

Comparison of Emulsion Residues Recovered by Forced Airflow and RTFO Drying

by

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Portions of this paper plus details of some recent developments in performance-based recovery methods developed by Dr. Takamura to be presented by C. Lubbers at the 2007 AEMA/ARRA/ISSA joint annual meeting. Full powerpoint presentation will be posted at AEMA-ARRA-ISSA websites following the annual meeting.

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ABSTRACT

At the last AEMA meeting in March 1999, we proposed a new residue recovery procedure for the asphalt emulsion. The procedure does not expose the emulsion to extreme heat, but dried in an ambient temperature under forced airflow, thus simulating a typical application condition. Water evaporates freely from the emulsion surface until the total solids are at above 90%. A sufficient amount of the residue samples can be prepared within 3 hours for the Superpave binder characterization. Optical microscope observation confirmed spontaneous formation of a microscopic SBR latex polymer network among asphalt particles upon drying.

M. Heslop and I. Walsh have been working for U.K. Highways Agency to develop a new residue recovery procedure based on the rotating thin film oven test (RTFOT) apparatus. Here, approximately 25g of an emulsion sample is placed in a polytetrafluoroethylene (PTFE) bottle containing a PTFE S-screw and placed in the carousel. A thin film of emulsion binder is rotated in PTFE bottles at a temperature of 85°C for 75 minute with a flow of heated nitrogen gas jetted over the binder film to evaporate the water phase. The emulsion binder is continuously disturbed by a rotating PTFE S-screw in the bottle, which is designed to draw material away from the entrance of the bottle.

We tested their RTFOT procedure and compared against our forced airflow drying using the SBR latex modified CRS-2 and CSS emulsions. The residue samples were taken every 15-minute during drying with RTFOT, and the Superpave dynamic shear rheometry was applied to characterize their physical properties. Test results demonstrate that these two different drying methods provide the emulsion residue having identical rheological properties. Optical microscope observation, again, confirmed the microscopic SBR polymer network in the residue by RTFO drying.

The emulsion residue of 130-140g can be obtained with the RTFOT procedure using eight bottles. This is enough to complete the Superpave binder characterization using the dynamic shear and bending beam rheometry. Our results indicate that the drying time could be reduced to below 30 minute and airflow instead of N₂ does not affect the residue property. Thus, the residue recovery by the RTFOT apparatus is the most promising method for obtaining the emulsion residue, which closely simulates typical application conditions.

BACKGROUND

[1] AEMA Round Robin Studies

The AEMA Materials Committee organized round robin studies on the vacuum distillation procedure and Superpave analysis of the neat and polymer modified asphalt emulsion. The studies involve five emulsion samples and four residue recovery procedures; vacuum distillation, 177°C distillation, 260°C distillation and ASTM evaporation. Both conventional and Superpave analysis of these residues were conducted by 11 participating laboratories. Analysis of these data quickly revealed that variations in measured values among laboratories were so large that Superpave grading is almost impossible.

This is demonstrated in **Figure 1** illustrating estimated rutting resistance temperature, T_r (temperature at $G^*/\sin(\delta)=1\text{kPa}$) of the emulsion residues for the latex modified CRS-2P and microsurfacing emulsions. Wide variation in T_r among different laboratory and distillation methods can be clearly seen in these figures. For example, the average $T_r = 71^\circ$ with the standard deviation of 3.1°C for the microsurfacing emulsion residue recovered by the vacuum distillation among 8 laboratories. However, T_r is only $66^\circ\text{C} \pm 2.1^\circ\text{C}$ (almost one PG grade difference) for the same emulsion but the residue recovered by the 260°C distillation as seen in **Figure 2**. In fact, T_r of the residues recovered by the 260°C distillation are consistently the lowest values for both microsurfacing and CRS-2P emulsions. The 177°C distillation and evaporation methods had large values of the standard deviation of 3.1 and 3.5°C , respectively.

Problems of reproducibility among laboratories and residue recovery methods were also confirmed with the conventional test methods, especially with the polymer modified asphalts. This is demonstrated with the measured softening points of the CRS-2 and latex modified CRS-2P(LM) in **Figure 3**. Results are relatively consistent for the unmodified emulsion. That is,

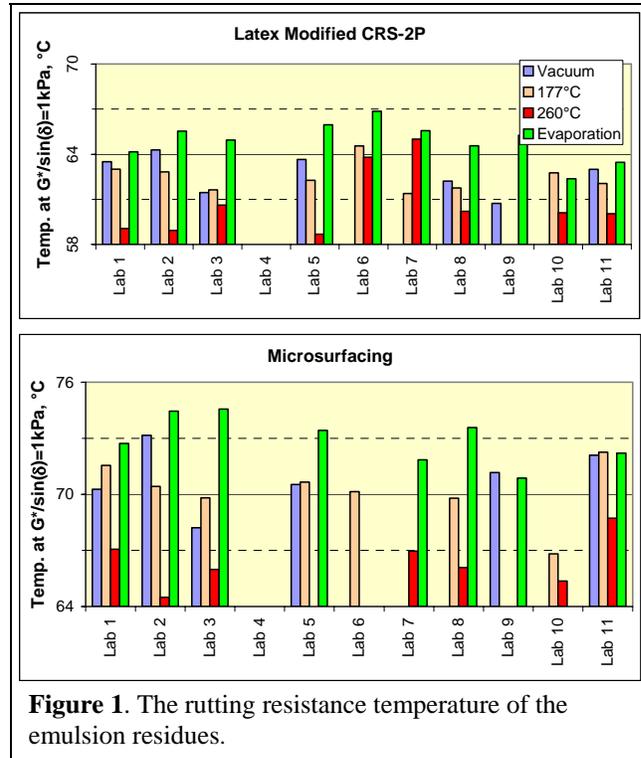


Figure 1. The rutting resistance temperature of the emulsion residues.

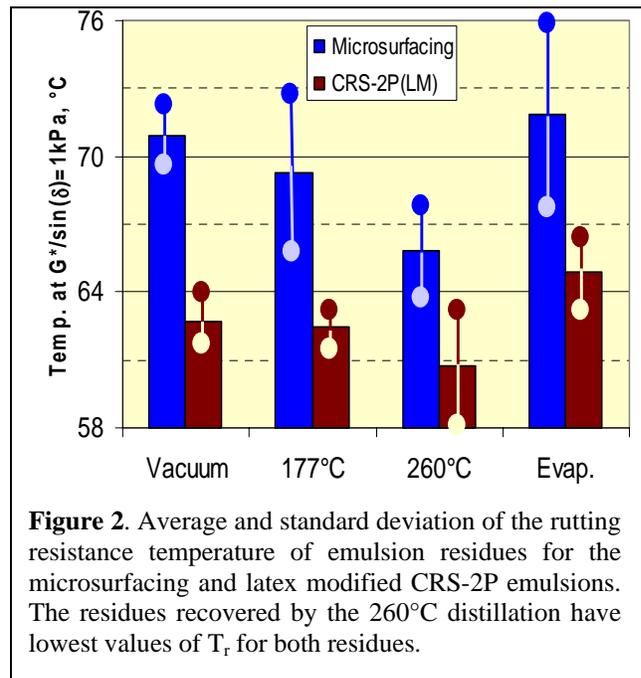


Figure 2. Average and standard deviation of the rutting resistance temperature of emulsion residues for the microsurfacing and latex modified CRS-2P emulsions. The residues recovered by the 260°C distillation have lowest values of T_r for both residues.

the average softening points of $44\pm 2.0^\circ\text{C}$ were obtained for the unmodified CRS-2 emulsion among 25 data points reported from nine laboratories. All four residue recovery methods provide relatively consistent softening points as seen in the figure.

However, this is not the case for the polymer-modified emulsion. The average softening points are $48\pm 4.4^\circ\text{C}$, showing the standard deviation is now more than doubled. The figure, again, clearly demonstrates lower values of the softening point with the residue recovered by the 260°C distillation, as observed with the rutting resistance temperature shown in **Figure 1** and **2**. The average softening point differs as much as 7°C between the residue recovered by the 260°C distillation and evaporation methods ($44\pm 2.9^\circ\text{C}$ vs $51\pm 2.9^\circ\text{C}$, respectively). We can not compare the absolute value of the average softening point of the CRS-2 and CRS-2P emulsions, since different asphalts were used for these two emulsions.

[2] Polymer Morphology Change during Residue Recovery

A small amount of samples were taken during the residue recovery process and observed under an optical microscope equipped with a hot stage. The stage temperature can be controlled from room temperature to above 200°C . A series of photomicrographs in **Figure 4** were taken for the sample at 1, 2 and 3 hours of distillation at 177°C .

A water droplet can still be seen in the sample taken at 1 hour of distillation. Only approximately 30% of water evaporated at this point. Latex polymers started to form discrete structure of tens of micrometer in diameter at this stage. Fine polymer networks can clearly be seen in the sample at 2 hours into the distillation, where more than 90% of water was evaporated. The polymer network observed here is similar to that formed under the hot mix condition, where the latex dispersion is added into molten asphalt of $165\text{-}170^\circ\text{C}$ under mechanical agitation.

Dramatic change in the polymer morphology can be seen during the last one hour of the distillation. Fine polymer networks are now coalesced to form macroscopic polymer globules as large as a few millimeters in diameter. It is not difficult to imagine that a minor difference in the distillation procedure, especially difference in the temperature and length of the final distillation stage, would cause variation in the polymer morphology. This would affect physical properties of the recovered residue, thus explaining the poor reproducibility among laboratories.

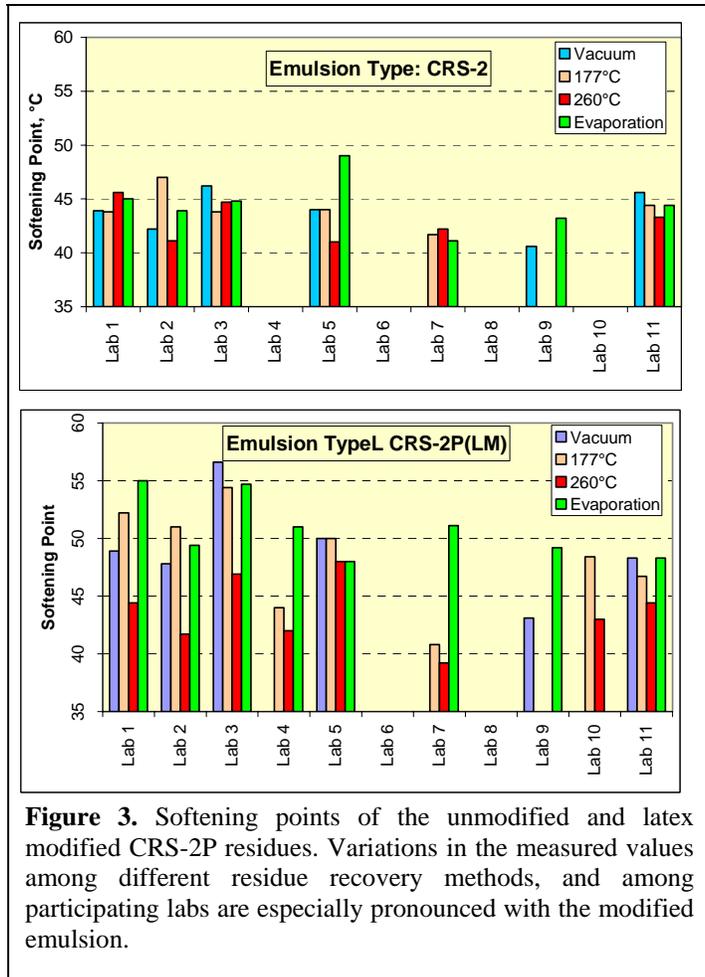
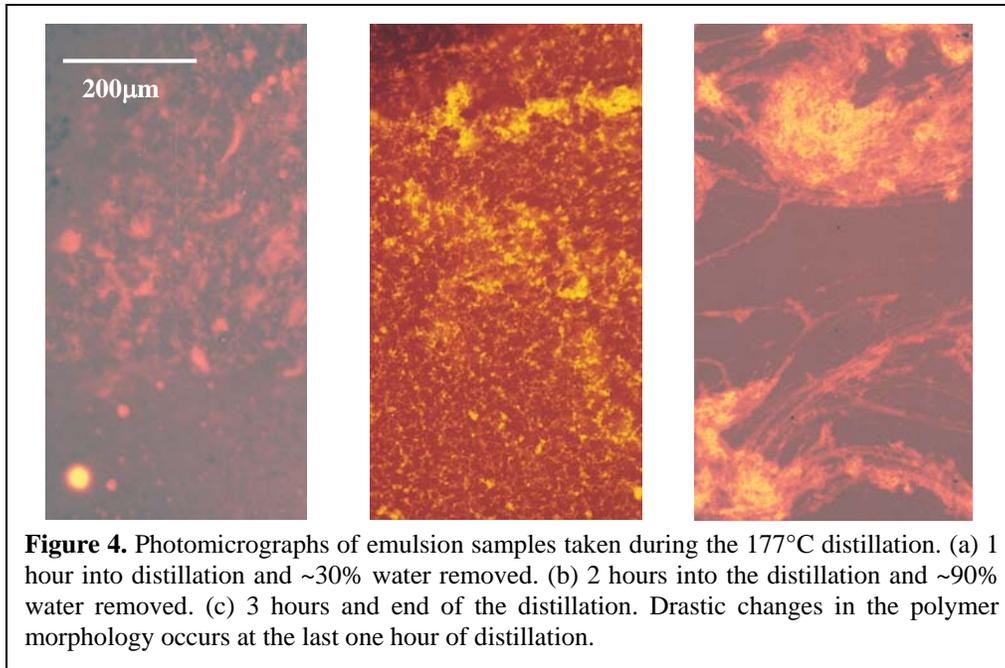


Figure 3. Softening points of the unmodified and latex modified CRS-2P residues. Variations in the measured values among different residue recovery methods, and among participating labs are especially pronounced with the modified emulsion.

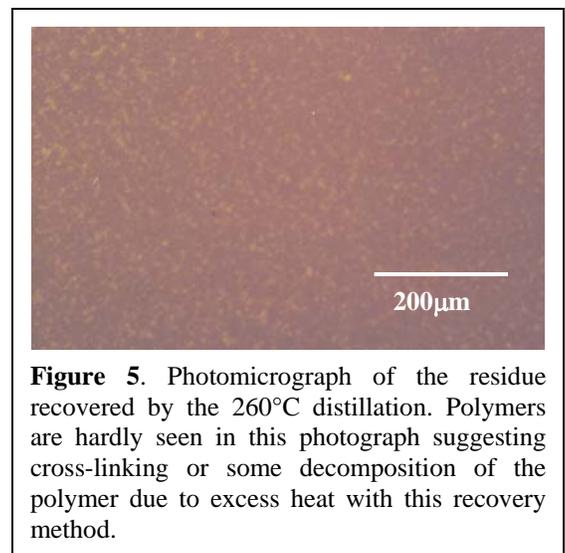
Optical microscope observation was also performed for the residue recovered by the 260°C distillation. The same asphalt emulsion was used for the 177°C and 260°C distillation for this microscope observation, thus eliminating potential effects of the asphalt property on the polymer morphology. It was very difficult to observe the polymer in the residue recovered with this 260°C distillation. Only a few micrometers in diameter polymer particles can be seen as shown in **Figure 5**. This could be due to cross-link or some decomposition of the latex polymer under excess heat with this distillation.



[3] Latex Polymer Morphology in the Cured Microsurfacing

A sample of the microsurfacing was prepared by mixing 100g aggregates, 12g of 65% asphalt emulsion containing 3% **BUTONAL® NS198**, 10g water and 1g cement. The aggregate-asphalt emulsion mixture was cured as the standard procedure for the cohesion measurement; cured in a metal ring of 50mm diameter and 5mm deep. The mixture developed cohesion of above 25kg-cm after one-hour curing.

The dried microsurfacing specimen was further cured for one month and fractured to expose a fresh cross-section. It was, then, treated with OsO_4 and the asphalt was extracted with the MEK (methyl ethyl ketone) solvent as described by D.L. Wolfe et al¹. The treatment with OsO_4



makes the SBR polymer insoluble to the organic solvent and also improves the contrast for the scanning electron microscope, SEM, observation. It was rather surprising to notice that the specimen maintained the original structure even after the complete extraction of the asphalt.

A series of SEM photographs of the fractured surface were taken and shown in **Figure 6**. These photographs, especially (b) and (c) clearly demonstrate the honeycomb structures of the SBR polymer formed around asphalt particles. Some latex polymers also adhere on the aggregate surface as seen in (c). It is especially important to realize that the latex polymers remain in the aqueous phase, not in the asphalt, and transform to continuous polymer film during the curing process. Since the cement particles also remain in the aqueous phase, the flexible polymer-cement complex creates these honeycomb structures. In contrast, the honeycombs made only with the cement would be very brittle and this would be the case when the polymer is present in the asphalt phase. Advantages of this flexible honeycomb structure with the SBR latex will be discussed later during the forced air drying of the emulsion residue.

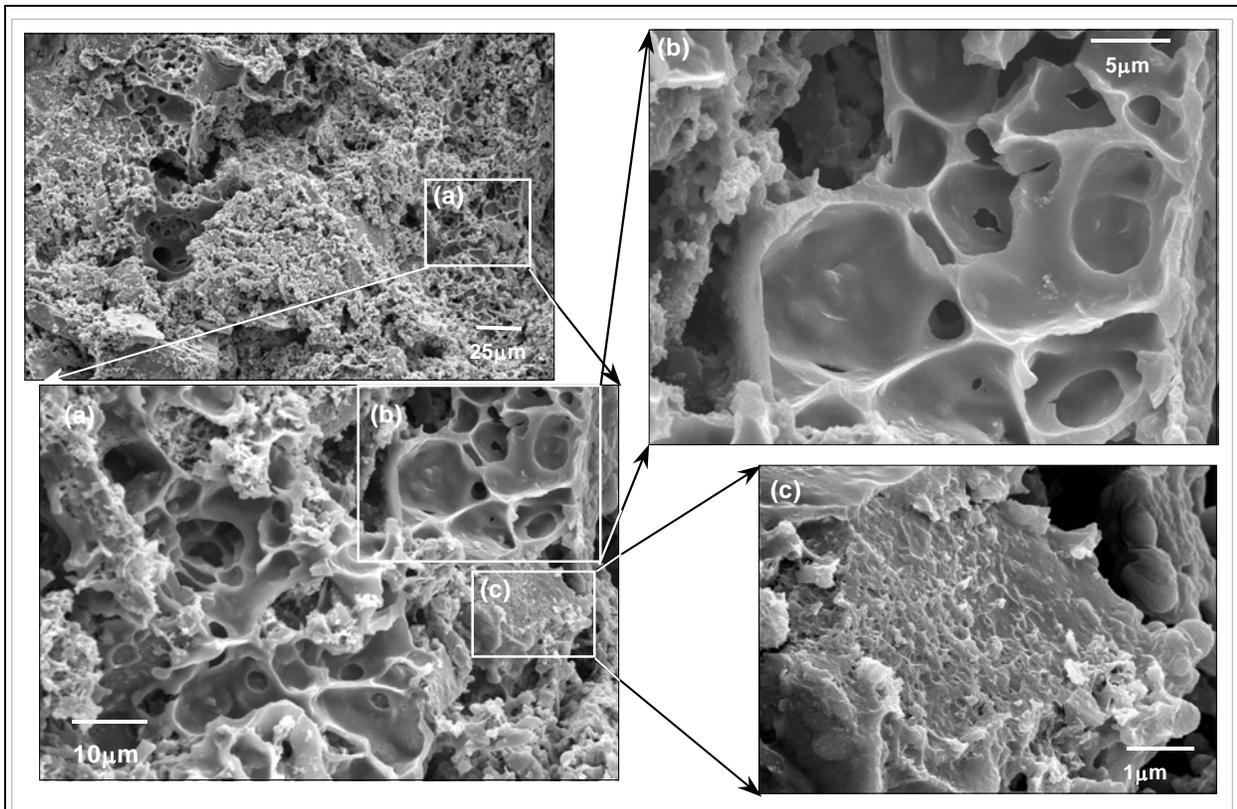


Figure 6. A series of scanning electron microscope photographs of the cured microsurfacing specimen demonstrating (b) SBR polymer honeycomb formed around asphalt particles. (c) Some polymers also adhere on the aggregate surface.

[4] Mechanism of the Polymer Honeycomb Formation

SEM photographs shown in **Figure 6** indicate that the modified asphalt with the SBR latex is not just an emulsion of the polymer modified asphalt, but rather the asphalt emulsion containing dispersed latex particles in the aqueous phase. This is schematically shown in **Figure 7**.

Menisci of water containing latex particles (and cement particles for the microsurfacing) form among asphalt particles when water starts to evaporate from the emulsion. The latex particles are partially destabilized during this process due to the increased electrolyte concentration in the aqueous phase by evaporation and the reduced surfactant concentration by adsorption to aggregate surface. The latex particles spontaneously transform to continuous polymer film surrounding asphalt particles².

The SBR latex for the asphalt modification is designed to create the polymer film without coagulum formation; warranting early cohesion development. Majorities of latex particles migrate together with water and present in the menisci, thus act as “spot welding” of asphalt particles to ensure the maximum binding power as shown in **Figure 7**. It is very important to realize that the asphalt emulsion should not break (coalesce) during this process to form the finest honeycomb structure.

The polymer honeycomb formation in the cured asphalt binder was successfully confirmed by optical microscopy. Here, the asphalt emulsion modified with **BUTONAL® NS198** was mixed with Portland cement to create an asphalt binder for the microsurfacing. The emulsion does not break, but rather becomes viscous after 2 minutes of mixing, resembling the mixing (open) time of the entire microsurfacing formulation. The emulsion-cement mixture was cured in the same metal ring as the cohesion measurement, forming an asphalt residue approximately 2mm thick and 5cm diameter.

The cured specimen was fractured and the freshly exposed surface imaged with the fluorescence contrast method using a Confocal Laser Scan Microscope (Leica TCS4D). **Figure 8** is a composite

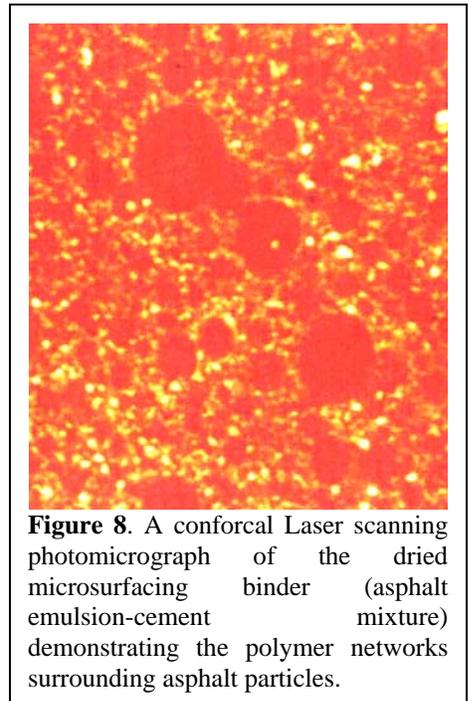
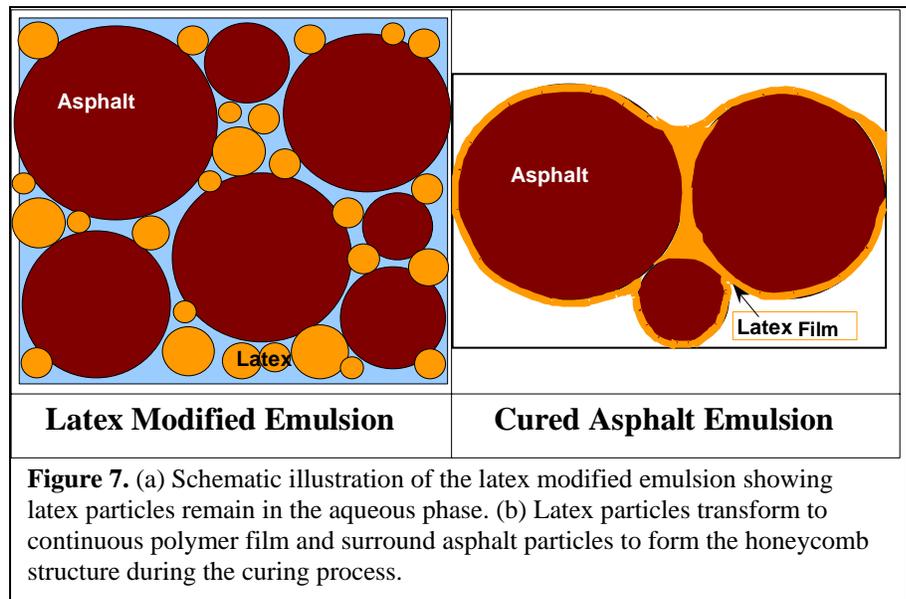


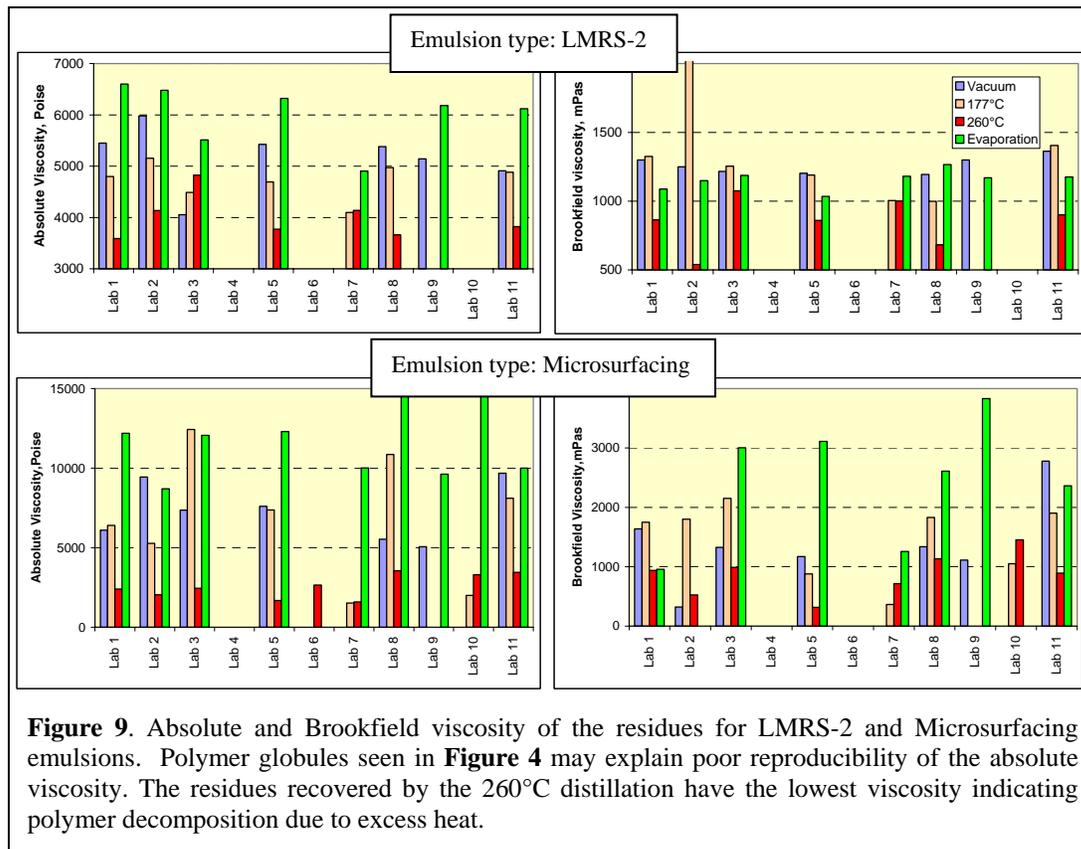
image of 32 thin sections illustrating approximately 50 μ m deep. Polymer networks surrounding asphalt particles are clearly seen. The presence of a few 20-50 μ m diameter asphalt particles indicates some coalescence caused by the cement addition.

We previously reported³, photomicrographs demonstrating latex polymer networks in the cured CRS-2 emulsion (without the cement) and during the chip seal application. It was surprising to observe that microscopically fine polymer networks (individual polymer threads of less than 1 μ m in diameter) remained intact even when they are observed under the hot stage at 180°C.

NEED FOR NEW RESIDUE RECOVERY METHOD

[1] Why Do We Need A New Recovery Method?

It is not difficult to understand that the emulsion residues having two distinctly different polymer morphologies shown in **Figure 4** and **Figure 5** would exhibit different physical properties. Presence of the polymer network shown in **Figure 4b** would increase the asphalt viscosity. In contrast, the polymer contribution to the asphalt viscosity would be the minimum when it is dispersed as non-swollen fine particles as seen in the residue recovered by the 260°C distillation (**Figure 5**). The optical microscope observation indicated that the macroscopic polymer networks were preserved in the residue recovered by the evaporation method, presumably due to the lower temperature.



Therefore, it is not surprising to observe that the residues recovered by evaporation and 260°C distillation methods show distinctly different values of absolute and Brookfield viscosity as shown in **Figure 9**. The absolute viscosity is measured using a capillary viscometer. Presence or absence of macroscopic polymer globules would significantly affect the reproducibility of the measured viscosity. This is indeed the case and it is impossible to perform meaningful statistical analysis of data shown in **Figure 9**.

All results discussed above demonstrate the need for a new residue recovery procedure. This procedure should not use excess heat to evaporate water from the emulsion, but still able to produce the residue within a few hours. It should reproduce a fine polymer network observed in the cured asphalt emulsion at the application condition. At the same time, it should be easy to perform and results should be highly reproducible. A new residue recovery method based on the forced air-drying was developed at BASF Corporation Charlotte Technical Center to meet these requirements.

[2] Ambient and Forced Air Drying

(a) Drying Rate: Conventional residue recovery methods assume that the emulsion dries too slowly at ambient temperature, thus heat is required to accelerate water evaporation from the emulsion. We found this is not the case: water evaporates freely from the emulsion surface and a residue enough to perform the SHRP binder characterization can be produced within 4-5 hours of drying. This is demonstrated in **Figure 10**.

Here, the drying rate of the neat and SBR latex modified emulsion was determined by measuring the weight of water evaporated. For this test, a 10g sample of the emulsion was poured into a stainless steel pan measuring 14cm in diameter and 1cm deep. The asphalt emulsion covers the entire pan surface and the pan was held at a room temperature of approximately 22°C. The weight loss was determined as a function of time. The CRS-2 emulsion used for this study had an average particle size of 2.0 μ m in diameter and total solids of 65%. An AC-5 asphalt was used for the base asphalt and 3% **BUTONAL**[®] **NS198** for the polymer modification.

As seen in **Figure 10**, weight loss is a linear function of time for the first 80 to 100min. This indicates water evaporate freely from the emulsion surface during this initial drying period until the total solids of approximately 90% are achieved. No skin formation was observed during this period. At the higher total solids, the water flow beneath the surface becomes restricted, resulting in a reduced drying rate. This reduced drying rate extends the time to complete dryness by a further 90 to 120 minutes. Presence of latex particles in the modified asphalt does not affect the drying rate of the asphalt

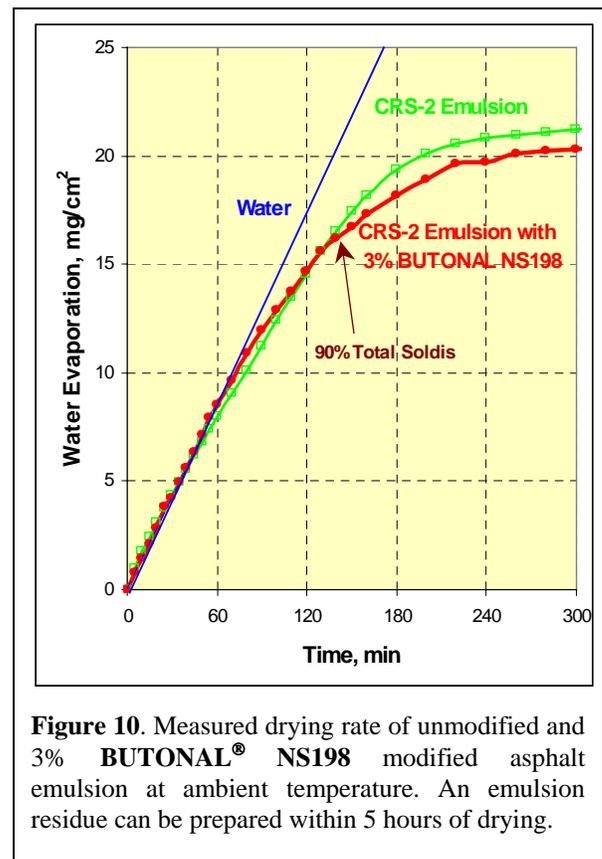


Figure 10. Measured drying rate of unmodified and 3% **BUTONAL**[®] **NS198** modified asphalt emulsion at ambient temperature. An emulsion residue can be prepared within 5 hours of drying.

emulsion. **Figure 10** also includes the measured drying behavior of distilled water. The drying rate of the water is almost identical to that of the emulsion for the first 70min. In this experiment, we obtained an emulsion residue within 5 hours.

We also tried to dry the emulsion at slightly elevated temperature of 60°C to enhance the water evaporation. In contrast to our expectation, the drying rate was drastically reduced due to thin asphalt skin formation on the emulsion surface. The total solids of the asphalt emulsion reached only to below 85% even after 1 day of drying.

We soon realized that generating an airflow above the emulsion surface could significantly enhance the drying rate at ambient temperature. We developed a prototype forced airflow residue recovery unit. The drying unit is a chamber 46cm high, 33cm wide and 28cm deep, containing two shelves for the emulsion sample (see Appendix). Two electric fans are attached at the back of the chamber to dry air through it, creating approximately 60m/min linear air velocity. The two shelves are located at a level just below the fans to allow optimum airflow over the surface of the samples. The fans are placed 16cm behind the shelves to assure uniform airflow. The unit is operated at the room temperature.

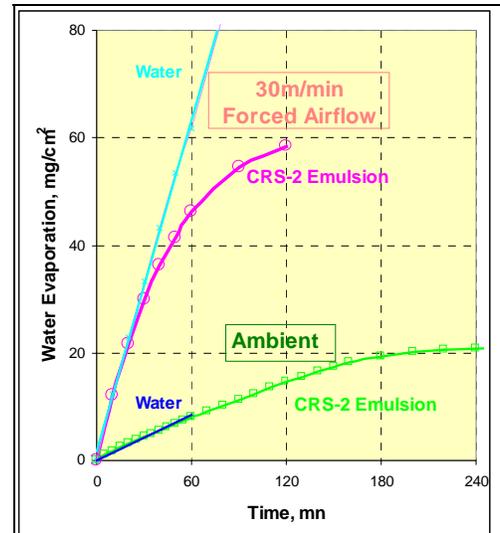


Figure 11. Ambient and forced airflow drying of the CRS-2P emulsion. The emulsion dries nearly 5x faster under the forced airflow.

Results of the emulsion drying experiment using this forced airflow unit are shown in **Figure 11**. Approximately 60g of an asphalt emulsion is placed in a shallow silicone tray having internal dimensions 5mm deep by 175mm wide. The tray and contents are then placed into the forced air-drying unit for 5 hours. More than 90% of water evaporates within the first three hours, forming the emulsion residue of approximately 2mm thick. The emulsion dries roughly 5 times faster in this unit as shown in **Figure 11**. The dried residue can simply be peeled from the tray for later binder characterization.

(b) Dynamic Shear Rheometry for CRS-2 Emulsion:

Rheological properties of the emulsion residue were determined using a dynamic shear rheometer, DSR, (Rheometrics RDA-700) at temperature between 25 and 90°C. Samples of asphalt residue containing 3, 5, and 10% **BUTONAL® NS198** were prepared using the procedure described above. Results of measured $G^*/\sin(\delta)$ at 10 rad/s for these samples are summarized in **Figure 12**, and compared with unmodified asphalt.

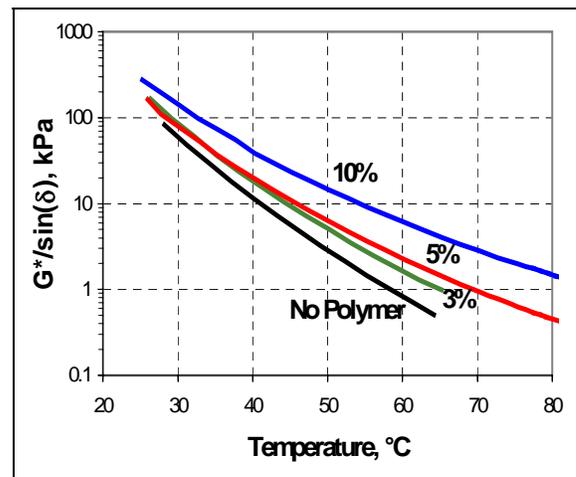


Figure 12. $G^*/\sin(\delta)$ value of the emulsion residues containing various amounts of **BUTONAL® NS198**. Two PG grades of improvement could be achieved with slightly above 5% latex polymer.

Sharp increase in $G^*/\sin(\delta)$ can be seen with the polymer modification at any given temperature, but especially at above 50°C. According to SHRP (Strategic Highway Research Program), the asphalt binder will provide sufficient rutting resistance at $G^*/\sin(\delta) > 1\text{kPa}$. Hence the benefit of the SBR latex polymer modification can clearly be seen on the rutting resistance. The rutting resistance temperature (temperature at $G^*/\sin(\delta) = 1\text{kPa}$), T_r , increased from 58°C to 65, 69 and 88°C with addition of 3, 5, and 10% **BUTONAL® NS198**, respectively. These data indicate two PG grade improvement could be achieved with slightly above 5% SBR polymer with this asphalt.

(c) Microsurfacing System: The cement in the microsurfacing formulation plays a critical role on the mixing time, cohesion development and final performance of the paved road. Thus it is essential to evaluate the emulsion residue containing a desired amount of the cement.

The drying rate of the microsurfacing emulsion, with and without cement, was tested and shown in **Figure 13**. The emulsion was dried under the forced-airflow. Both emulsions were modified with 3% **BUTONAL® NS198**. The figure demonstrates that the microsurfacing emulsion dries much slower than the CRS-2 emulsion. This is especially the case with the cement addition, and asphalt content reached only 75% after 4 hours of drying. This emulsion takes at least one day to fully dry under the forced airflow.

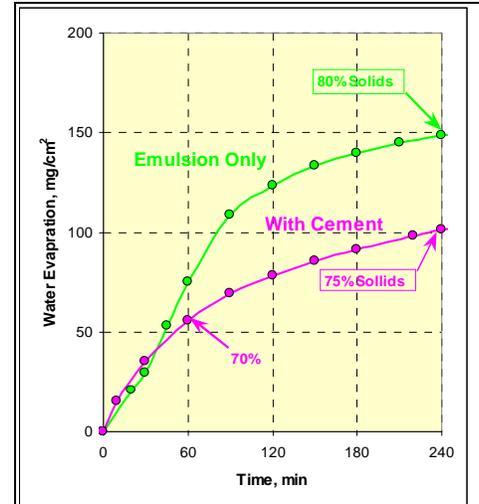


Figure 13. The drying rate of the microsurfacing emulsion. The Portland cement addition slows the drying rate of this emulsion.

The dynamic shear rheometer was again used to determine the rheological properties of the emulsion residue. Although the drying rate is slower with the cement addition, the mechanical strength develops quickly enough for the DSR measurement within a few hours. The rutting resistance temperature, T_r , was determined as a function of the curing time for the emulsion only, emulsion plus cement, and emulsion, cement and 3% **BUTONAL® NS198** to the asphalt. A typical microsurfacing formulation consists of 100g aggregates, 12g of 65% asphalt emulsion containing 3% latex polymer, 10g water and 1g Portland cement. The formulation used for this study is the same but without the aggregate and 10g water.

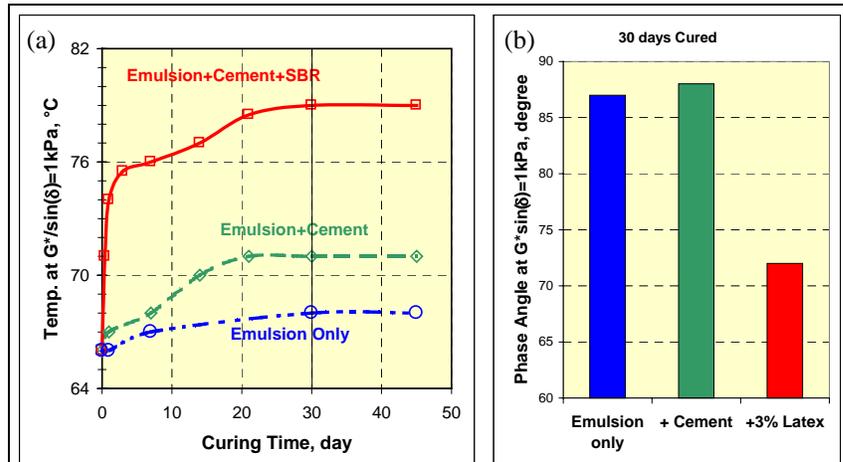
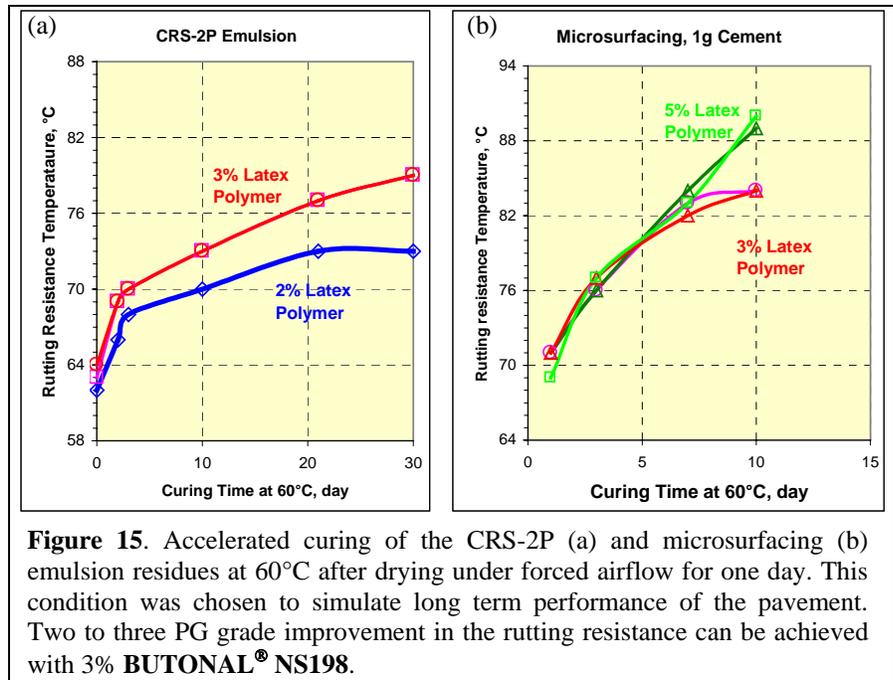


Figure 14. The rutting resistance temperature of the microsurfacing emulsion, emulsion plus cement and the emulsion, cement and 3% SBR latex. Two PG grade improvement can be observed with the polymer-cement system, which maintains elasticity of the residue as seen with the low measured phase angle.

As seen in **Figure 14a**, the emulsion was made with a PG64 grade asphalt (AC-20) and the T_r increased slightly from 66°C to 68°C after one month. The cement addition gives gradual increase in T_r to 71°C within three weeks. This increase in T_r is mostly due to stiffening of the asphalt as the phase angle of the residue increased from 82° to 88° at T_r . The value of T_r showed a rapid increase to 76°C within the first 3 days of curing when 3% of the SBR latex is also present in the mix. Two PG grade improvement in the rutting resistance materializes after two weeks of curing. The phase angle at T_r remained nearly constant value of 77-78° throughout the curing, confirming the SBR modified asphalt binder maintains the elasticity. Differences in the phase angle of these three samples are summarized in **Figure 14b**.

(d) Accelerated Curing: The forced airflow drying creates the fine SBR latex polymer network in the asphalt binder. To evaluate potential benefits of this polymer morphology for the pavement performance during its lifetime, an accelerated curing test was designed. Here, the emulsion was dried one day under the forced airflow unit, and transferred into an oven at 60°C simulating pavement temperature during daytime. The DSR measurement of the SBR latex modified CRS-2P and the microsurfacing mixture were selected for this study and results are shown in **Figure 15a** and **b**, respectively.



Two different latex polymer levels of 2 and 3% for the CRS-2P, and 3 and 5% for the microsurfacing mix were studied. Sharp increase in the rutting resistance temperature, T_r , was observed for the CRS-2P residue within 3 days of the accelerated curing at 60°C. This was followed by steady increase in T_r until 20-30 days of curing. The neat asphalt used for this emulsion was PG58 grade and two and three PG grades improvement was achieved with 2 and 3% BUTONAL® NS198 after 20 days, respectively. The hot mix addition of similar SBR latex in the asphalt provides only one PG grade improvement with the same polymer level. This demonstrates advantages of the fine polymer network in the emulsion residue.

Improvement in the rutting resistance is even significant with the microsurfacing mix, presumably due to the cement in the mixture. Three PG grade improvement with 3% latex polymer takes only 10 days of curing as seen in the **Figure 15b**. Results demonstrated here explain the rut filling capability of the microsurfacing system. The data may also indicate that use of softer asphalts should be explored for the microsurfacing to improve the cold temperature flexibility of the pavement.

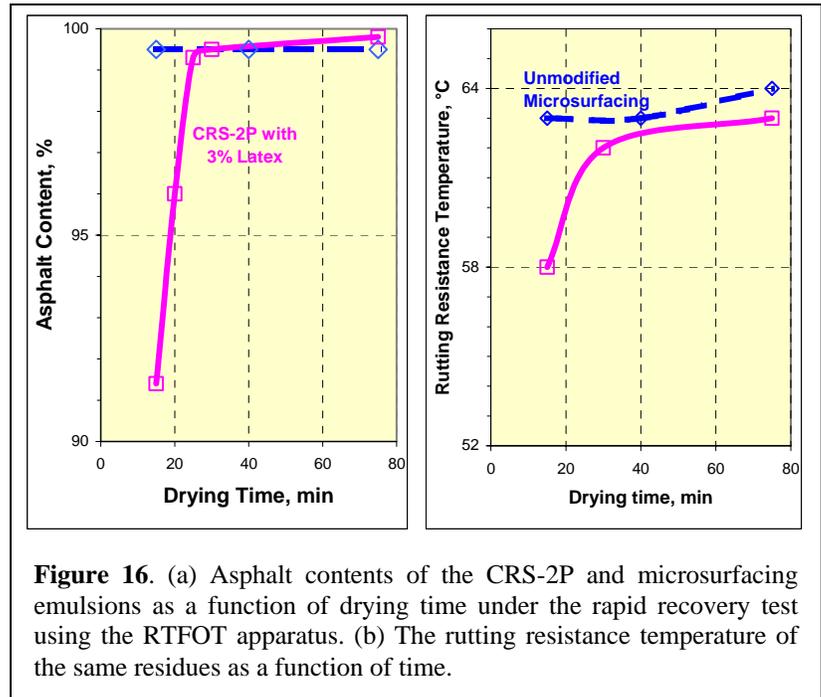
Figure 15 includes two sets of data for each latex levels demonstrating excellent reproducibility of the residue recovery as well as following accelerated procedures.

[3] Rapid Recovery Test with Rotating Thin Film Oven

The residue recovery with the forced air drying represent the application condition, thus the ideal procedure for the research and development laboratory. However, it takes one day to prepare the residue and may be too slow for quality control labs at asphalt production sites. M. Heslop and I. Walsh have been working for U.K. Highways Agency to develop a new rapid residue recovery procedure based on the rotating thin film oven test, RTFOT, apparatus. Here, approximately 25g of an emulsion sample is placed in a polytetrafluoroethylene, PTFE, bottle containing a PTFE S-screw and placed in the carousel. A thin film of emulsion binder is rotated in PTFE bottles at a temperature of 85°C for 75min with a flow of heated nitrogen gas jetted over the binder film to evaporate the water phase. The emulsion binder is continuously disturbed by a rotating PTFE S-screw in the bottle, which is designed to draw material away from the entrance of the bottle. Since we can mount 8 bottles in the RTFOT carousel, the test will provide 120-130g emulsion residue within 75 minutes.

We conducted a preliminary evaluation of this rapid recovery test using an unmodified microsurfacing and CRS-2P with 3% **BUTONAL® NS198**. A small amount of samples were taken during the drying process and the asphalt content and T_r were determined as discussed above. As seen in **Figure 16a**, the microsurfacing emulsion dried within 15min with this rapid recovery test. Values of measured T_r are also consistent for all three samples taken at the drying time of 15, 40 and 75 minutes.

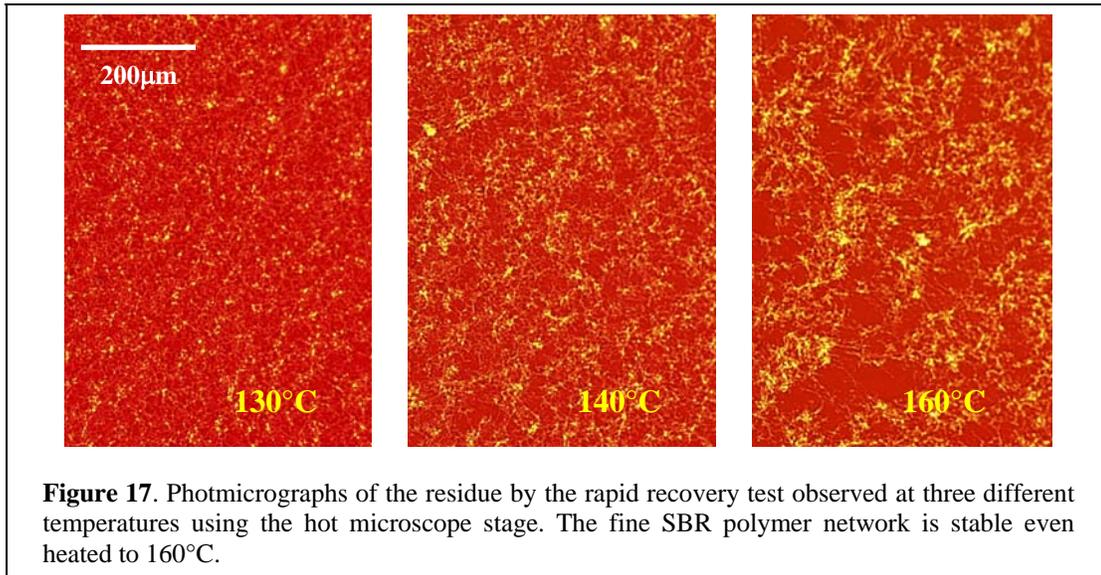
The latex modified CRS-2P dried slightly slower than the unmodified microsurfacing emulsion and took 30min to reach near 100% asphalt content. The measured T_r exactly follows the observed drying trend.



Separate tests using the airflow were also conducted. Based on these test results, we could draw following conclusions:

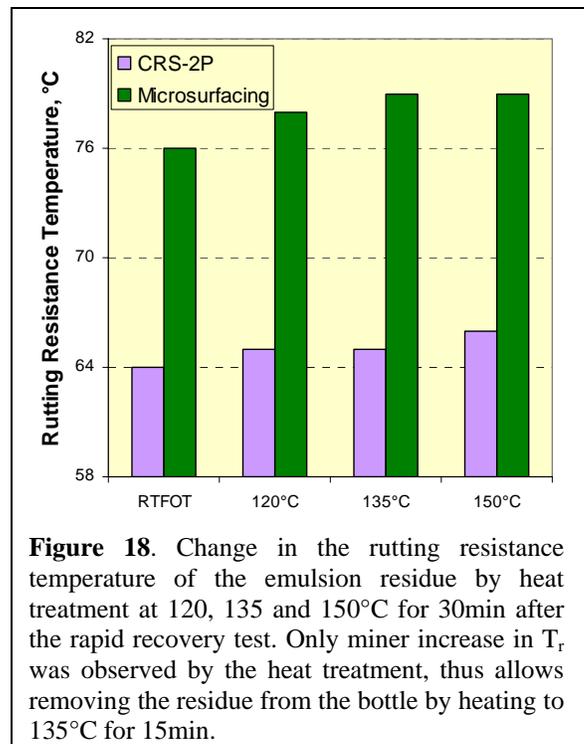
1. Air instead of N_2 gas can be used without affecting the residue property.
2. Emulsions dry quickly with this procedure.
3. The emulsion residue, especially the one modified with the SBR latex, adheres to the PTFE bottle and the S-screw surfaces. This makes almost impossible to remove the residue from the bottle.
4. Measured T_r of the emulsion residues with the rapid recovery test and the forced air-drying agree each other.

Based on the above preliminary conclusions, we decided to use a glass bottle with a metal bar and conducted additional tests. After completing 75min of drying, the bottle was placed in an oven of 135°C for 15min and the molten residue was removed as the RTFOT procedure. This procedure also allows us to prepare samples for additional tests, such as the bending beam rheometer, softening point, and elastic recovery, etc.



Before accepting this procedure, we need to prove that the heat treatment proposed here does not cause any adverse effects on residue properties. This was done by heating the emulsion residues at three different temperatures for 30min. The CRS-2P and the microsurfacing emulsions were used for this study and both emulsions were modified with 3% **BUTONAL® NS198**. The optical microscope observation was also conducted to monitor potential changes in the polymer morphology during this heat treatment.

Photomicrographs shown in **Figure 17** demonstrate the emulsion residue maintains its fine SBR polymer network even at 160°C. Results shown in **Figure 18** suggest that the heat treatment of the emulsion residues at 120, 135 and 150°C for 30min causes only minor increase in T_r . Therefore, the above-discussed procedure of heating the residue at 135°C for 15min would be acceptable. As discussed, this procedure is essential to prepare samples for all binder characterizations. It also helps removing trapped air bubbles and minute residual water from the residue recovered both by the rapid recovery test and forced airflow drying.



[4] Conventional Tests

Examples of the conventional test results of the emulsion residue recovered by the rapid recovery test are shown in **Figure 19** and **20** for the CRS-2P and microsurfacing emulsions, respectively. Results for the unmodified and modified with 3% **BUTONAL® NS198** are compared in these figures. The residue recovery and the measurement were repeated twice to demonstrate reproducibility.

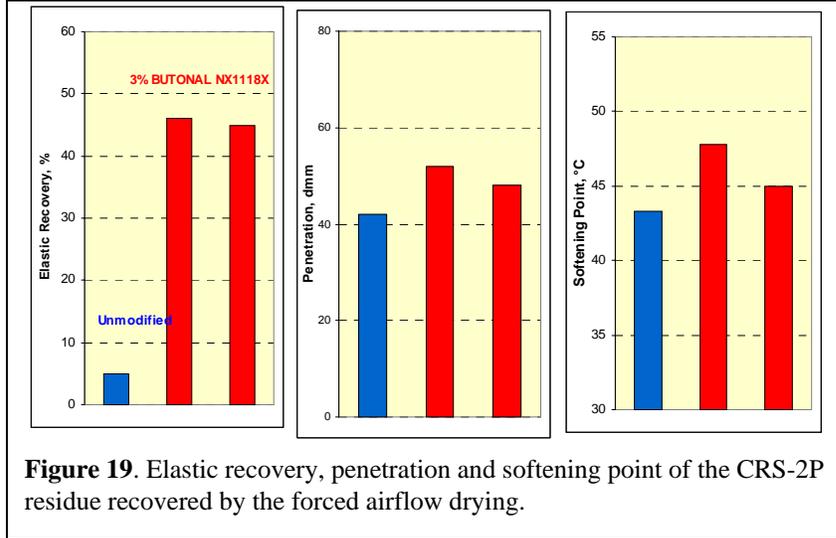


Figure 19. Elastic recovery, penetration and softening point of the CRS-2P residue recovered by the forced airflow drying.

The CRS-2P residue for this particular study was recovered by the forced airflow drying. The elastic recovery measured at 4°C was only 5% for the unmodified emulsion residue and increased sharply to above 45% with 3% addition of **BUTONAL® NS198**. Reduction in the penetration from 142dmm to below 130dmm was also confirmed. Surprisingly however, the polymer modification resulted in only minor increase in the softening point with this particular asphalt.

Similar results for the microsurfacing emulsion residue were shown in **Figure 20**. These residues were with 3% **BUTONAL® NS198** but without the cement addition, thus do not

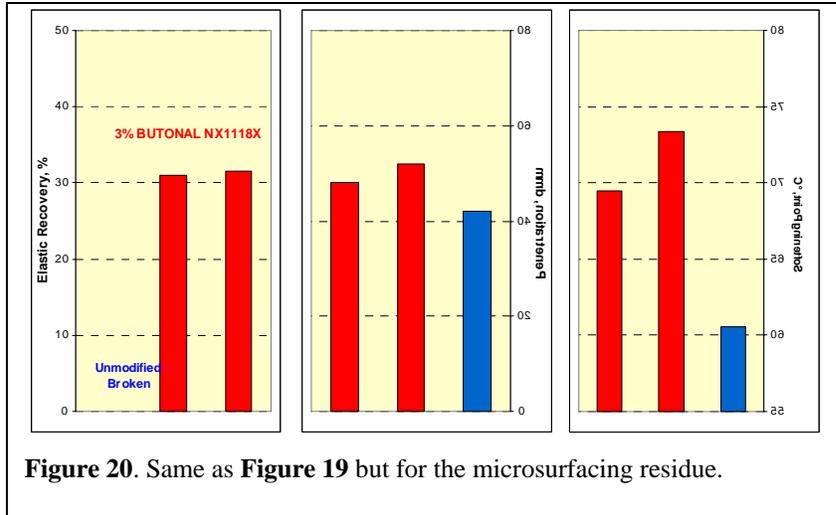


Figure 20. Same as **Figure 19** but for the microsurfacing residue.

exactly reflect the application condition. The short drying time at 85°C and constant mechanical stress by the metal bar could have abnormal influence on the cement curing and may not simulate the application condition.

The base asphalt used for this emulsion was very brittle at 10°C and broken below specific elongation for the elastic recovery measurement. Above 30% elastic recovery was obtained with 3% latex polymer addition. Effect of the polymer modification on the penetration is limited for this hard asphalt, but more than 10°C increase in the softening point was observed with the polymer modification.

[5] Potential Problems with Rapid Recovery Test

As many as a dozen microsurfacing emulsions were tested with the rapid recovery test and all of them dried within 30min. However, this was not the case with the CRS-2P emulsion. Some of them reached only slightly above 95% after 75 minutes drying because of trapping macroscopic water droplets in the residue. This water residue did not evaporate even after 2 hours of drying. The slow drying behavior of these emulsions is illustrated in **Figure 21**. The residual water in these residues unexpectedly boils when the bottle is removed from the oven for preparation of the conventional tests. This creates potential hazard.

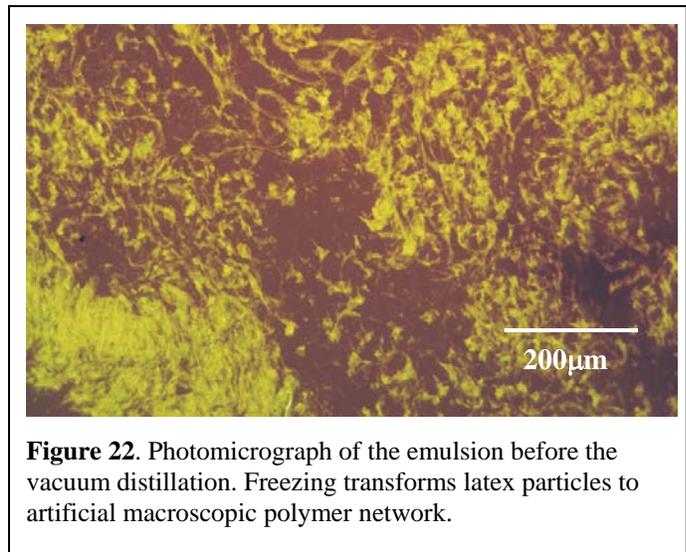
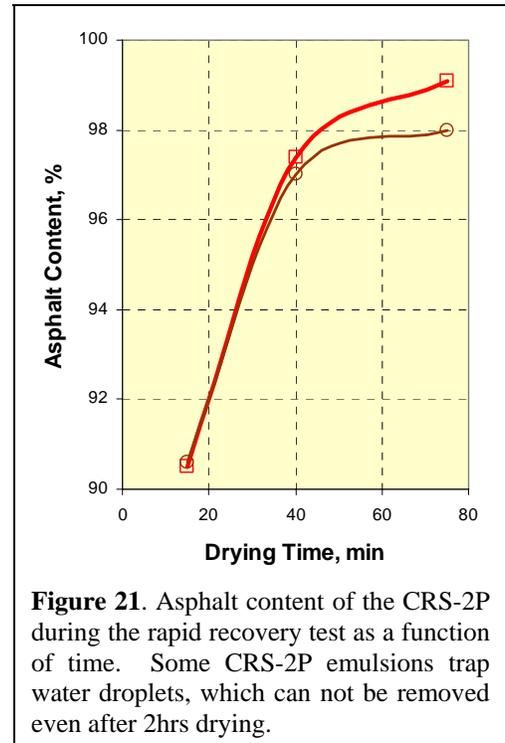
This can be avoided by immediately transferring slightly softened residue into a container and gently agitate during heating to 135°C. These slow drying emulsions are less stable and skin during the rapid recovery test at 85°C. We need additional studies to fully understand and find solutions to this problem.

CONCLUSION

The modified emulsion with the SBR latex is not just an emulsion of the polymer-modified asphalt, but rather the emulsion containing dispersed latex particles. Latex particles remain in the aqueous phase and spontaneously transform to continuous microscopic polymer film surrounding asphalt particles upon curing.

In the microsurfacing mix, Portland cement particles are also dispersed in water, thus honeycomb structures of flexible cement-polymer complex present among aggregates and within the entire pavement. This is the reason of the excellent rut filling capability of the microsurfacing mixture. The honeycombs made only with the cement would be very brittle and this would be the case when the polymer is present in the asphalt phase.

The forced air drying procedure was developed for the residue recovery. Here, the emulsion is dried for 5-6 hours at ambient temperature. The optical microscope observation confirmed the microscopic polymer network in the residue. This polymer network is stable even at 180°C. The dynamic shear rheometry and a series of conventional tests were conducted for residues recovered with this procedure. This forced air-drying



is an ideal method for the research and development laboratory since the residue recovered closely simulates application conditions.

In contrast, the distillation and evaporation procedures apply excess heat to the emulsion, causing significant changes in the polymer morphology. Cross-link formation and decomposition of the polymer may also occur. The heat damage to the polymer is most pronounced with the 260°C distillation. Although the vacuum distillation uses the lowest temperature among four residue recovery methods studied by AEMA round robin studies, freezing of the emulsion creates artificial macroscopic polymer structure as shown in **Figure 22**.

The rapid recovery method using the RTFOT apparatus could be for the quality control lab at the emulsion production side. The residue can be produced for 75min drying at 85°C. The microscopic polymer network was also observed in the residue. All microsurfacing emulsions tested here dried within 30 min with this test. However, some CRS-2P emulsions dry slower and trap water droplets, which suddenly boil during subsequent testing. Further studies are needed to solve this problem.

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APPENDIX: Schematic illustration of a forced airflow residue recovery unit.

