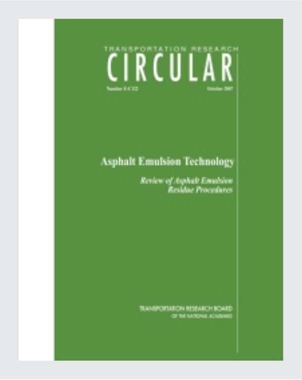

Asphalt Emulsion Technology: Review of Asphalt Emulsion Residue Procedures

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Asphalt Emulsion Technology

Review of Asphalt Emulsion Residue Procedures

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Foreword

Bituminous emulsion paving materials continue to be a challenging area of research and practical applications. Transportation Research Circular E-C102: Asphalt Emulsion Technology presented an overview of asphalt emulsion technology covering its chemistry, manufacturing, and applications. Continuing with this international sharing of asphalt emulsion technology from researchers and practitioners, this circular reviews methods used to recover the residue of bituminous emulsions.

While there is consensus on standard tests to be performed on the recovered asphalt residue, there is little or no consensus on procedures to remove water from an asphalt emulsion. This is particularly true when it comes to the recovery of residue from a polymer-modified emulsion. Today, it is well accepted that the high-temperature recovery procedures, such as distillation, used to obtain the residue from non-polymer–modified emulsions is not suitable for polymer-modified emulsions. It has been shown that high recovery temperatures and extended recovery times degrade the polymer significantly, hence measurement of residue property gives misleading results on its ultimate performance.

The hot-mix asphalt industry has accepted that measuring rheological properties of asphalt binders tells something about their performance. Therefore, many of the traditional tests have been abandoned as specifications for asphalt binders. Conversely, the traditional tests continue to be used for asphalt emulsions. There have been some attempts to perform rheological tests on the asphalt emulsion residue. However, the results were questioned because of the recovery procedure used. While instrumentation exists to perform rheological tests, the stumbling block for the asphalt emulsion industry to implement such a rheological approach is the lack of an acceptable recovery method that would be suitable to all emulsions. With modern instrumentation it is not hard to imagine one instrument whose temperature can be programmed for the particular type of emulsion and that can be automated, making measurement of rheological measurements simple provided the residue for testing can be obtained.

The three papers in this circular summarize methods used worldwide and suggest new avenues for research and practice that at some juncture will lead to a consensus standard for the recovery of an asphalt residue. They offer a rich perspective of the many different procedures that have been tried and put forth the challenges faced by the industry as it moves towards performance standards for bituminous emulsion paving products.

The papers are based on presentations from a session on this topic at the 86th Annual Meeting of the Transportation Research Board (TRB), January 2007. The papers have not undergone a formal peer review.

Appreciation is expressed to the authors for their valuable contributions and to Robert McGennis, Chair of the Characteristics of Bituminous Materials Committee, who facilitated this second TRB bituminous emulsion technology session. A special thanks to Rebecca McDaniel who provided valuable editorial input to the text.

—Delmar Salomon
Pavement Preservation Systems, LLC

Contents

Characterization of the Different Phases in the Life Cycle of the Binder in a Bitumen Emulsion

Recovery Methods

CAROLE GUEIT
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Several techniques are available to recover a binder from a bitumen emulsion for further characterization. Some of these consist of allowing a thin film of emulsion to stand in an oven at a given temperature; other methods consist of heating the emulsion, and then the residual binder, under various conditions.

Processes which enable the recovery of the binder without changing its characteristics are especially useful when performing an assessment study, during which it is often necessary to determine the characteristics of the binder before emulsification.

Furthermore, as regards latex emulsions, these techniques represent the only method to yield young age-modified binder properties. As latex is incorporated in the aqueous phase of the emulsion, the modified binder is formed only after the emulsion breaks.

This study was carried out in the laboratory, on "reference" emulsions: pure bitumen, fluxed bitumen, styrene-butadiene-styrene (SBS)-modified binder and latex emulsion. For each emulsion, the binder was recovered using the various techniques above, and then characterized. These various methods have been evaluated and classified according to their influence on binder characteristics.

One of the processes proved to be especially interesting. It consists of breaking the emulsion by the addition of ethanol and then allowing the recovered binder to dry in an oven.

This study showed that the various recovery methods yield binder representing several states of aging: "recovered" binder, whose characteristics are near those of the binder before emulsification; "stabilized" binder, where the binder aging simulates a 6- to-12-month aging period; and "aged" binder, where the simulated aging represents several years of aging.

Various characteristics of bitumen emulsions can be tested with a view to predicting their in-service performance: storage and transport stability, emulsion break time, and rate of cohesion build-up, etc. However, another factor is equally crucial for the quality of a road surfacing: the characteristics of the binder itself, after the emulsion has broken.

In order to appraise the performance and the durability of a surfacing, it is therefore necessary to be able to simulate in the laboratory short-term aging (immediately after the emulsion breaks and the road is reopened to traffic), medium-term aging (after between 6 months and 1 year in situ), and long-term aging (after several years in situ).

Furthermore, in the context of certain investigations it is useful to be able to recover the binder as it was prior to emulsification. In this case it is important to select a recovery method that recovers the original binder with as little influence as possible on its properties.

To this end, a number of laboratory binder recovery or aging techniques are available. A study was performed at the Colas Campus Scientifique et Technique in order to evaluate the following for each method:

- The types of emulsion for which the technique is suited;
- The influence of the recovery method on the characteristics of the binder and its ability to recover the binder as it was prior to emulsification; and
- The benefits and limitations of the technique from a practical standpoint and with regard to the safety of the technician.

DIFFERENT RECOVERY METHODS AVAILABLE

The recovery methods available can be divided into two types: thin film oven methods, and other methods that involve heating "in the mass" or chemical action under clearly defined operating conditions.

Thin Film Oven Methods

These techniques, for which French or European standards exist, are used to simulate the short-, medium- or long-term aging of a bituminous binder. They involve heating the emulsion, then the residual binder film to a given temperature for a fixed duration.

The method described in EN 13074 standard is designed for short-term aging simulation (just after the emulsion breaks and the road is reopened to traffic). It enables, according to the analysis of the standard itself, recovering a binder from an emulsion with only minor changes in its characteristics. The procedure consists of storing the emulsion for 24 h at ambient temperature and then for 24 h at 50°C. The film of residual binder is 1 mm thick.

To simulate medium-term aging (stabilization, 6 months to 1 year of in situ aging), the following additional methods can be applied, also with a residual binder film 1 mm thick:

- NF T 66-031 (French standard), which consists of storage for 14 days at 50°C; and
- EN 14895 (European standard): storage for 24 h at ambient temperature, then 24 h at 50°C ("recovered" binder, short-term aging) and, finally, 24 h at 85°C ("stabilized" binder, medium-term aging).

The binder that is stabilized by this last method can then be subjected to simulated long-term aging (several years of in situ aging). The long-term aging procedure involves maintaining a 3.2 mm thick film of the binder in a pressure aging vessel (PAV) at a pressure of 2.1 MPa and a temperature of between 80°C and 115°C for 20 or 65 h. This technique is described in the standard EN 14769. The conditions applied in this study were 65 h at 85°C. A similar method is described in AASHTO R 28-02, with slightly different operating conditions (2.1 MPa, 90°C to 110°C, 20 h).

After this period in the oven, irrespective of the duration of simulated aging, the binder consists of a thin film which has to be manipulated and homogenized in order to carry out the subsequent tests.

Other Methods

Unlike thin film oven methods, these provide a binder which can be used directly for testing. This eliminates the difficult process of scraping off the thin film of binder in order to homogenize it. These techniques consist of heating or chemically treating the emulsion under specified conditions. The methods evaluated in this study are as follows.

- **Belgian Procedure 08-34.** In this procedure, the binder is recovered by evaporating off the aqueous phase of 1 L of emulsion in a 2 -L tall beaker heated with a Bunsen burner and stirred continuously. A temperature of 163°C is reached in the course of the test. The binder is then placed in an oven at 163°C for half an hour in order to eliminate any remaining traces of water.
- **Spanish Method NLT 147.** This method consists of heating 50 g of emulsion for 2 h at 163°C in a 600-ml beaker. The shape of the beaker is specified because the emulsion foams when it is heated. This method is based on the same principle as the ASTM D244 evaporation method (Procedure A, formerly in D244 and now in D6934). The only difference stands in the shape of the beaker, which is 1-L low shape in the case of the ASTM method. This may lead to slight differences in recovered binder characteristics: both methods involve the same amount of emulsion, but in the ASTM method, the binder film is thinner, and thus may be slightly more sensitive to the effects of heating.
- **Distillation according to ASTM D244/EN 1431.** This procedure is performed on 200 g of emulsion and provides a distillation-based means of recovering the aqueous phase and the volatile fractions of the organic phase. At the end of distillation, the binder reaches 260°C, and it is maintained at this temperature for 15 min. The binder that remains in the reactor is then poured directly into the test containers. (The ASTM D244 Distillation method is now a separate standard, D6997.)
- **Ethanol precipitation method.** This method was developed within the Colas group and differs from the various methods which involve heating. In this method, the separation of the aqueous phase is not performed by heating but chemically, by a process that also leads to the partial or complete separation of the emulsifier. This is therefore the only method which makes it possible in some cases to recover a binder with characteristics which are identical to those of the base bitumen, not only with regard to consistency (penetration, softening point) but also chemically (acid value). This technique consists of adding ethanol to the emulsion; a lump of bitumen forms immediately. This lump is compressed and rinsed in water until the foam produced by the emulsifier remaining in the bitumen disappears. The resulting bitumen is then “dried” at 140°C until air bubbles no longer appear on the surface.

THE STUDY

Various types of bitumen emulsions were manufactured from binders which are frequently used in roads, and the binder of each emulsion was recovered using the different available methods. Three types of emulsion were studied: pure bitumen emulsions, modified binder emulsions and fluxed bitumen emulsions.

Pure Bitumen Emulsions and Modified Binder Emulsions (*1*)

The spraying emulsions involved in the study were the following:

- Two pure bitumen emulsions, one using 160/220 pen bitumen and the other using 35/50 pen bitumen; and
- Two modified bitumen emulsions, one using 160/220 pen SBS-modified binder and the other a 160/220 pen bitumen emulsion with latex in the aqueous phase.
- All the binders recovered from these emulsions using the methods listed above were subjected to a penetration test at 25°C according to EN 1426 and a ring and ball softening point test according to EN 1427. The same tests were also performed on the binders prior to emulsification (with the exception of the latex emulsion binder). The impact of the recovery method on the characteristics of the binder was then evaluated by calculating the following parameters:
  - Residual penetration (ResPen, in %): the percentage ratio between the penetration of the recovered binder and that of the binder before emulsification and
  - Difference in the softening point (ΔR&B, in °C), between the initial binder and the recovered binder.

Figure 1 shows the residual penetration values obtained using the different recovery methods.

For the binder recovered from the latex-modified emulsion, the penetration and softening point values used for reference purposes were those of the binder recovered by distillation according to ASTM D244/NF EN 1431, as this method allowed the binder to be recovered from the 160/220 pure bitumen and 160/220 SBS-modified binder without any significant alteration in its properties.

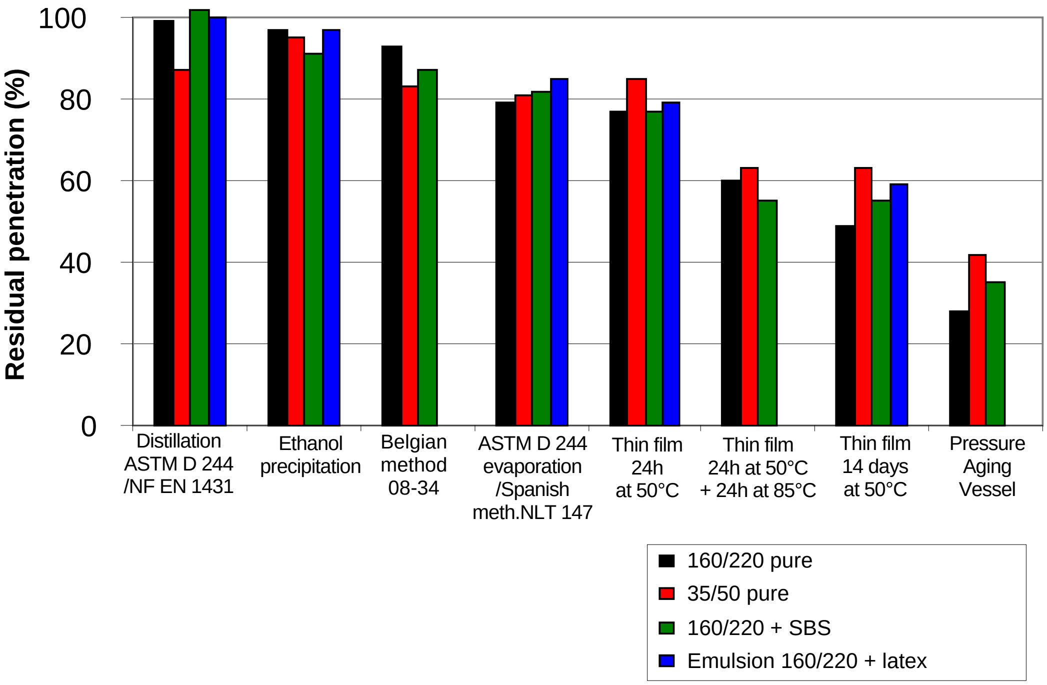

FIGURE 1 Influence of the recovery method on the residual penetration values.

In order to refine the classification of methods with respect to their hardening capacity (revealed by a reduction in penetration) and to investigate the impact of the different recovery methods on polymer conservation, additional tests were performed on the modified binders (SBS and latex). Infrared absorption spectroscopy was used to confirm the continued presence of the polymer by detecting characteristic peaks at 966 cm^{-1} and 700 cm^{-1}. Ultraviolet (UV) microscopic examination of the SBS-modified binders revealed the structure taken on by the polymer in the binder. It turns out that this structure depends, in particular, on the form in which the binder is recovered (thin film or heating in the mass), but cannot logically be linked to the degree of aging of the binder (revealed by its residual penetration). Photos are given in Figure 2 as examples. It should be noted that the tests confirmed the presence of polymer in the binder but did not permit any judgment to be made regarding its state of conservation as they gave no indication of the size of the macromolecules. Gel-permeation chromatography could provide this information.

In order to check whether the recovered binders still exhibit the performance of a modified bitumen, investigation was continued by conducting an elastic recovery test at 10°C (EN 13398) and a pendulum cohesion test (EN 13588). These showed that PAV aging led to a marked deterioration in the elastomer (fracture of specimens during the elastic recovery test, considerable reduction in low temperature cohesion). However, the binders recovered using the other techniques all retained equivalent performance to the initial binder. These additional tests did not therefore lead to a refinement of the first classification of methods based on residual penetration and softening point values. Using these two conventional parameters, we obtain the following three category classification:

- "Negligible aging" methods (minimum residual penetration of 80% and a maximum change in the softening point of 4°C),
- "Low aging" methods (residual penetration of less than 80% or a change in the softening point of more than 4°C), and
- "High aging" method: PAV method specifically intended for this purpose.

Table 1 shows the resulting classification.

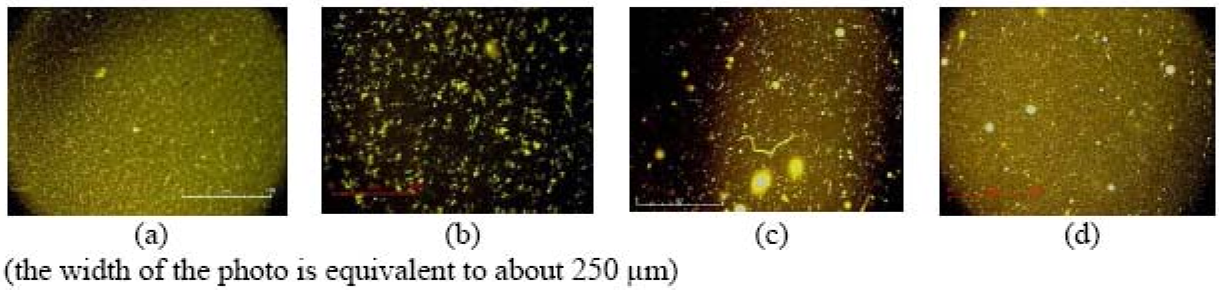

FIGURE 2 Ultraviolet microscopic observation of the modified binders: (*a*) initial SBS-modified binder; (*b*) thin film 24 h at 50°C + 24 h at 85°C; (*c*) Belgian method; and (*d*) after PAV.

TABLE 1 Classification of the Recovery Methods on the Basis of the Experimental Results: Pure Bitumens and Polymer-Modified Binders

Recovery Method	**Residual Pen, ResPen(%)/ Change in Softening Point, ΔR&B(°C)**			
	160/220 pure	**35/50 pure**	**160/220 +SBS**	**Emulsion of 160/220 + latex**
Distillation ASTM D244/NF EN 1431	**99/–0.7**	**87/+0.2**	**102/+1.6**	**Reference**
Precipitation with ethanol	**97/–0.3**	**95/–0.4**	**91/+0.4**	**97/+2.2**
Belgian procedure (heating with a Bunsen burner)	**93/–0.1**	**83/–0.2**	**87/+2.8**	
ASTM D244 evaporation method/Spanish method NLT 147	**79/+1.3**	**81/+1.8**	**82/+3.0**	**85/+3.4**
EN 13074 (thin film 24 h at 50°C)	**77/+0.7**	**85/+1.4***	**77/+4.0 (*)**	**79/+8.2 ***
EN 14895 (thin film 24 h at 50°C + 24 h at 85°C)	**60/+4.5**	**63/+6.0**	**55/+8.2**	
NF T 66-031 (thin film 14 days at 50°C)	**49/+5.5**	**63/+6.6**	**55/+11.4**	**59/+9.0 ***
EN 14769 (PAV 2.1 MPa, 65 h at 85°C	**28/+12.5**	**42/+12.8**	**35/+14.8**	

Light gray = negligible aging method; medium gray = low aging method; dark gray = high aging method.
* Binder heated for subsequent tests at a higher temperature than that specified in the standard (50°C for EN 13074, 100°C for NF T 66-031).

These results elicit several remarks. Various binder recovery techniques are available that produce a binder which is almost identical to the initial binder and directly usable for subsequent tests: distillation as described in EN 1431/ASTM D 244, the Belgian method with heating by Bunsen burner, the evaporation method with heating to 163°C (ASTM D244 Procedure A/Spanish NLT 147), and the ethanol precipitation method. From a practical standpoint, the ethanol method has the advantage of being easy to apply and safe for the user.

Thin film oven methods are responsible for greater aging than the methods that involve heating the emulsion "in the mass," particularly in the case of bitumens with high penetration.

More specifically, the thin-film short-term aging simulation method (EN 13074) is supposed to yield a binder with minor changes in its characteristics; however, it actually leads to a slightly hardened binder. Besides, the thin binder film must be heated in order to be homogenized for further characterizations, but the temperature should not exceed 50°C according to the standard. This can be performed for 160/220 bitumen, but a higher temperature is necessary for 35/50 bitumen and for modified binders.

The two methods that are available for simulating aging of between 6 months and 1 year (holding for 14 days at 50°C or for 24 h at ambient temperature + 24 h at 50°C and 24 h at 85°C) provide equivalent results, which shows how much temperature influences binder hardening speed. The first method can therefore advantageously be replaced by the second.

As one would expect the pressure method for simulating long-term aging (PAV as described in EN 14769) leads to considerable hardening of the binder. In the case of modified binders, the polymer can always be detected by UV microscopy and infrared absorption spectroscopy after the procedure. But the results from the elastic recovery and the pendulum cohesion tests show that the properties in question have been considerably altered, which does not agree with the situation in the field where the polymer retains its performance for many years.

Results for Fluxed Bitumen Emulsions *(2)*

Three fluxed bitumen emulsions were used for this part of the study: two emulsions made from 160/220 pen bitumen fluxed with 2% of F1 and F2 fluxes (F2 being more volatile than F1) and a fluxed bitumen emulsion with 10% of F2 flux. The base binders and the recovered binders were subjected to the same test procedures as the pure bitumen emulsions: penetration as described in NF EN 1426 and ring and ball softening point as described in NF EN 1427.

The influence of each method was assessed by comparing the characteristics of the recovered binders, on the one hand to those of fluxed bitumens, on the other hand to those of the initial 160/220 bitumen. Thus, these methods can be classified on the basis of the type of binder they are able to recover: fluxed bitumen, a binder that is intermediate between pure and fluxed bitumen; flux-free pure bitumen; flux-free bitumen that is slightly aged; and flux-free bitumen that is highly aged. The classification obtained is shown in Table 2.

This classification exhibits some differences from the one that was established on pure bitumens and polymer-modified binders. None of these methods enables the recovery of the binder containing 10% flux. Regardless of the method, the flux always evaporates, at least partially. For both emulsions of bitumen with 2% flux content, the method producing the least aging was the Belgian method of heating by Bunsen burner, which permits recovery of the original fluxed binder.

The results obtained with the ethanol precipitation method depend on the flux that is used. The method permits the recovery of the initial fluxed bitumen in the case of a binder with an F1 flux content of 2% and a binder which is relatively close to the initial fluxed bitumen in the case of a binder containing 2% of F2 flux. In any case, the results clearly show that the binder was initially fluxed; this information is quite important in situations where no information is available about the emulsion.

Distillation as described in ASTM D 244/NF EN 1431, during which a temperature of 260°C is attained, leads to partial evaporation of the flux: the binder obtained is intermediate between the fluxed bitumen and the flux-free bitumen.

The evaporation method with heating to 163°C (ASTM D244 Procedure A/Spanish NLT 147), which leads to the total evaporation of the flux, permits recovery of the pure bitumen regardless of the percentage of flux throughout the tested range of contents (up to 10%).

The results obtained on fluxed bitumens confirm that the EN 13074 standard method, which involves holding a thin film for 24 h at 50°C and which simulates short-term aging, significantly changes the binder properties. It leads to noteworthy evaporation of the flux. In the case of the more volatile F1 flux, the recovered bitumen properties are similar to those of pure bitumen.

TABLE 2 Classification of the Recovery Methods on the Basis of the Experimental Results: Fluxed Bitumens

Recovery Method	Residual pen, ResPen(%)/Change in Softening Point, ΔR&B(°C), Calculated from Pure Bitumen Characteristics		
	Bitumen + 2% F1 flux	Bitumen + 2% F2 flux	Bitumen + 10% F2 flux (penetration at 5°C for softest samples)
Belgian procedure (heating with a Bunsen burner)	180/–2.4	164/–4.0	**/–16.6
Precipitation with ethanol	169/–6.8	140/–5.0	**/–19.6
Distillation ASTM D6934/NF EN 1431	141/-1.0	132/–2.4	**/–6.8
EN 13074 (thin film 24 h at 50°C)	107/+0.4	125/–2.8	**/–11.0
ASTM D6997 evaporation method/Spanish method NLT 147	107/+0.4	102/+0.4	102/–2.0
EN 14895 (thin film 24 h at 50°C + 24 h at 85°C)	62/+4.8	65/+5.4	74/+1.9
NF T 66-031 (thin film 14 days at 50°C)	54/+6.2	61/+4.4	50//+1.6
EN 14769 (PAV 2.1 MPa, 65 h at 85°C)	36/+11.2	33/+11.2	41/+11.0

Fluxed bitumen recovery
Intermediate between fluxed and pure bitumen
Flux-free bitumen recovery **Low aging of bitumen**
High aging of bitumen

** Not available.

Emulsions of bitumen +2% flux:

Pure bitumen recovery:

- ResPen 90% to 110%
- ΔR&B between –2°C and +2°C

Fluxed bitumen recovery:

- ResPen higher than 160%
- ΔR&B between –2.5°C and –8.5°C

The techniques which aim to simulate medium-term aging (6 months to 1 year) or long-term aging give similar results for fluxed and pure bitumens. According to penetration and softening point values, the recovery step can be shortened by replacing the NF T 66-031 method (14 days at 50°C) with the EN 14895 standard method (24 h at room temperature + 24 h at 50°C + 24 h at 85°C).

GENERAL CONCLUSION

Several methods of binder recovery are available. They do not all provide the same type of information, but the range of methods allows control of the state in which the binder is recovered:

- Base binder used in the emulsion (pure, modified or fluxed bitumen);
- Flux-free base bitumen;
- "Recovered" binder: simulation of short-term aging (state of the binder after the emulsion breaks in situ);
- "Stabilized" binder: simulation of medium-term aging (6 months to 1 year in situ); or
- "Aged" binder: simulation of long-term aging (several years in situ).

The recovery method must therefore be selected according to the objective.

When the objective is to recover the binder in its state prior to emulsification, the recommended technique depends on the knowledge available about the binder in question. If the bitumen is not fluxed, the ethanol precipitation method can be applied. If the bitumen is known to be or considered likely to be fluxed, it is better to use the Belgian Bunsen burner heating method. This method is more versatile than the ethanol precipitation method, but less safe.

In cases where the objective is to recover the base bitumen in a flux-free state, the evaporation method with heating to 163°C [ASTM D244 Procedure A (now D6934)/Spanish NLT 147] is appropriate for all flux contents up to 10%.

If the recovered bitumen characteristics are to be compared to specifications, it is essential to follow the recovery method for which the specifications were defined. Indeed, the study shows that results strongly depend on the method used.

For the method described in the European standard EN 13074, the results must be assessed in view of the fact that this method, which simulates short-term aging by heating a thin film of binder for 24 h at 50°C, necessarily results in an evaporation of some of the flux and an evaporation (minimal but not negligible) of the lightest bitumen fractions. Therefore, as one would expect, the characteristics of the binder recovered using this method do not correspond exactly to those of the initial binder. Furthermore, when heating the bitumen in order to carry out the subsequent tests, it was necessary to exceed the maximum temperature stipulated in the standard in all cases except the pure or fluxed 160/220 pen bitumen.

In order to simulate medium-term aging ("stabilized" binder, after 6 months to 1 year in situ), the method specified in French standard NF T 66-031 (thin film held for 14 days at 50°C) can always be beneficially replaced by the method specified in the standard EN 14895 (thin film maintained for 24 h at ambient temperature + 24 h at 50°C + 24 h at 85°C).

Finally, the PAV is intended to be used to simulate several years of aging in situ. This standardized method (described in EN 14769 and following the same principle as the method described in standard AASHTO R 28-02) is considered to be usable for modified binders. It can certainly be of a certain value in the case of a comparative study, but the results should be taken with caution in absolute terms. In this study, the tests conducted on binders aged using this method have shown that the polymer had deteriorated greatly, which is not the case in the field.

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Emulsified Asphalt Residue Recovery Procedures in the United States

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The demand for a well-maintained efficient highway network continues. Asphalt is essential to meet these requirements (*1*). Asphalt emulsions also provide the variety of materials needed for maintenance applications of the U.S. road network. The necessity of testing the asphalt used in the manufacture of emulsions makes the recovery of residue important to suppliers, contractors, and agencies. The following report outlines the reasons for recovering the residue, the main methods that are being used, and what may be necessary for future development.

REASONS FOR RECOVERING THE RESIDUE FROM ASPHALT EMULSIONS

There are two main reasons for recovering the residue from an asphalt emulsion. The first reason is to determine the amount of asphalt, or nonwater phase, in the emulsion, and the second reason is to obtain this asphalt residue for further testing. If the recovery of the residue was just to determine the amount of asphalt in the emulsion, many methods could be used and the quantities of the emulsion necessary could be limited to very small amounts.

The importance of testing the asphalt residue from these emulsions, however, brings forward many variables that need to be understood. The residue must also be recovered with the least amount of damage possible for accurate testing of the residue. Recent uses of modified emulsions created an even further challenge for the residue recovery procedures because of the risk of changing the properties of the modified binder through the recovery process. The amount needed to do sufficient testing on the recovered residue must be taken into consideration in the recovery method selected.

Distillation Methods

Distillation methods require a prescribed amount of asphalt emulsion to be placed into a still and heated to flux out the water phase at the prescribed temperature. Once the temperature is achieved it is usually held for a certain period of time to ensure that all of the water phase has been released. The temperature that is traditionally used is 260ºC (500ºF). Figure 1 shows a picture of a standard distillation setup for an asphalt emulsion.

With the introduction of modified asphalts for the manufacture of emulsion, the distillation temperatures were adjusted lower to try to maintain the integrity of the modified binder. Temperatures of 177ºC (350ºF) and 204ºC (400ºF) have been used by many agencies for these modified binders. Lowering the temperature does help in maintaining the integrity of the polymer, but it still is far above the application and curing temperatures for asphalt emulsions during their use. A vacuum distillation procedure was developed to try to lower the temperature further, to 135ºC (275ºF), to get a closer representation of the binder in the field. A schematic of this method can be seen in Figure 2.

FIGURE 1 Picture of standard distillation method for emulsified asphalts.

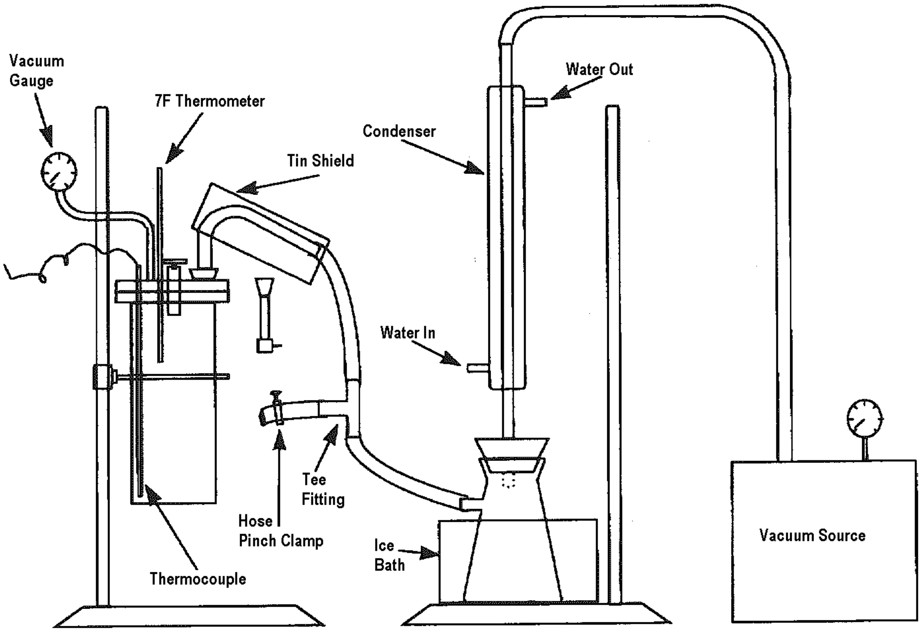

FIGURE 2 Vacuum distillation schematic for emulsified asphalts (*3*).

Evaporation Methods

Evaporation methods for recovering asphalt emulsions are used to obtain residue contents as well as providing materials for testing. When multiple containers are required, they are usually combined and mixed prior to pouring test samples for determining the material properties. The evaporation techniques being used vary in temperature conditions. The higher temperatures have the same issues as the distillation techniques on modified emulsions and the degradation of the polymer. Techniques such as ASTM D6934 and California test methods 330 and 331 are examples of this process.

Recently, evaporative techniques have been developed that lower temperatures closer to those seen by the products on the road during the curing process. Difficulties occur when trying to test the residue due to its high viscosity at lower temperatures, making it difficult to pour into molds or other devices for testing.

There are new evaporative techniques using moisture analyzers that give an accurate reading of the amount of residue using a heated balance technique. The moisture analyzers use very small quantities of emulsion which make it difficult to obtain samples for residue testing.

Comparisons of standard deviations of the recovered residue from various emulsions and test procedures for the penetration and Dynamic Shear Rheometer (DSR) tests are shown in Tables 1 and 2. These were taken from a study by the Asphalt Emulsion Manufacturers Association (*2*).

TABLE 1 Standard Deviation of Penetration Test of Recovered Residue at 25°C

Emulsion	# Labs	Vacuum Dist.	# Labs	177ºC Dist.	# Labs	260ºC Dist.	# Labs	Oven Evaporation
HFRS-2P	7	7.9	10	5.9	10	6.3	10	16.0
CRS-2P	7	11.0	10	16.8	10	4.9	11	10.0
CRS-2(LM)	7	6.9	10	11.3	10	17.4	10	7.5
RS-2(LM)	7	6.9	7	3.1	7	2.4	8	4.1
CRS-2	7	10.9	8	12.3	8	9.3	8	12.3
Micro	7	6.8	9	13.5	10	8.1	11	8.6

TABLE 2 Standard Deviation of DSR Test of Recovered Residue, G*/sin δ at 58°C

Emulsion	# Labs	Vacuum Dist.	# Labs	177ºC Dist.	# Labs	260ºC dist.	# Labs	Oven Evaporation
HFRS-2P	7	0.46	9	0.52	8	0.31	10	0.57
CRS-2P	7	0.42	9	0.24	9	0.41	10	0.33
CRS-2(LM)	7	0.16	9	0.11	9	0.24	10	0.48
RS-2(LM)	7	0.33	7	0.59	7	0.75	8	0.58
CRS-2	7	0.44	8	0.09	8	0.16	9	0.29
Micro	7	0.87	9	1.21	7	0.71	8	1.13

There are no real trends in the data between methods. Higher temperatures, in most cases, do exhibit lower standard deviations. One interesting point to consider is that the unmodified emulsion, CRS-2, had a relatively high standard deviation when compared to the modified emulsions tested.

WHAT IS THE FUTURE FOR ASPHALT EMULSION RESIDUE RECOVERY?

Probably the greatest need that exists is a method of recovering an asphalt emulsion residue that is appropriate for field applications of the materials. The distillation methods used are completed at temperatures that are well above those that the product is exposed to in the field. Even with vacuum procedures to bring the temperature down, the question remains as to the true values one can achieve on the residue as compared to that which is seen in the field. With the use of polymer-modified asphalts or latex additions to the emulsions, there is an even greater concern to recover the appropriate material as applied. Recent efforts that have used evaporative techniques on thin films at 25 ºC to 60ºC have produced encouraging results.

Field comparisons of applications to the recovered residue properties will be necessary to appropriately characterize the proper recovery method to be used. There are some views in industry that emulsion testing may not be important if the applications can be tested rather than the components.

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Emulsion Residue Recovery Techniques

How Do We Get Emulsion Residue Representative of In-Service Binder?

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ASTM and AASHTO have specifications for emulsified asphalt (D977, M140) and cationic-emulsified asphalt (D2397, M208), but not for polymer-modified emulsions. These specifications require the distillation test (ASTM D6997) to acquire emulsion residue for further testing.

Many makers of polymer-modified emulsions believe the high temperatures seen in the standard distillation test (ASTM D 6997) harm the polymer additives. The producers want any specification for polymer-modified emulsions to show the benefits of their polymer. This requires a residue recovery technique that does not harm polymer modifiers.

There is a desire by some [both departments of transportation (DOTs) and producers] to develop more uniform specifications for polymer-modified asphalt emulsions. Some even want to develop more performance-based specifications to apply to all seal coat binders. Both of these require the acceptance of a mechanism of acquiring asphalt emulsion residue that is representative of on-the-road binder.

This paper describes an experiment conducted at the Texas Department of Transportation (TxDOT) to compare six asphalt emulsion residue recovery techniques. Emulsion residues were recovered with each technique and tested by Dynamic Shear Rheometer (DSR).

The paper describes the test procedures and compares techniques using DSR results. Additionally, it makes comparisons relative to equipment cost, test time, sample volume, binder aging, level of testing effort, and interferences (external factors that can affect testing).

Based on this work, the stirred air-flow test (SAFT) appears to produce residue properties that are the most unaffected by the recovery technique. Future work may produce refinements in other techniques to address current perceived deficiencies.

ASTM and AASHTO have specifications for emulsified asphalt (D977, M140) and cationic emulsified asphalt (D2397, M208). These specifications have been in existence for many years and are used or have become the basis for many of the emulsified asphalt specifications used by state DOTs. These specifications essentially use penetration (ASTM D6997) as the sole descriptor of residual binder properties. Like penetration-graded asphalts, the drawback is that this is one point in the temperature continuum of viscoelasticity.

While ASTM and AASHTO do not have specifications for polymer-modified emulsions, many DOTs do. Since penetration tests generally do not distinguish between properties of neat and polymer-modified binders, DOTs have developed other tests to characterize the residue of these emulsions. These additional tests may include viscosity, low-temperature ductility, elastic recovery, force-ductility, and other tests that may target specific polymers.

Many makers of polymer-modified emulsions believe the high temperatures seen in the standard distillation test (ASTM D 6997) harm the polymer additives. The producers want any specification for polymer-modified emulsions to show the benefits of their polymer. This requires a residue recovery technique that does not harm polymer modifiers.

There is a desire by some (both DOTs and producers) to develop more uniform specifications for polymer-modified asphalt emulsions. Some even want to develop more performance-based specifications for seal coat binders. Both of these require the acceptance of a mechanism of acquiring asphalt emulsion residue that is representative of on-the-road binder.

This paper describes an experiment conducted at the TxDOT to compare six asphalt emulsion residue recovery techniques. The optimal procedure would be one that

- Completely removes water from the sample,
- Produces a residue representing on-the-road binder,
- Has low equipment cost,
- Has a short total elapsed time,
- Produces a large enough sample for specification testing,
- Requires a low level of technician effort or attention, and
- Has no interference from outside sources that can affect the outcome.

This paper evaluates the recovery techniques according to these factors.

RESIDUE RECOVERY PROCEDURES EVALUATED

The following residue recovery procedures were evaluated in this study:

- Distillation (ASTM D6997, AASHTO T59);
- Evaporation (ASTM D6934, AASHTO T59);
- Weathering Rack (real-time);
- Thermostatically controlled hot plate;
- Dehydrator; and
- SAFT with nitrogen.

This section describes each procedure.

Distillation

The test used in emulsion specifications today is a distillation test (ASTM D6997, T59), which requires raising the temperature of a 200-g emulsion sample in a distillation still to 260°C (500°F) to boil off all the water. The procedure takes approximately 1 h to complete. Many makers of polymer-modified emulsions believe this high temperature harms the polymer additives. To address these concerns, some DOTs have modified this test for polymer-modified emulsions to limit the temperature to 177°C (350°F). In this testing, a maximum temperature of 177°C (350°F) was used for polymer-modified emulsions and 260°C (500°F) for unmodified emulsions.

Advantages of this procedure are that it is relatively fast, produces a comparatively large quantity of residue for further testing, equipment costs are low (most testing labs already have the equipment), and there are no external environmental factors (weather, lab temperature or humidity) that affect the test.

Disadvantages are that conducting the test takes substantial time and attentiveness on the part of the technician and there are questions about the final temperature affecting the binder and any polymers contained in it.

Evaporation

Evaporation (ASTM D6934) is a standard rapid procedure for determining the asphalt content of asphalt emulsions. Specifications do not normally use this procedure for acquiring residue for subsequent testing. In this procedure, a 50-g emulsion sample is preweighed in a beaker and heated in a 163°C (325°F) oven for 2 h, stirred, heated for 1 h more, and weighed again.

Advantages of this procedure are that it is relatively fast, produces a comparatively large quantity of residue for further testing, equipment costs are low (most testing labs already have the equipment), it does not take substantial time and attentiveness on the part of the technician, and there are no external environmental factors (weather, lab temperature or humidity) that affect the test.

The disadvantage is that there are questions about the final temperature affecting the binder and any polymers contained in it. Until a revision in 2004, this procedure was included in ASTM D244; D244 included a note that indicated this method tends to give asphalt residue properties lower in penetration and ductility. The guidance was that materials could be accepted, but not failed, based on evaporation residue properties.

Weathering Rack (Real Time)

The Weathering Rack procedure uses preweighed 50-g samples of emulsion poured in thin film oven pans. Samples are placed in the TxDOT Materials Lab's weathering facility in standard exposure racks meeting the requirements of ASTM G7: Atmospheric Environmental Exposure Testing of Nonmetallic Materials. Samples are weighed periodically over the course of days until they attain constant weight. The procedure typically takes 2 to 7 days to complete.

Advantages of this procedure are that it produces a comparatively large quantity of residue for further testing, equipment costs are low, it should not produce artificially aged binder or damage polymer modifiers, and it does not take substantial time and attentiveness on the part of the technician.

The disadvantages are the long procedure time, and that external environmental factors (weather, temperature, and humidity) affect the test. During this study, several samples that were rained on before they were substantially cured had to be discarded. This procedure was conducted in the late fall in Austin, Texas; testing in the summer would probably produce shorter cure times.

Thermostatically Controlled Hot Plate

The thermostatically controlled hot plate procedure uses approximately 5 g of emulsion in a disposable aluminum foil container. The hot plate is adjusted to produce a temperature in the container of 79°C to 90°C (175°F to 195°F). Each is stirred using a paperclip. Samples are stirred every 30 min and weighed periodically until they attain constant weight. The procedure typically takes 7 to 8 h.

The advantage of this procedure is that equipment costs are low, and that the residue can be assumed to be very similar to on-road residues, since the recovery occurs under real environmental conditions.

The disadvantages are the longer procedure time, low volume of sample obtained (although multiple samples can be used to generate more residue), there is a question about binder aging as the samples are held at elevated temperatures for a prolonged time, it takes a substantial amount of effort on the part of a technician to stir samples every 30 min, and that external environmental factors (lab temperature and humidity) can affect the test.

Dehydrator

This procedure uses a commercially available food dehydrator with stackable trays. The maximum temperature setting produces 68°C to 74°C (155°F to 165°F) in the dehydrator. Samples consist of 10 g of emulsion poured into thin film oven pans. Samples remain in the dehydrator, unagitated, until they attain constant weight. The procedure typically takes 24 to 72 h to complete.

Advantages of this procedure are that equipment costs are low and it does not take substantial time or attentiveness on the part of the technician.

The disadvantages are the long procedure time, sample volume is low unless multiple samples are used, there are questions about binder aging due to long procedure time and air exposure (discussed later), and that external environmental factors (lab temperature and humidity) affect the test.

Stirred Air Flow Test with Nitrogen

The SAFT is a specialized device, developed by the Texas A&M–Texas Transportation Institute under a research project funded by TxDOT, to replace the rolling thin-film oven test for the artificial short-term aging of asphalt binders used in hot-mix asphalt. The device is essentially a small air-blowing still for asphalt that simulates hot mix plant aging of binders. The binder aging procedure uses 250 g of binder (not emulsion), heats it to 163°C (325°F), stirs the sample vigorously, and bubbles air through the binder. To remove water from asphalt emulsion samples, TxDOT adapted the procedure by lowering the maximum temperature to 104°C (220°F), increasing the sample size to 300 g, and replacing the air with nitrogen. With these changes, the procedure takes approximately 1 h.

Advantages of this procedure are the short procedure time, the large residue volume obtained for subsequent testing, assumed low aging effect due to the nitrogen, and external factors do not affect the procedure.

Disadvantages include the high equipment cost (approximately $15,000) and the level of technician effort required (although it is no more than current distillation requirements).

RESIDUE CONTENTS

Seven total emulsions were acquired from two different locations of one company. All cationic rapid-setting (CRS) emulsions (four CRS-2, one CRS-2P) were from one plant location and all rapid-setting high float (HFRS) emulsions (one HFRS-2, one HFRS-2P) were from another plant location. Residue contents are shown in Table 1 for one typical sample. All samples showed similar results. Some procedures required continuing exposure until samples achieved constant weight and consequently required a longer time to get water out of the system. Three replicates were performed for all procedures except for the evaporation and distillation procedures.

Dynamic Shear Rheometer Testing of Emulsion Residue

TxDOT has funded several research projects to investigate development of a performance-based specification for surface treatment binders (*1–4*). The outcome of the latest project (*4*) suggested considering the DSR on original binder and the bending-beam rheometer test on pressure aging vessel-aged binder as specification parameters. The DSR recommendation was that the binder should have a G*/sin δ value greater than 0.650 kPa at the test temperature. The test temperatures are the same as those in the Superpave performance-graded binder specification.

Because TxDOT has DSRs readily available in the lab and the sample volume required for the test is small, DSR testing was performed on the emulsion residue samples in this study. The grade temperature at which the G*/sin δ equaled 0.650 kPa was determined as was the phase angle at the same temperature. This gave a common ground for comparison of the residues.

Figure 1 shows the grade temperature where G*/sin δ equaled 0.650 kPa for all the emulsions tested using all the residue recovery techniques. Figure 2 shows the phase angle at the temperature where the G*/sin δ equaled 0.650 kPa for all the emulsions tested using all residue recovery techniques.

OBSERVATIONS AND DISCUSSION

One encouraging result of the experiment, as shown in Table 1, is that all of the recovery techniques gave similar residue contents for any given emulsion. This suggests that complete removal of water was accomplished reasonably well by all of the methods. There was a tendency for the weathering rack to show higher residue amounts, which could indicate some residual water left in these samples.

A review of the data in Figure 1 shows that all the samples except the HFRS-2P follow a similar pattern where the SAFT produces the lowest grade and the dehydrator produces the highest grade. The HFRS-2P exhibited an interesting phenomenon in the dehydrator that may explain why it did not follow the same pattern. When the dehydrator samples were poured, they did not uniformly coat the pans, but instead formed circular pools of material in the pans. For most of the samples these pools ruptured during the test, allowing the residue to form a thinner film on the bottom of the pan. The dehydrator samples for the HFRS-2P stayed in the same circular pattern through the entire test.

TABLE 1 Residue Content for a CRS-2

	Replicate 1	Replicate 2	Replicate 3	Average
Weathering Rack	70.08	69.81	69.85	69.91
Hot Plate	70.28	70.46	70.43	70.39
Dehydrator	73.59	73.63	73.94	73.72
SAFT	69.45	69.69	69.63	69.59
Evaporation	71.27	—	—	71.27
Distillation	68.97	—	—	68.97

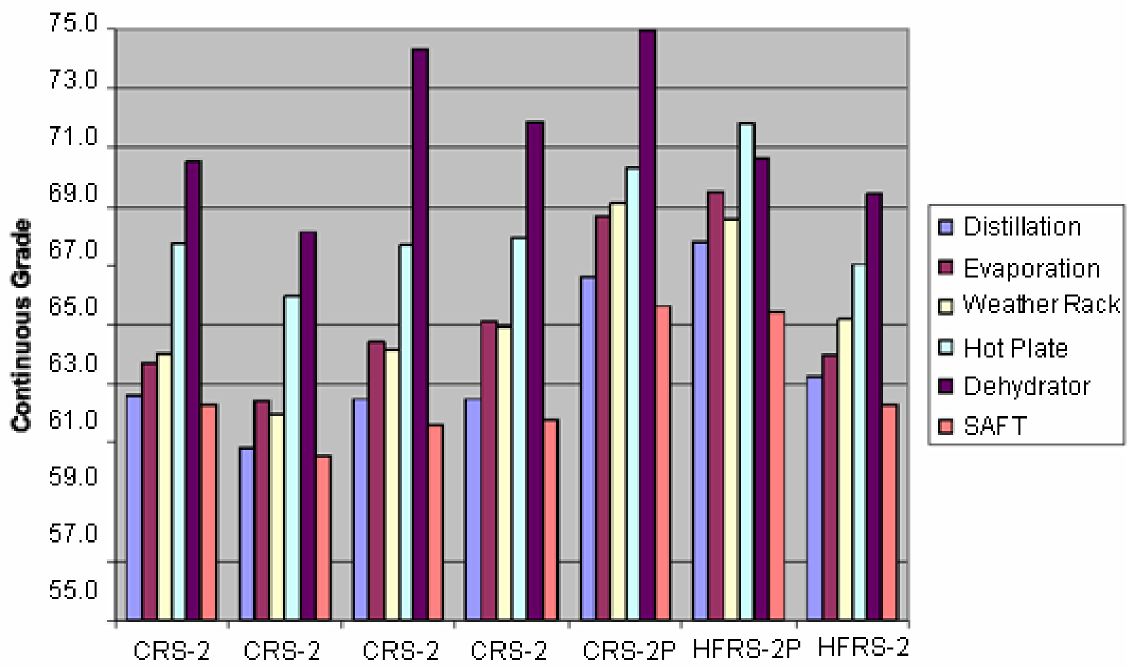

FIGURE 1 Continuous grade temperature with recovery procedure.

In Figure 2, the SAFT generally produced the lowest phase angle and distillation produced the highest phase angle. Also, the CRS-2P and HFRS-2P have the lowest phase angles as expected since they contain polymer modifiers. The high phase angles from Distillation of polymer-modified emulsions could indicate that industry fears of polymer damage are correct.

Because of the advantages of the dehydrator, it was hoped that it would produce good results, but it appears that some aging or oxidation of the binder may be taking place from the high-grade temperatures in Figure 1. Further investigation into this was accomplished by conducting Fourier Transform Infrared Spectroscopy (FTIR) on samples to study the carbonyl area of the spectrum, indicative of oxidation. Figure 3 shows FTIR chromatograms for one emulsion. Figure 4 shows the carbonyl region, wave number 1650 to 1820, in greater detail (*2*). A substantial increase in the carbonyl area for the dehydrator processed samples is observed.

Table 2 shows an evaluation of the test procedures according to several criteria stated in the introduction section of this paper. Every procedure has a “poor” evaluation or a “caution” in at least one category. For the Distillation and Evaporation methods, cautions are due to the commonly held belief that these procedures damage polymer modifiers.

CONCLUSIONS AND RECOMMENDATIONS

One can make the following arguments relative to the procedures evaluated.

1. The best procedure is the one that produces the lowest DSR grade temperature.

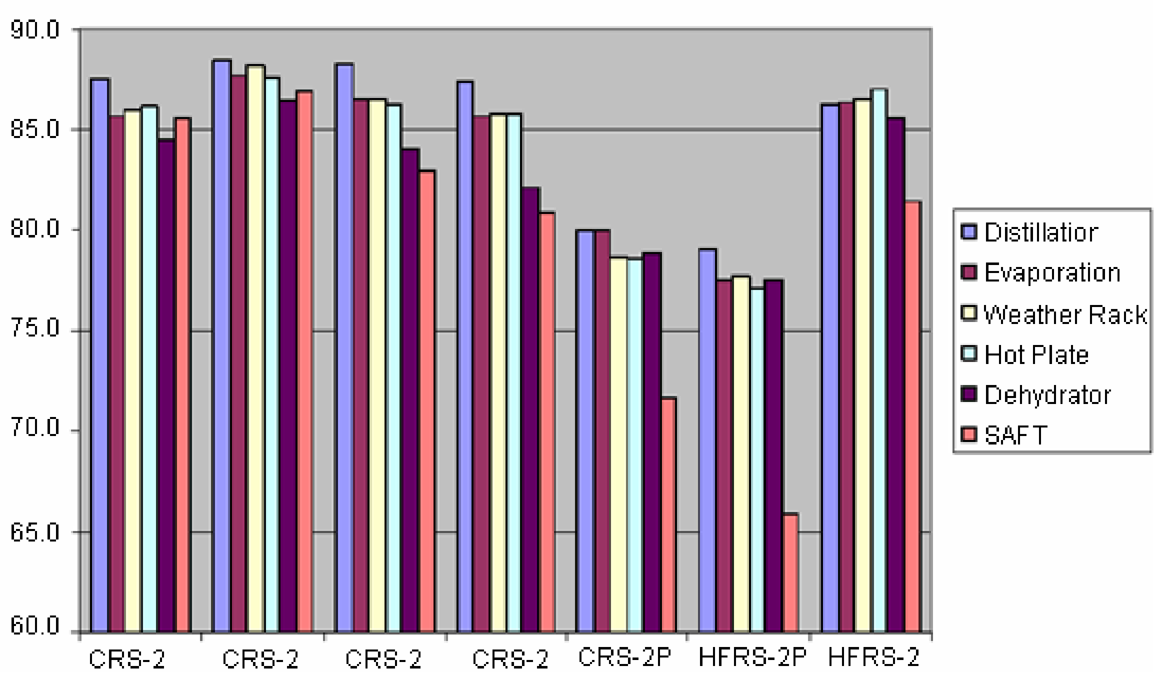

FIGURE 2 Phase angle with recovery procedure.

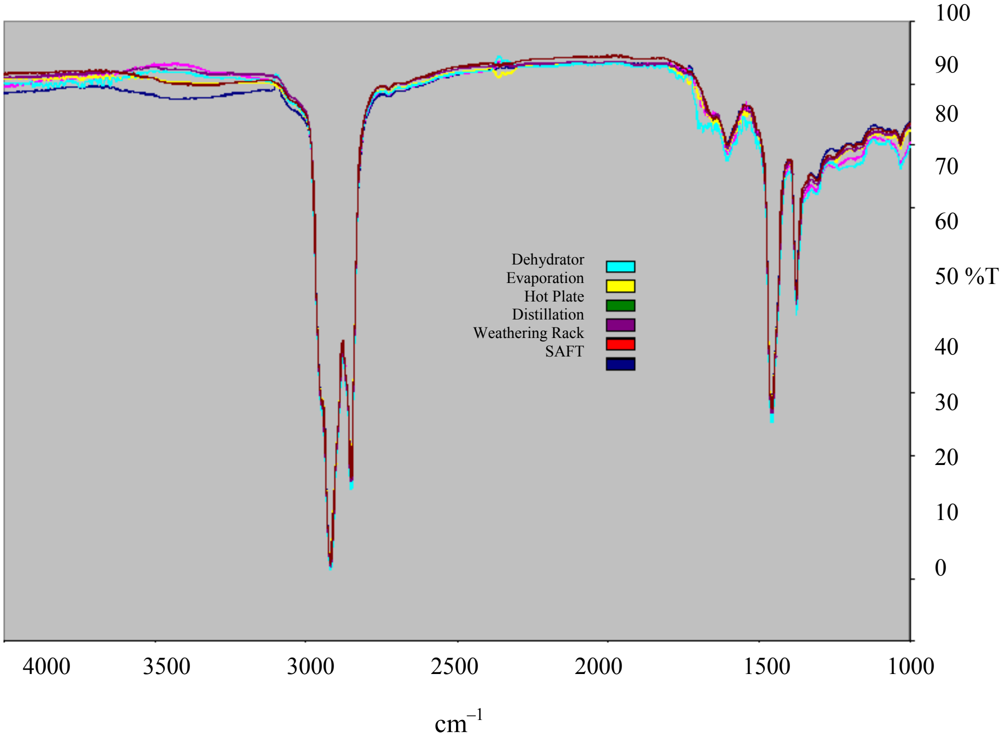

FIGURE 3 FTIR chromatograms.

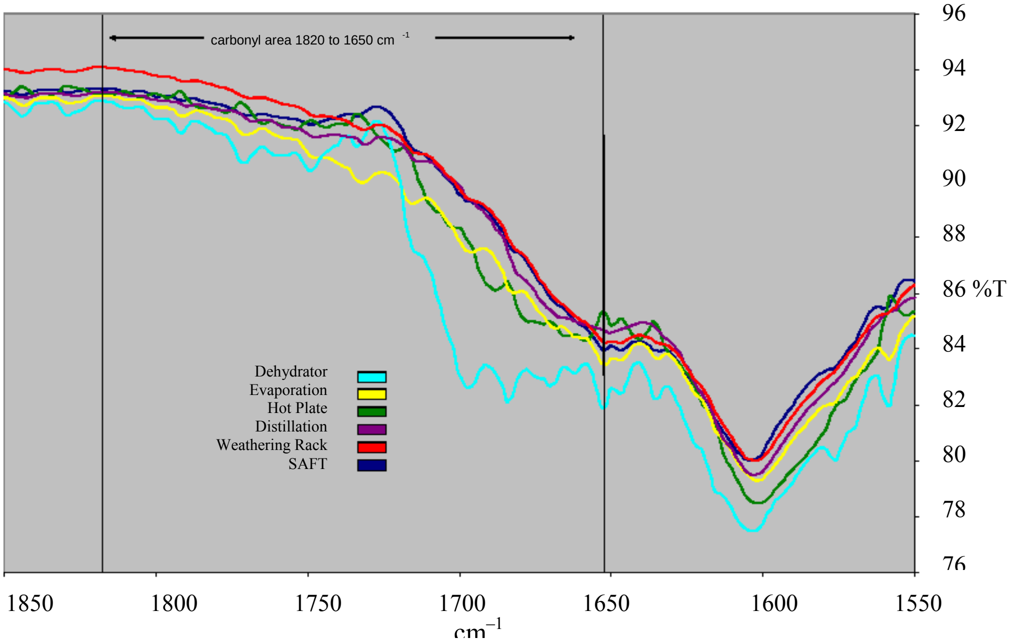

FIGURE 4 FTIR carbonyl regions.

TABLE 2 Procedure Evaluation Matrix

Test	Equipment Cost	Test Time	Sample Volume	Binder Aging	Effort	Interferences
Distillation	Low	Short	High	Low	High	Low
Evaporation	Low	Short	High	Low	Low	Low
Weathering	Low	Long	High	Low	Low	High
Hot Plate	Low	Medium	Low	Medium	Medium	Medium
Dehydrator	Low	Long	Low	High	Low	Medium
SAFT	High	Short	High	Low	High	Low

Good	░
Caution	▨
Poor	▓

2. Procedures that produce high DSR grade temperatures are oxidizing the binder.
3. Procedures that produce high phase angles in polymer-modified emulsions are damaging the polymers.

This work may indicate that arguments 2 and 3 are true, but the data set was small. These criteria suggest the SAFT is the procedure of choice. The SAFT also produces a sufficient amount of residue for further testing.

On the other hand, one can argue that a procedure should be chosen to match on-the-road binders. If so, the weathering rack is the gold standard, and the procedure that produces similar grade temperatures and phase angles to the weathering rack is the best recovery technique. This would indicate that the evaporation and even distillation methods should be considered good matches. These are procedures conducted currently, with industry saying it is concerned about the properties of a residue subjected to the temperatures required.

This study did conclude that the dehydrator procedure, in its present form, is not a good choice to meet the emulsion industry's needs. It appears to produce artificial aging in binders due to oxidation and it takes too long.

Where does this leave us? More research is needed. Some possible procedure modifications and additional testing to evaluate include:

- Use silicone containers and silicone sheets to modify the dehydrator procedure (to lower the temperature and produce thinner films thereby avoiding oxidation and speeding up the procedure);
- Automate the SAFT to make the test more user friendly (by using a different controller that can monitor temperature and/or humidity and make procedure changes automatically thus requiring less intervention on the part of the technician);
- Perform procedures on hot applied binders and compare before and after properties (looking at the oxidation and polymer degradation issues); and
- Test more samples.

ACKNOWLEDGMENTS

Terry Cost and Mark Pavlik, of TxDOT Construction Division's Asphalt Binder Lab, performed the work presented in this report, under supervision of Larry Miller. Jerry Peterson reviewed the data and the report.

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