Comparison of the Physical Properties of Recovered Asphalts Using N-Propyl Bromide as an Altermative Solvent to Trichloroethylene

By

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ABSTRACT

With increased awareness of health safety in the work place and environmental protection, great efforts are currently put forward to research safer alternatives for chemicals deemed toxic or carcinogens. Trichloroethylene (TCE) is a chemical solvent currently used by the Florida Department of Transportation and many other state highway agencies for the separation of asphalt from the aggregates in asphalt paving mixtures, and has recently been identified as a carcinogen and proven to contribute to ozone depletion. This paper presents the results from the second year of a two-year study on an N-Propyl Bromide solvent as a safer alternative for TCE. The first phase included the evaluation of binders extracted and recovered from plant mixtures using TCE, EnSolv (the trademark name for the N-Propyl Bromide used), and reclaimed EnSolv. The second phase of the study included an evaluation of the effects of these solvents on the physical properties of virgin asphalt binders. The additional test results in the second phase of the study reinforce the conclusions that EnSolv and reclaimed EnSolv can be used as a suitable replacement for TCE, for the purpose of extraction and recovery of asphalt from asphalt paving mixtures. When a virgin binder is dissolved in EnSolv and recovered from it, a significant aging effect on the asphalt was noted. However, this effect was not noted when EnSolv was used to extract and recover an asphalt binder from an asphalt mixture, which has already undergone some hardening during mixing.

INTRODUCTION

Background

The extraction of ashalt binders from mixtures is a vital component of quality control and assurance for production of recycled asphalt paving mixtures. Unfortunately one of the components of the extraction and recovery procedure includes the use of the solvent trichloroethylene (TCE), which has been identified as a carcinogen [1]. Exposure to high concentrations of TCE has been proven to pose a severe health risk for the people working with the solvent, as well as contributing to the depletion of the earth's ozone layer. With greater concern being placed on health safety in the workplace and environmental protection, the Florida Department of Transportation (FDOT) felt the need to replace TCE with a different solvent that can reduce health and environmental hazards and yet be able to maintain present testing methods.

In recent years, an alternative solvent for TCE with the tradename of EnSolv has been introduced. EnSolv, whose primary component is n-Propyl Bromide, has been found to contain no significant toxicity at moderate concentrations (100 – 400 ppm) and it contains no chlorinated solvents or carcinogens in its inhibitor package [2]. Unlike the effects of TCE, EnSolve has not been known to cause death or respiratory failure. The use of EnSolv has however been found to cause skin, eye, and lung irritations, headaches, dizziness and nausea, as many chemicals do, and should be used in a well-ventilated area.

EnSolv has recently been evaluated for its suitability as a solvent in the extraction and recovery of asphalts and has been found to provide favorable and repeatable results [3]. In 1998, the FDOT along with the University of Florida began a research study to compare the solubility of asphalt binders and compare the physical properties of extracted asphalt binders in EnSolv with that in TCE. The study also included an evaluation of re-using the EnSolv recovered from the recovery procedure. The findings in the first year of this two-year study has been presented in a paper by Gracia et al. [1]. This paper presents the results from the second year of this study.

SUMMARY OF FINDINGS IN THE FIRST PHASE OF STUDY

Solubility Testing

The standard asphalt solubility test was initially conducted to compare the solubility of asphalt samples in EnSolv with that in TCE. Eigth asphalt cements (typically used in Florida) were used in the testing program. These included five AC-30 asphalt binders, one AC-20, and two rejuvenating agents. The results of the solubility test indicated that EnSolv was not significantly different from the TCE and that it could be used as a replacement for TCE in the ASTM test method D 2042 without modification to the existing method.

Extraction Testing

Extraction tests were run on three plant mixtures for the comparison of the solvents. The mix designs tested in the study included a Marshall mix, a SUPERPAVE mix and a crumb rubber modified mix. The solvents tested in the procedure included TCE, EnSolv and recovered EnSolv. Observations made in this testing program included time required for extraction, ease of method and asphalt content of the mixtures. The results of the extraction indicated that the use of EnSolv and recovered EnSolv actually reduced the time required to complete the extraction while giving the same asphalt content. These results led to the conclusion that both EnSolv and recovered EnSolv could be a suitable replacement for TCE in ASTM D 2172 Method B without modifications to the existing method.

Rotavapor Recovery

Asphalts were recovered from the solvents using the ASTM D 5404 standard Rotavapor recovery process. The time required for the Rotavopor recovery procedure was recorded to see if there were any differences between the solvents. The results indicated that binders recovered from the EnSolv and recovered EnSolv were recovered faster than those from TCE. The results also indicated that EnSolv and recovered EnSolv can be used as a suitable replacement for TCE in ASTM D 5404 without any modification to the method.

Penetration Testing

The asphalt samples recovered from the Rotavapor recovery process were tested for their penetration at 25 ^{o}C. The Student's t-test was performed on the penetration data to determine if the results were statistically different. The statistical analysis on the data indicated that there was no statistical difference between the binders recovered using all three solvents at a 95% confidence level. The differences in the results from different solvents were within expected variation as stated in the precision statement for the penetration of recovered binders, in ASTM D5404.

Viscosity Testing

Using the same samples as in the penetration testing, the Brookfield Viscosity test was performed at 60 ^{o}C. Once again the means and standard deviations of the viscosity data were subjected to the Student t-test. In all cases, there was no statistical difference between the viscosities of any of the recovered binders at a 95% confidence level.

Scope of the Second Phase of Study

Additional testing to evaluate the possible differences between EnSolv and TCE include running Dynamic Shear Rheometer tests, Bending Beam Rheometer tests, and FT-IR Spectral Analysis on all recovered binders from EnSolv, TCE and recovered EnSolv.

An evaluation of the effects of TCE, EnSolv and recovered EnSolv on the physical properties of asphalt binders was also performed. In order to provide comparative samples for this evaluation, 60 grams of asphalt binder was dissolved in 400 ml of solvent and recovered in accordance with ASTM D 5404. The physical properties of the recovered binders were measured and compared with the properties of the original binder samples. The binder tests included penetration at 25 ^{o}C, Brookfield Viscosity at 60 °C, Dynamic Shear Rheometer at 25 ^{o}C and 64 ^{o}C, Bending Beam Rheometer at –18 ^{o}C and FTIR Spectral Analysis.

EVALUATION OF RECOVERED BINDERS FROM MIXTURES

Dynamic Shear Rheometer Results

The Dynamic Shear Rheometer tests were performed in accordance with AASHTO Designation TP5-98 at the temperatures of 25 ^{o}C and 64 ^{o}C. The DSR testing was used to determine the rheological properties of the binder in terms of G* (complex shear modulus) and δ (phase angle). The Student's t-test was performed on the G*Sin(δ) results from the DSR tests performed at 25 °C. The results of the t-test, which are presented in Table 1, indicate that, for all mixtures, there was no significant statistical difference in the results when comparing EnSolv (and reclaimed EnSolv) with TCE at a 95% confidence level.

The Student's t-test was also performed on the G*/Sin (δ) for the tests performed at 64 °C for all three mixtures. The results, which are also displayed in Table 1, indicate that for the Superpave and crumb rubber modified mixtures, there was no significant statistical difference when comparing EnSolv (and reclaimed EnSolv) with TCE at a 95% confidence level. For the Marshall mixture, a significant difference became evident in the results when comparing TCE with reclaimed EnSolv.

Bending Beam Rheometer Results

The Bending Beam Rheometer tests were performed in accordance with AASHTO TP1-98 at a temperature of -18°C. The BBR testing was performed to determine the rheological properties of the binders in terms of creep stiffness (S) and the rate at which the creep stiffness changes with loading time (m-value)[4]. The Student's t-test was performed on both the stiffness (S) and the m-value and the results are presented in Table 2. For all three mixtures tested, it was determined that there was no significant statistical difference between binders recovered using EnSolv and reclaimed EnSolv to the binders recovered using TCE, at a 95% level of confidence.

Infrared Spectroscopy

The infrared spectral analysis was performed on all recovered binders using the Fourier Transform Infrared (FTIR) spectrometer. The FTIR analysis provides an infrared (IR) absorption spectrum of the tested material, which can serve as a "fingerprint" of the chemical characteristics (functional groups) of the tested material. Materials that have dissimilar chemical compositions will show dissimilar IR absorption spectra. The IR absorption spectrum between wavenumbers $1500cm^{-1}$ and $2000cm^{-1}$ is of particular interest (and will be focused on) since it contains the absorption bands for the functional groups of carboxylic acids, ketones, and anhydrides, which are usually formed upon age-hardening of asphalts [5].

The recovered binders that were tested were all dissolved in an HPLC-grade tetrahydrofuran (THF) at a 5% (by weight) concentration. The solution containing the THF and the asphalt was then injected into a de-mountable cell and scanned in the FTIR spectrometer. To remove the absorption spectrums due to the THF, the cell and the environment, a background scan was done on the cell containing only the pure THF. This background scan was then subtracted from the total IR absorption spectrum (by means of the software SPECTRUM used to operate the equipment) to obtain the absorption spectrum of the recovered asphalt sample.

The IR absorption spectra of the EnSolv and reclaimed EnSolv recovered asphalts were compared with those of the TCE recovered asphalts using the COMPARE function provided in the software program SPECTRUM. The compare function calculates the correlation coefficient between the two spectra being compared. A perfect match between the two spectra will have a correlation coefficient of 1.00, while a correlation of coefficient of zero means that the two spectra are completely dissimilar. Table 3 displays the ranges of correlation coefficients between the IR spectra of all sample replicates, as well as the ranges of correlation coefficients between EnSolv and reclaimed EnSolv recovered asphalts to the TCE recovered asphalts.

The results of the FTIR Spectral Analysis indicated that correlation coefficients between EnSolv and reclaimed EnSolv recovered asphalts and TCE recovered asphalts were no less than the correlation coefficients between the three replicates for each solvent.

EVALUATION OF EFFECTS OF SOLVENTS ON RECOVERED BINDERS

Rotavapor Recovery

Two different grades of asphalts (an AC-30 and an AC-20) were dissolved in three different solvents (TCE, EnSolv and reclaimed EnSolv). The asphalts were then recovered in the Rotavapor apparatus in accordance with ASTM D 5404. For each of the solvents investigated, three replicate samples were used. The time required for the recovery procedure was observed to see if the results would compare with the recovery times observed in the first phase of the study.

The mean and standard deviation of the recovery times, along with the Student's t-test results, are presented in Table 4. The analysis of the time data indicated that there was a significant difference in the times required to recover binders from TCE as compared with both EnSolv and reclaimed EnSolv for both binders.

The results of the recovery method indicated that the binders recovered using EnSolv and reclaimed EnSolv were consistently faster than the binders recovered using TCE, for all binders tested. These results support the conclusions made in the first part of the study, that the use of EnSolv and reclaimed EnSolv provides a significant reduction of recovery time as compared with the use of TCE.

Penetration Results

The penetration test on the recovered asphalts from the various solvents was performed in accordance with ASTM D 5, at a temperature of 25 °C. The mean and standard deviation of the results, along with the results of the Student's t-test performed on the data, are provided in Table 5. The results of the statistical analysis identified significant difference between the virgin binder and both the EnSolv and TCE recovered binders, as well as between the TCE recovered binders and the EnSolv recovered binders, for the AC-30 graded asphalt. For the AC-20 graded asphalt, the results identified a much closer comparison with a significant difference being found in the comparison between the virgin binder and the reclaimed EnSolv.

Brookfield Viscosity Results

The Brookfield viscosity test was performed at 60 °C on all binder samples and the mean and standard deviations of the viscosity data, along with the results of the Student's t-test, are provided in Table 6. Comparisons using the Student's t-test were conducted between the recovered binders and the virgin material, as well as between the TCE-recovered binders and the binders recovered using EnSolv and reclaimed EnSolv. The results of the t-test performed on the AC-30 material identified significant differences between all comparisons, with the exception of the virgin-TCE binder and the TCE-reclaimed EnSolv samples. For the AC-20 binder, significant differences where identified when comparing the virgin binder to the EnSolv recovered samples, as well as comparing the TCE recovered samples with the EnSolv recovered samples.

Dynamic Shear Rheometer Results

Following the same procedure for the Dynamic Shear Rheometer, as in the evaluation of the recovered binders from the mixtures, DSR tests were performed on the virgin and all recovered binders at both 25 °C and 64 °C. The Student's t-test results for both testing temperatures and both binders are presented in Table 7. The results of the t-test indicate that there was a significant difference between the virgin binder and the binders recovered using EnSolv. This difference was consistent for both binders and both temperatures tested. The remaining two solvents (reclaimed EnSolv and TCE) were found to provide statistically similar results when compared to the virgin binder.

Bending Beam Rheometer Results

The BBR tests were once again performed at –18 °C, following the same procedure as outlined in the evaluation of recovered binders from mixtures. The Student's t-test was performed on both the stiffness (S) and the m-value, and the results are presented in Table 8. For all binders tested, only one comparison was determined to be statistically different from its virgin binder. The comparison of the m-value, for the AC-30 binder, between the virgin material and the EnSolv recovered binders was found to be the only one that is significantly different at a 95% confidence level.

Infrared Spectroscopy Results

The infrared spectral analysis was performed on all binders following the same procedure outlined in the preceding sections. The IR absorption spectra for all recovered binders were compared with those of their replicates, as well as to the replicates of the virgin binders. The ranges of the correlation coefficients are presented in Table 9. The results of the correlation coefficients identify correlations between all spectra to be over 75%, which signifies good correlations between all binder samples tested. The results also show that correlations between the virgin binder replicates provide higher coefficients with less variability when compared with the solvent-recovered samples. As for the binders recovered with the various solvents, the ranges of correlations overlap closely between themselves, which identifies similarities between the spectra.

CONCLUSIONS

The use of EnSolv and reclaimed EnSolv for the purpose of extraction and recovery of asphalt mixtures has been found to perform equally to TCE. No significant difference was found in the binder properties tested between samples recovered using TCE and the samples recovered with EnSolv and reclaimed EnSolv, although a significant reduction in time was observed.

In the evaluation of the effects of solvents on pure asphalt binders, it was found the solvent-recovered binders had higher variability than the original binder did. This increase in variability was anticipated since dissolving an asphalt in a solvent and recovering the asphalt from it would add another source of variability. It was also observed that the use of EnSolv on virgin material caused significant aging of the binders tested. However, interestingly the use of reclaimed EnSolv displayed very little sign of binder hardening. Although the use of EnSolv was seen to have a hardening effect on the virgin material, EnSolv had been observed to be no different from TCE when it was used to extract and recover binders from an asphalt mixture. It was postulated that the use of EnSolv has a hardening effect only on unaged binders. When EnSolv is used to extract and recover a binder from an asphalt mixture, which has already undergone some hardening during plant mixing, there is no significant difference between EnSolv, reclaimed EnSolv and TCE.

DISCLAIMER

The opinions, findings and conclusions expressed in this paper are those of the authors and not necessarily those of the Florida Department of Transportation or the U.S. Department of Transportation.

REFERENCES

1. Garcia, H., M. Tia, B. Ruth, R. Roque, An Evaluation of an Alternative Solvent for Extraction of Asphalt to Reduce Health and Environmental Hazards, Preprint, Transportation Research Board Annual Meeting, Washington, DC, 2000.

2. EnviroTech International, EnSolv, Technical Data, EnviroTech International, Melrose Park, IL, 1997.

3. Peterson, R. L., H. R. Soleymani, R. M. Anderson, and R. S. McDaniel, Recovery and Testing of RAP Binders from Recycled Asphalt Pavements, Preprint, Transportation Research Board Annual Meeting, Washington, DC, 1999.

4. Roberts, Kandhal, Brown, Lee, Kennedy, *Hot Mix Asphalt Materials, Mixture Design and Construction*. NAPA Research and Education Foundation, Maryland, 1991.

5. Tia, M., Ruth, B.E., Chiu, C.T., Huang, S. C., Richardson, D. *Improved Asphalt Cement Specifications to Ensure Better Performance*. Final Report C3546. University of Florida, 1994.

6. Tia, M., Ruth, B.E., Chiu, C.T., Huang, S. C., Richardson, D. *Development of Criteria for Durability of Modified Asphalts*. Final Report C3532. University of Florida, 1994.

7. Agency for Toxic Substances and Disease Registry (ASDR), Toxicological Profile for Trichloroethylene, U.S. Public Health Service, Department of Health and Human Services, Atlanta, GA, 1993.

8. Clark, Larry, Re: EnSolv Vapor Degreasing and Cleaning Solvent, EnviroTech International, Melrose Park, IL, 1998

9. Cipione, C.A., R. R. Davison, B. L. Burr, C. J. Glover, and J. A. Bullin, Evaluation of Solvents for Extraction of Residual Asphalt from Aggregates, Transportation Research Record No.1323, TRB, National Research Council, Washington, DC, 1991, pp. 47-52.

10. Burr, B. L., R. R. Davison, C. J. Glover, ad J. A. Bullin, Solvent Removal from Asphalt, Transportation Research Record No.1269, TRB, National Research Council, Washington, DC, 1990, pp. 1-8.

LIST OF TABLES

TABLE 1: Student's t-Test Performed on DSR Results on Binders Recovered from Mixtures

Tested Mixture	Test Temperature	Solvent	Mean (Pascals)	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
Marshall Mixture	25 °C (G*Sinδ)	TCE	1.24E+06	1.41E+05	0.306	2.776	NO
		EnSolv	1.21E+06	7.39E+04			
		TCE	1.24E+06	1.41E+05	1.368	2.776	NO
		RE*	1.40E+06	1.51E+05			
	64 °C (G*/Sinδ)	TCE	6483	717	2.298	2.776	NO
		EnSolv	5236	608			
		TCE	6483	717	2.819	2.776	YES
		RE*	4875	680			
Superpave Mixture	25 °C (G*Sinδ)	TCE	1.49E+06	1.21E+05	1.277	2.776	NO
		EnSolv	1.34E+06	1.65E+05			
		TCE	1.49E+06	1.21E+05	0.558	2.776	NO
		RE*	1.41E+06	2.23E+05			
	64 °C (G*/Sinδ)	TCE	6421	320	0.514	2.776	NO
		EnSolv	6125	946			
		TCE	6421	320	1.485	2.776	NO
		RE*	5757	706			
Crumb Rubber Mixture	25 °C (G*Sinδ)	TCE	8.60E+05	1.35E+05	0.789	2.776	NO
		EnSolv	7.97E+05	2.72E+04			
		TCE	8.60E+05	1.35E+05	0.118	2.776	NO
		RE*	8.71E+05	6.28E+04			
	64 °C (G*/Sinδ)	TCE	4025	131	0.529	2.776	NO
		EnSolv	4115	265			
		TCE	4025	131	0.961	2.776	NO
		RE*	4381	629			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 2: Student's t-Test Performed on BBR Results on Binders Recovered from Mixtures

Tested Mixture	Test Parameter	Solvent	Mean	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
Marshall Mixture	Stiffness (MPa)	TCE	167	16	0.099	2.776	NO
		EnSolv	168	7			
		TCE	167	16	1.510	2.776	NO
		RE*	183	9			
	m - value	TCE	0.335	0.007	0.426	2.776	NO
		EnSolv	0.332	0.010			
		TCE	0.335	0.007	2.502	2.776	NO
		RE*	0.324	0.003			
Superpave Mixture	Stiffness (MPa)	TCE	181	9	0.219	2.776	NO
		EnSolv	178	22			
		TCE	181	9	1.093	2.776	NO
		RE*	166	22			
	m - value	TCE	0.325	0.002	0.114	2.776	NO
		EnSolv	0.326	0.015			
		TCE	0.325	0.002	1.189	2.776	NO
		RE*	0.332	0.010			
Crumb Rubber Mixture	Stiffness (MPa)	TCE	129	6	0.548	2.776	NO
		EnSolv	127	2			
		TCE	129	6	0.320	2.776	NO
		RE*	127	9			
	m - value	TCE	0.352	0.008	0.859	2.776	NO
		EnSolv	0.356	0.001			
		TCE	0.352	0.008	0.775	2.776	NO
		RE*	0.348	0.004			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 3: Ranges of FTIR Spectra Correlation Coefficients Between Different Recovered Asphalts

<table>
<tr><td rowspan="3">Tested Mixture</td><td colspan="6">Ranges of Correlation Between Same Solvents</td></tr>
<tr><td colspan="2">TCE</td><td colspan="2">EnSolv</td><td colspan="2">Reclaimed EnSolv</td></tr>
<tr><td>Minimum</td><td>Maximum</td><td>Minimum</td><td>Maximum</td><td>Minimum</td><td>Maximum</td></tr>
<tr><td>Marshall</td><td>0.9138</td><td>0.9785</td><td>0.7284</td><td>0.9570</td><td>0.9253</td><td>0.9462</td></tr>
<tr><td>Superpave</td><td>0.8928</td><td>0.9109</td><td>0.6319</td><td>0.8867</td><td>0.8618</td><td>0.8934</td></tr>
<tr><td>Crumb Rubber Modified</td><td>0.7131</td><td>0.8123</td><td>0.8887</td><td>0.9298</td><td>0.7261</td><td>0.8952</td></tr>
<tr><td rowspan="3"></td><td colspan="6">Ranges of Correlations Between two Different Solvents</td></tr>
<tr><td colspan="2">TCE and EnSolv</td><td colspan="4">TCE and Reclaimed EnSolv</td></tr>
<tr><td>Minimum</td><td>Maximum</td><td colspan="2">Minimum</td><td colspan="2">Maximum</td></tr>
<tr><td>Marshall</td><td>0.8480</td><td>0.9320</td><td colspan="2">0.8937</td><td colspan="2">0.9613</td></tr>
<tr><td>Superpave</td><td>0.7351</td><td>0.9239</td><td colspan="2">0.8013</td><td colspan="2">0.9010</td></tr>
<tr><td>Crumb Rubber Modified</td><td>0.7803</td><td>0.9144</td><td colspan="2">0.6707</td><td colspan="2">0.8888</td></tr>
</table>

TABLE 4: Student's t-Test Performed on Recovery Times for Each Solvent

Tested Material	Solvent	Mean (Minutes)	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
AC-30	TCE	83.3	7.64	6.423	2.776	YES
	EnSolv	53.7	2.31			
	TCE	83.3	7.64	6.416	2.776	YES
	RE*	55.0	0.00			
AC-20	TCE	78.3	10.41	4.481	2.776	YES
	EnSolv	51.0	1.73			
	TCE	78.3	10.41	4.140	2.776	YES
	RE*	52.7	2.52			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 5: Student's t-Test Performed on Penetration Results on Recovered Binders

Tested Material	Solvent	Mean (0.1 mm)	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
AC-30	Virgin	48.5	0.58	20.662	2.776	YES
	EnSolv	38.6	0.60			
	Virgin	48.5	0.58	0.431	2.776	NO
	RE*	48.9	1.50			
	Virgin	48.5	0.58	3.210	2.776	YES
	TCE	51.6	1.57			
	TCE	51.6	1.57	13.397	2.776	YES
	EnSolv	38.6	0.60			
	TCE	51.6	1.57	2.154	2.776	NO
	RE*	48.9	1.50			
AC-20	Virgin	69.2	0.50	0.141	2.776	NO
	EnSolv	69.5	3.66			
	Virgin	69.2	0.50	3.482	2.776	YES
	RE*	72.0	1.30			
	Virgin	69.2	0.50	1.723	2.776	NO
	TCE	73.2	3.99			
	TCE	73.2	3.99	1.184	2.776	NO
	EnSolv	69.5	3.66			
	TCE	73.2	3.99	0.495	2.776	NO
	RE*	72.0	1.30			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 6: Student's t-Test Performed on Viscosity Results on Recovered Binders

Tested Material	Solvent	Mean (Poises)	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
AC-30	Virgin	4759	83.8	26.091	2.776	YES
	EnSolv	8963	266.2			
	Virgin	4759	83.8	8.796	2.776	YES
	RE*	5737	173.4			
	Virgin	4759	83.8	1.274	2.776	NO
	TCE	5611	1155.2			
	TCE	5611	1155.2	4.897	2.776	YES
	EnSolv	8963	266.2			
	TCE	5611	1155.2	0.187	2.776	NO
	RE*	5737	173.4			
AC-20	Virgin	2442	22.3	5.350	2.776	YES
	EnSolv	3378	302.2			
	Virgin	2442	22.3	0.674	2.776	NO
	RE*	2381	155.2			
	Virgin	2442	22.3	0.886	2.776	NO
	TCE	2590	288.6			
	TCE	2590	288.6	3.266	2.776	YES
	EnSolv	3378	302.2			
	TCE	2590	288.6	1.105	2.776	NO
	RE*	2381	155.2			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 7: Student's t-Test Performed on DSR Results on Recovered Binders

Tested Material	Test Temperature	Solvent	Mean (Pascals)	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
AC-30	64°C (G*/Sin δ)	Virgin	2329	37	10.483	2.776	YES
		EnSolv	3566	201			
		Virgin	2329	37	2.304	2.776	NO
		RE*	2432	68			
		Virgin	2329	37	0.198	2.776	NO
		TCE	2306	198			
	25°C (G*Sin δ)	Virgin	8.62E+05	1.70E+04	3.652	2.776	YES
		EnSolv	1.21E+06	1.64E+05			
		Virgin	8.62E+05	1.70E+04	2.227	2.776	NO
		RE*	8.17E+05	3.09E+04			
		Virgin	8.62E+05	1.70E+04	1.740	2.776	NO
		TCE	8.13E+05	4.61E+04			
AC-20	64°C (G*/Sin δ)	Virgin	1231	11	4.119	2.776	YES
		EnSolv	1765	224			
		Virgin	1231	11	0.803	2.776	NO
		RE*	1249	37			
		Virgin	1231	11	1.053	2.776	NO
		TCE	1335	170			
	25°C (G*Sin δ)	Virgin	5.90E+05	2.68E+04	3.657	2.776	YES
		EnSolv	8.34E+05	1.12E+05			
		Virgin	5.90E+05	2.68E+04	0.057	2.776	NO
		RE*	5.92E+05	6.82E+04			
		Virgin	5.90E+05	2.68E+04	0.386	2.776	NO
		TCE	6.14E+05	1.02E+05			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 8: Student's t-Test Performed on BBR Results on Recovered Binders

Tested Material	Test Parameter	Solvent	Mean	Standard Deviation	$t_{Calculated}$	$t_{Critical}$ for 95% Confidence	Significantly Different
AC-30	Stiffness (MPa)	Virgin	184	1	1.464	2.776	NO
		EnSolv	193	11			
		Virgin	184	1	1.479	2.776	NO
		RE*	173	13			
		Virgin	184	1	2.200	2.776	NO
		TCE	166	14			
	m-value	Virgin	0.359	0.004	3.234	2.776	YES
		EnSolv	0.346	0.006			
		Virgin	0.359	0.004	1.304	2.776	NO
		RE*	0.364	0.005			
		Virgin	0.359	0.004	0.352	2.776	NO
		TCE	0.361	0.009			
AC-20	Stiffness (MPa)	Virgin	251	15	0.568	2.776	NO
		EnSolv	260	23			
		Virgin	251	15	1.658	2.776	NO
		RE*	232	13			
		Virgin	251	15	1.309	2.776	NO
		TCE	227	28			
	m-value	Virgin	0.379	0.013	0.785	2.776	NO
		EnSolv	0.370	0.015			
		Virgin	0.379	0.013	1.267	2.776	NO
		RE*	0.391	0.010			
		Virgin	0.379	0.013	1.483	2.776	NO
		TCE	0.396	0.015			

Note: RE* denotes the use of reclaimed EnSolv

TABLE 9: Ranges of FTIR Spectra Correlation Coefficients Between Different Recovered Binders

<table>
<tr><td rowspan="3">Tested Material</td><td colspan="8">Ranges of Correlations Between Replicates</td></tr>
<tr><td colspan="2">Virgin Binder</td><td colspan="2">EnSolv</td><td colspan="2">Reclaimed EnSolv</td><td colspan="2">TCE</td></tr>
<tr><td>Minimum</td><td>Maximum</td><td>Minimum</td><td>Maximum</td><td>Minimum</td><td>Maximum</td><td>Minimum</td><td>Maximum</td></tr>
<tr><td>AC-30</td><td>0.9013</td><td>0.9224</td><td>0.8663</td><td>0.8882</td><td>0.8343</td><td>0.9136</td><td>0.9072</td><td>0.9510</td></tr>
<tr><td>AC-20</td><td>0.9200</td><td>0.9513</td><td>0.8502</td><td>0.8619</td><td>0.8300</td><td>0.9203</td><td>0.8474</td><td>0.8953</td></tr>
<tr><td rowspan="3"></td><td colspan="8">Ranges of Correlations Between the Virgin Binder and Recovered Binders</td></tr>
<tr><td colspan="2">Virgin and EnSolv</td><td colspan="4">Virgin and Reclaimed EnSolv</td><td colspan="2">Virgin and TCE</td></tr>
<tr><td>Minimum</td><td>Maximum</td><td colspan="2">Minimum</td><td colspan="2">Maximum</td><td>Minimum</td><td>Maximum</td></tr>
<tr><td>AC-30</td><td>0.8171</td><td>0.8822</td><td colspan="2">0.8051</td><td colspan="2">0.9095</td><td>0.8021</td><td>0.8742</td></tr>
<tr><td>AC-20</td><td>0.7967</td><td>0.8929</td><td colspan="2">0.7583</td><td colspan="2">0.8637</td><td>0.7914</td><td>0.9134</td></tr>
<tr><td rowspan="3"></td><td colspan="8">Ranges of Correlations Between the Recovered Binders</td></tr>
<tr><td colspan="4">TCE and EnSolv</td><td colspan="4">TCE and Reclaimed EnSolv</td></tr>
<tr><td colspan="2">Minimum</td><td colspan="2">Maximum</td><td colspan="2">Minimum</td><td colspan="2">Maximum</td></tr>
<tr><td>AC-30</td><td colspan="2">0.7156</td><td colspan="2">0.8082</td><td colspan="2">0.8481</td><td colspan="2">0.9434</td></tr>
<tr><td>AC-20</td><td colspan="2">0.8016</td><td colspan="2">0.8550</td><td colspan="2">0.7009</td><td colspan="2">0.8111</td></tr>
</table>