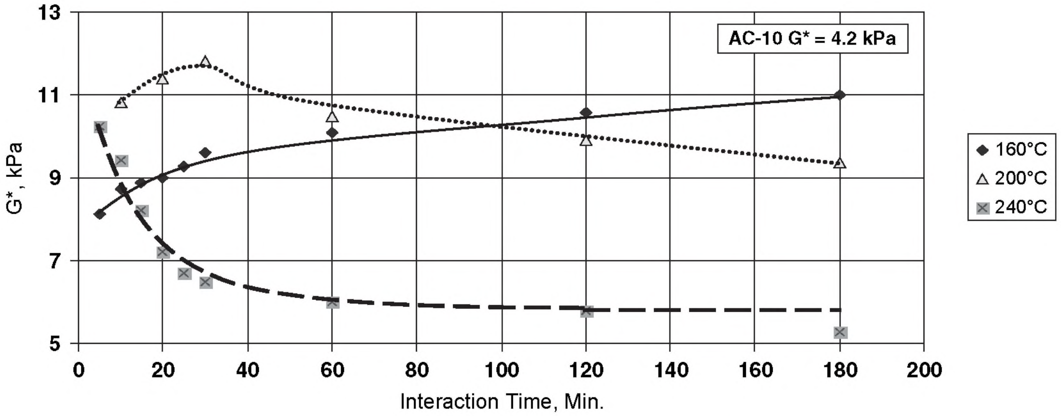


FIGURE 2 Property development of asphalt with nonmodified CRM—G* [10% CRM (30–40) size, tested at 52°C and 10 radians/s].

PERFORMANCE OF NONMODIFIED CRM

The following sections present the testing of the first experiment and examine the development of the binder properties, G* and δ, under precisely controlled interaction conditions extending to 3 h immediately after the CRM is mixed with asphalt. The sections relate the changes in the binder properties, G* and δ, to two process characteristics, or mechanisms, swelling and depolymerization–devulcanization, as they will be called in this study. The purpose is to provide insights into the interaction process and to show that binder properties could be controlled through controlling the interaction process.

Figure 2 presents the G* data of nonmodified CRM with AC-10 asphalt as outlined in the description of the first experiment. The data illustrate the expected relationships with temperatures and follow the trend of Figure 1*a*. At the low temperature, 160°C, swelling is continual over the entire time period as illustrated by the continual increase in G*. At the intermediate temperature, 200°C, swelling is still occurring at the beginning of the process. The development of G* in the first few minutes at 200°C is more significant than that after 3 h at 160°C. After the first 30 min at 200°C, swelling is offset as the swollen rubber particles are depolymerizing, releasing more components back to the liquid phase of the binder and decreasing G*. At the high temperature, 240°C, swelling of the CRM material has been mostly completed before the first sample at 5 min. The G* value is decreasing continually during the time period. The more significant decrease in G* at 240°C suggests a higher degree of depolymerization as compared with that at 200°C. This clearly shows the effect of temperature on the extent of swelling and on the degree of depolymerization–devulcanization of the rubber particles; the higher temperature causes more depolymerization–devulcanization, which results in a lower stiffness. The effect of high interaction temperatures causing partial or full depolymerization of rubber particles is confirmed in the literature (*1, 2*). The data for the 160°C interaction follow the first part of the trend of Figure 1*a*. The 160°C temperature is not a high temperature; most activities consist of swelling. The data for the 240°C interaction follow the last part of the trend of Figure 1*a*. The 240°C temperature is high, and most activities are depolymerization. Figure 3 shows the effect of temperature on the modification of the phase angle (δ). A similar trend for the low, the intermediate, and the high temperatures is obtained. At low-temperature interaction, modifications continue during the entire time period. The interaction starts with a higher value and ends after 180 min with a significantly lower value than the original material, indicating the same modification trend as for G*. The intermediate-temperature interaction shows a unique behavior of the CRM material, which drops quickly to a lower δ and then increases at a slow rate.

The 240°C interaction is a faster progression of the last part of the 200°C interaction; the CRM material experiences an increase in the phase angle as the interaction progresses over time. The literature suggests that G* and δ are not developed by the same interaction

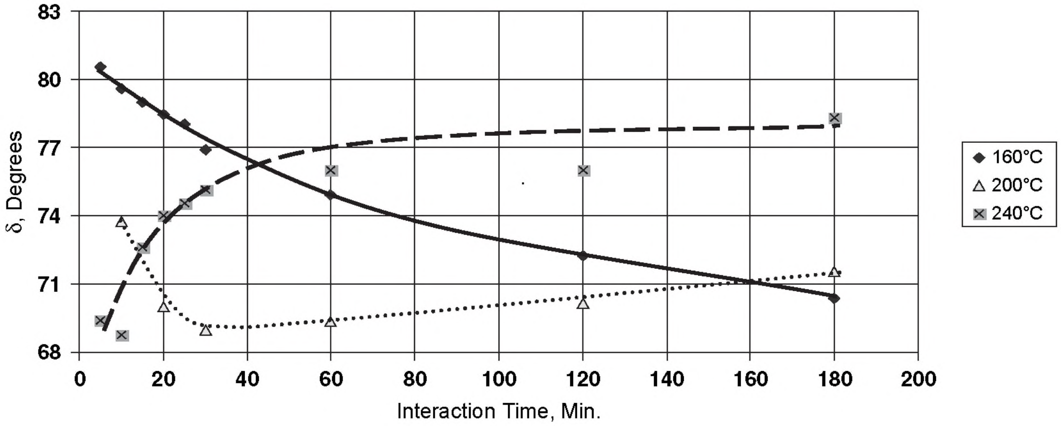


FIGURE 3 Property development of asphalt with nonmodified CRM—δ [10% CRM (30–40) size, tested at 52°C and 10 radians/s].

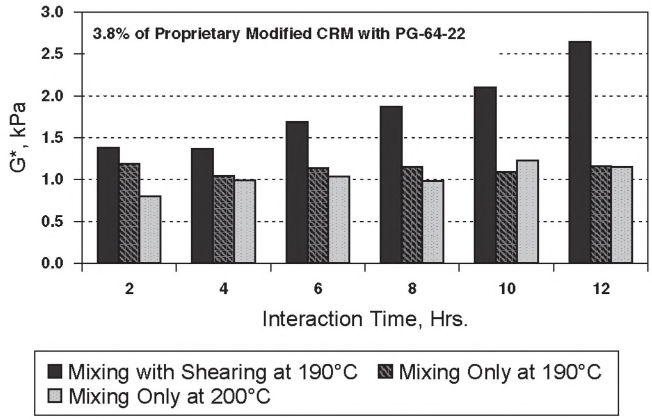


FIGURE 4 Property development of proprietary CRM binder—G* (unaged).

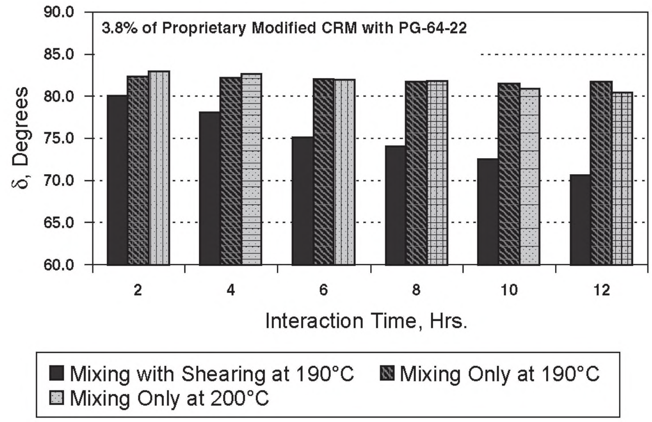


FIGURE 5 Property development of proprietary CRM binder—δ (unaged).

mechanisms (*1, 2*). Testing other combinations of asphalt–CRM materials shows a time lag in the development of the two properties (*2*). The literature indicates that the increase in G* is due mainly to particle swelling. The decrease in the phase angle continues during early stages of depolymerization–devulcanization and indicates that swelling is not the only factor affecting the development of the phase angle. Components exchange between asphalt and rubber in the early depolymerization–devulcanization stages stiffens the binder liquid phase with a more elastic component (*1, 2, 6*). This modifies the phase angle. Destruction of the cross-linking as the binder is exposed to high interaction temperature causes a reduction of the phase angle (δ).

PERFORMANCE OF POLYMER-ADDED CRM BINDERS

The following sections will focus on the 200°C interaction temperature in evaluating the effectiveness of added polymers to CRM binders. Figures 4 and 5 present details of the interaction of PG 64-22 asphalt with a preprocessed CRM material tested at 76°C. One of the two main reasons for considering the 200°C temperature first as a comparison with the interactions of nonmodified CRM as property development for both G* and δ was that changes occurred at this temperature. The nonmodified CRM interactions started with property improvement but changed to property loss in both cases. Thus, G* and δ were evaluated for testing conditions that were found critical for nonmodified CRM. The proprietary CRM interactions continue to improve properties with time. Development of the proprietary system requires energy to break the vulcanized links in the CRM material (*1*). Second, as a production consideration, the 200°C temperature is more efficient than lower temperatures. A main advantage of preprocessing CRM is to reduce the need for mixing energy, shearing energy, or both through the interaction with asphalt as partial devulcanization of CRM is achieved before mixing with asphalt. It can be seen that additional shearing at 190°C has increased G* and reduced δ significantly; this indicates improvement in the material behavior over time. The binder system uses the shearing energy to develop a new network between CRM and SBS that improves the binder properties. As presented in Figure 2, the 200°C temperature achieves maximum property modifications much sooner than lower temperatures. However, increasing the interaction temperature, alone, from 190°C to 200°C for proprietary CRM is not sufficient to alter the binder properties.

Table 2 presents an example of quality control testing on PG 76-22 binder made with 3.8% proprietary CRM product and original PG 64-22 asphalt at 190°C. The table includes testing on the original PG 64-22. It shows that the produced PG 76-22 binder complies with the AASHTO M-320 specifications. According to the asphalt supplier, about 3.0% of virgin SBS would be required to modify the PG 64-22 binder into a PG 76-22 grade with less than 5% separation, as per Equation 1 (*11*).

In addition to preprocessing, changing SBS type, SBS content, or both may lead to variations in binder properties. Figure 6 pre-

TABLE 2 Quality Control Testing on Original and Modified Proprietary Binder

AASHTO M-320	Measured Parameter–Test Method	Original PG 64-22	Modified PG 76-22
Flash point	AASHTO T48		278 C
Rotational viscometer	Viscosity at 135°C, Pa-s, ASTM D4402		1.140 Pa-s
Dynamic shear rheometer (DSR)	Un-aged G*/sin δ, at 64°C (original) and at 76°C (modified), AASHTO TP5	1.224 kPa	1.321 kPa
	Un-aged δ at 64°C (original) and at 76°C (modified), AASHTO TP5	85.4 deg	80.83 degrees
	RTFO-aged G*/sin δ, at 64°C (original) and at 76°C (modified), AASHTO TP5	2.64 kPa	3.030 kPa
	PAV-aged G*sin δ, at 25°C (original) and at 31°C (modified), AASHTO TP5	3201 kPa	1180 kPa
Bending beam rheometer (BBR)	Creep stiffness at −12°C, AASHTO TP1	139 MPa	124 MPa
	m-value at −12°C, AASHTO TP1	0.367	0.358
Solubility	ASTM D2042		99.71%
Separation test	% separation based on DSR test as per Equation 1, ASTM D-5976		5.4%

RTFO = rolling thin-film oven, PAV = pressure aging vessel.

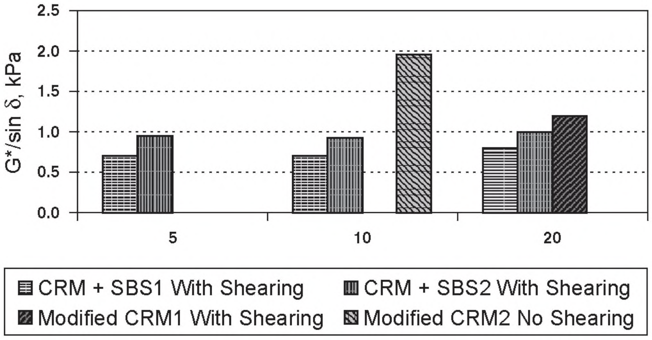


FIGURE 6 Performance of binder with modified CRM versus nonmodified CRM with added polymers.

sents a comparison between four polymer-added CRM materials that were prepared by two processes: the proprietary preprocessing and the straight addition of SBS polymer to the asphalt–rubber interaction. The interaction was extended to 20 h at 190°C to examine the binder quality under extended storage time. As can be seen, the binders made with the preprocessed CRM achieve higher parameters than the binders made with the straight addition of SBS. This is true particularly when more SBS content is added to the interaction than is used to preprocess the proprietary CRM; the case of "CRM + SBS2" versus "modified CRM1" as shown in Figure 6 and Table 1. Adding extra polymer alone to rubber is not that significant in improving binder quality unless combined with the proper CRM processing. Testing nonmodified CRM with added SBS shows the property stays and does not fall over time, and that is an improvement in the property development trend resulting from the addition of extra SBS polymer. Comparing results from the two materials, the proprietary preprocessed CRM and the CRM with added SBS, indicates that the addition of polymer is more effective through preprocessing. The effect of the preprocessing is that the binder forms a new networking system that continues to improve over time (*10*). Shearing the binder with preprocessed CRM has even accelerated property development and guaranteed continued improvements in both G* and δ.

BINDER COMPATIBILITY

Compatibility of the asphalt with modifiers is necessary for long-lasting pavement (*12, 13*). Incompatibility leads to premature product failure because of rapid aging and loss of properties including adhesion (*13*). Binder compatibility was evaluated with two techniques: the separation tube test and fluorescent microscopy. Results of the separation tests are given in Figures 7 and 8. The comparison was made with control material in which the modifier did not have any added virgin polymer components, GF-40, and also between the two CRMs with polymer modifiers, as interacted in Figure 6. From the figures, it is readily seen that the preprocessed CRM led to the lowest percentage separation, especially at longer interaction time. GF-40 material presented a typical CRM performance that would significantly separate when no shearing applies, particularly at 190°C interaction temperature. To illustrate the effectiveness of preprocessing in interacting with asphalt, no binder shearing was applied. All materials started the interaction with relatively high separation values. At the end of the interaction period, only preprocessed material achieved the desired separation quality that is close to that of virgin polymers. CRM with added polymer showed a slight improvement as compared with GF-40 material but still not fully interactive as compared with the preprocessed proprietary material. Adding extra polymer alone to rubber is not that significant in improving binder stability unless combined with the proper process. In this case, preprocessing showed significantly better compatibility and stability in storage than both CRM with added polymer and GF-40 materials.

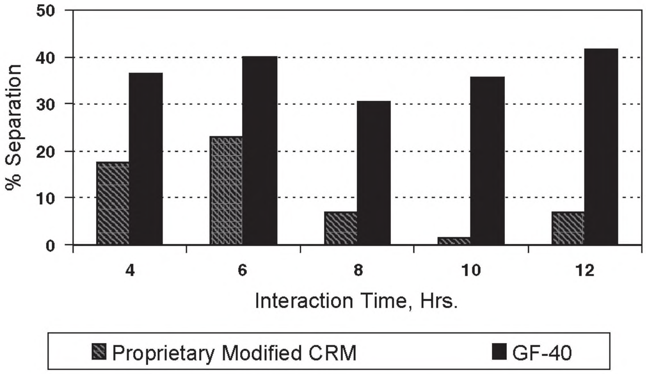


FIGURE 7 Stability of proprietary modified CRM versus nonmodified CRM.

Fluorescent microscopy has been used by the asphalt industry to examine the macrostructure of asphalt–polymer blends that are two-phase systems. The binder blend is illuminated by using an ultraviolet light. The polymer phase reemits a yellow light, whereas the asphalt phase does not give rise to observable fluorescence. The distribution of the polymer in the asphalt and consequently the compatibility of the blend could be visually assessed. The binders tested in this research may well be three-phase systems: asphalt, polymer, and CRM. Figure 9 shows images of the proprietary binder as tested in Figures 4, 5, and 6, the case of 190°C with mixing and shearing. Figure 9 indicates significant changes in the binder matrix as it converts from a liquid phase with polymer and rubber particles early in the process to a more well-distributed and finally a smooth matrix with small rubber particles and no visible polymer particles after 12 h. It is not clear whether the proprietary processing will convert CRM and the virgin polymer into one compound. The images show one type of particle, the yellow polymer particles, but no indication of CRM particles.

The image after 1 h shows signs of incompatibility, but the images after 6 and 12 h show a more compatible binder. No significant differences between the 6-h and 12-h interactions are observed. Modifier particles become more dispersed in the liquid phase over time.

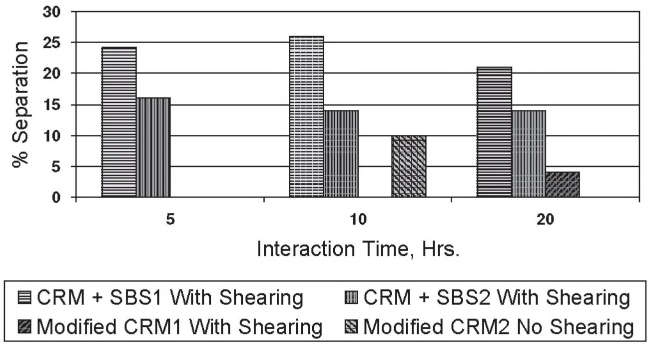
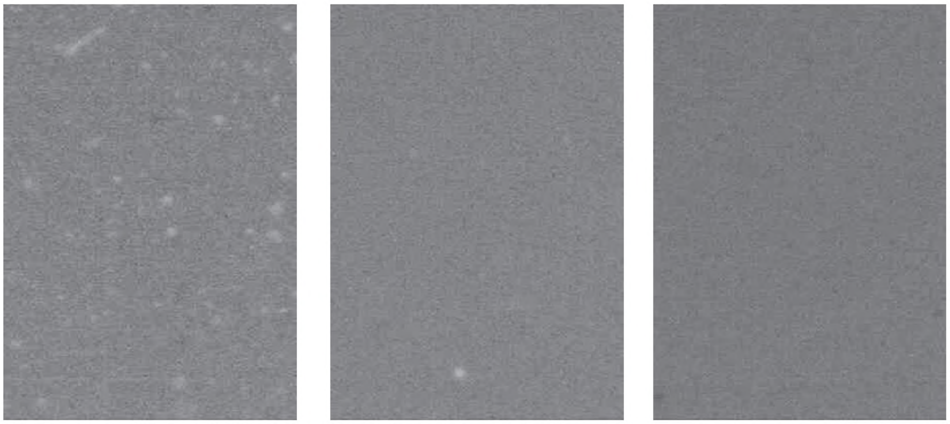


FIGURE 8 Stability of proprietary modified CRM versus nonmodified CRM with added polymers.



(a) (b) (c)

FIGURE 9 Fluorescent scanning of binder with modified CRM: (*a*) 1h, (*b*) 6 h, and (*c*) 12 h.

That agrees with the changes in the binder matrix of the nonmodified CRM, as illustrated in Figure 1. Unlike with nonmodified CRM, the property development continues as the modifier (CRM + SBS) particles disperse in the liquid phase, confirms that the improvement in performance properties is the result of changes in the liquid phase, and indicates that there is significant improvement in the binder cross-linking.

The proprietary process is flexible and can be tailored to produce binders with specific properties (*11*). The relatively high interaction temperature (190°C to 200°C) is selected to supply the system with the needed energy to break the vulcanized rubber bonds (*1*). High interaction temperature was not a concern to asphalt suppliers (*12*). In practice, binders are interacted with high-concentration modifiers at high temperatures and then mixed with original asphalt to produce the desired PG grade.

CONCLUSIONS

Processing rubber can be a key factor in improving the effectiveness of CRM to alter asphalt properties. Proper processing of CRM can be a viable alternative to virgin polymers. The research presented in this paper demonstrates the effectiveness of the CRM preprocessing in producing modifiers that are competitive with virgin polymers, with excellent compatibility, stability, and lower cost. The key advantage here is that while achieving better engineering properties of the modified binders, the compatibility and separation properties are highly desirable. The sensitivity of basic CRM to high interaction temperature does not apply to preprocessed CRM. The damaging effect of extended time at high temperature is reversed in the case of preprocessed CRM. Performance properties G* and δ continue to improve after 12 h at 200°C. Adding extra polymer alone to CRM binders is not that significant in improving binder quality unless combined with the proper processing.

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