AN EVALUATION OF AN ALTERNATIVE SOLVENT FOR EXTRACTION OF ASPHALT TO REDUCE HEALTH AND ENVIRONMENTAL HAZARDS

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ABSTRACT

Trichloroethylene is a solvent currently used by the Florida Department of Transportation (FDOT) and many state highway agencies for separation of asphalt binders from asphalt paving mixtures in their quality control programs. However, trichloroethylene has been proven to contribute to ozone depletion and is also known as a carcinogen. The goal of this study was to determine if a more environmentally sound and less hazardous solvent could be used for this purpose. The solvent investigated is an n-propyl bromide with a trade name of EnSolv. Preliminary studies show that it is safer than many other solvents available today. This study was to determine if EnSolv could be a substitute for trichloroethylene without changing current testing methods. The tests performed included solubility, extraction and recovery of asphalt binders from mixtures, and penetration and viscosity tests on the recovered binders. The results of the study showed that EnSolv could be a suitable replacement for trichloroethylene. In addition, EnSolv could also be recycled and reused in the extraction and recovery procedures.

INTRODUCTION

Background

It has been recognized that asphalt extraction and recovery procedure is a vital part of quality control of asphalt paving mixtures, especially for recycling projects. Unfortunately one of the main components of this process, the solvent trichloroethylene, has been identified as a carcinogen. It has been known to cause headaches, dizziness, tremors and high exposures have even been known to cause death [1]. Trichloroethylene is also hazardous to the environment and contributes to the depletion of the earth's ozone layer. Because of the dangers of trichloroethylene, it will probably be banned under the U.S. Clean Air Act. This means that in order to maintain the present testing methods, it is necessary to find a less hazardous alternative solvent which would be suitable to replace trichloroethylene.

There is now a new solvent on the market with the trade name of EnSolv. EnSolv is a solvent whose primary component is n-propyl bromide. It has been proven that EnSolv contains no chlorinated solvents and at this time has not been designated as a carcinogen [2]. Unlike trichloroethylene, EnSolv has not been known to cause death or respiratory failure. It has however been found to cause irritation to the lungs, which is increased if there is a preexisting lung condition [3]. There has also been some skin and eye irritation associated with the use of EnSolv, and as with many chemicals, EnSolv should be used in a well-ventilated area to avoid headaches, dizziness and nausea. Overall, EnSolv has been designated as a moderate health hazard, which is better than trichloroethylene.

Initial findings have indicated that it is possible to use EnSolv for the extraction and recovery of asphalt binder from asphalt mixtures. In addition it may be possible to use the EnSolv remaining after the recovery. This solvent may be a suitable replacement for trichloroethylene, but it is necessary to evaluate the use of this solvent for extraction and recovery procedures to see if EnSolv and recovered EnSolv can produce the same results when compared with trichloroethylene.

Background on Asphalt Extraction and Recovery Procedures

There are five different methods of extraction in ASTM D 2172, which include Centrifuge (Method A), Reflux (Method B, C and D), and Vacuum (Method E) extraction. The most popular methods are the Centrifuge Method A and the Reflux Method B. The asphalt extraction method used by the Florida Department of Transportation (FDOT) is ASTM D 2172, Method B. This method is used because it is simple to perform; however this method has some disadvantages. It has been shown that Method B causes an aging effect on the asphalt binder [4]. This is most probably attributed to the high temperatures and long-term exposure to the solvent. Another problem with this method is that it tends to leave some of the asphalt remaining on the aggregate, which may not give accurate results when the properties of the binders were analyzed. It has actually been suggested that this method should not be used when the asphalt properties are going to be determined and should be used for the determination of asphalt content and aggregate gradation only.

In the past, several solvents have been used for the extraction procedure. Benzene was one the first solvents to be used. Later, in the 1950s and 1960s, new solvents were used because of the toxicity of benzene. These solvents included a group of chlorinated solvents, which consisted of trichloroethylene, 1,1,1-trichloroethane, and methylene chloride [5]. With the recent banning of 1,1,1-trichloroethane, trichloroethylene had become on the most popular and effective of these solvents. Currently, this is the solvent used for the extraction procedure at the FDOT.

There are two main methods that have been used in the past for the recovery of asphalt. These methods include the Abson Recovery method, which was introduced in 1933 and the more recent Rotary Evaporation (also called Roto-vap) which has been used since the 1970s. The ASTM Standards for these methods are ASTM D 1856, Recovery of Asphalt from Solution by Abson Method, and ASTM D 5404, Recovery of Asphalt Using the Rotavapor Apparatus. Several studies have been performed to evaluate both methods for their effectiveness in the complete removal of the solvent from recovered binder [5]. In 1983, the Pacific Coast Users Group tested the Abson and Roto-vap methods along with two other methods and found that there was no method that outperformed the rest. The Abson method had low repeatability and failed to remove all the solvent in some cases while the others caused excessive hardening. When the problem was studied closer using trichloroethylene as the solvent, there were several things noted about each method. The Abson method tended to leave significant amounts of solvent in the recovered binder [5]. The Roto-vap method was found to be less consistent and less reproducible than the Abson method, but could be used to recover larger sample sizes. Presently, the Roto-vap method is popular, because it is simple and less labor intensive than the Abson method.

Something important that has been noted in the recent years about the extraction and recovery tests is the high variability in the viscosity of the recovered binders. The results from American Association of State Highway and Transportation Officials (AASHTO) laboratory proficiency tests show that the standard deviation of the viscosities of the recovered binders have ranged from 25 to 42 percent during the years of 1986 to 1991 [6]. Some reasons for the large variation in results are the following:

1. The asphalt is not completely extracted from the aggregate.
2. Solvent remains in the asphalt after recovery.
3. The reaction of asphalt while in solvent may alter the properties during extraction and recovery.

In an attempt to reduce these variations, several studies have been performed to find a method or combination of methods that would provide better repeatability and less variation in the properties of the recovered binders. These methods include extraction performed in a cylinder rock polishing type apparatus and low temperature and high vacuum recoveries in the roto-vap apparatus [7]. Presently, none of these new methods have been adopted by the FDOT.

Objectives of the Study

This study was mainly a laboratory investigation to evaluate the effectiveness of EnSolv and its suitability for use in the asphalt solubility test, and for the asphalt extraction and recovery procedures. The recycled EnSolv obtained from the recovery process was also evaluated for its possible use in the extraction and recovery procedures.

In order to perform the extraction and recovery procedures, three plant mixtures were obtained for the study. These mixtures came from existing paving projects in Florida. The mixtures tested in this study included a Marshall, Superpave and crumb rubber modified mixture. The Marshall and Superpave mixture both contained Reclaimed Asphalt Pavement (RAP). The extraction and recovery procedures were performed in accordance with ASTM standards D 2172 (Method B) [8] and D 5404 [9]. The recovered binders were then tested and analyzed to see if there were any differences due to the use of different solvents. The binder tests included Penetration at 25°C and Viscosity using the Brookfield Rotational Viscometer at 60°C. The results of all testing were analyzed using the Student t test for paired data.

SOLUBILITY TESTING

Testing Method

The solubility test is aimed at determining the bitumen content and non-bitumen matter in a given sample of asphaltic material. In this test, the insoluble matter is collected on a filter and measured. The solubility of asphalt binders in EnSolv was evaluated by performing the standard asphalt solubility test as specified in ASTM D 2042 [10], using both trichloroethylene and EnSolv. Typical asphalt cements used in Florida were provided for testing by the FDOT. These included five AC-30 asphalt binders, one AC-20, and two rejuvenating asphalts. Testing on each asphalt cement was performed in replicates of five and possible differences between the solvents were studied.

Results

The means and standard deviations of the data (in terms of % solubility) collected are shown in Table 1. The difference between the mean solubility in trichloroethylene and that in EnSolv varies from 0.098% (for Asphalt #7) to 0.000% (for Asphalt #1). The standard deviation of the solubility in trichloroethylene varies from 0.0084% (for Asphalt #3) to 0.0303% (for Asphalt #1). The standard deviation of the solubility in EnSolv varies from 0.0090% (for Asphalt #8) to 0.1077% (for Asphalt #2).

The Student t test was run on the solubility data for each of the asphalt cements to determine if the solubility in EnSolv was significantly different from that in trichloroethylene at a confidence level of 95%. The results from this analysis are also displayed in Table 1. The statistical analysis indicates that, with the exception of

Asphalts #6 and #7, the difference in solubility in the two solvents is statistically insignificant.

In order to verify the results of the tests on Asphalts #6 and #7, solubility tests were repeated on these two asphalts with 5 replicates per asphalt. The results of the repeated tests are also displayed in Table 1. The same statistical analysis was performed on the data and the results are also contained in Table 1. The results of the analysis on the two new sets of data indicate that the difference in solubility is not significant for Asphalt #6, but was still significant for Asphalt #7. In addition to analyzing each sample sets separately, all the replicates for Asphalt #6 were grouped together so that each set had a total of ten replicates. The same was done for Asphalt #7. The Student t test was performed on the data comparing the solvents and the results showed that in both asphalt samples, there was a significant statistical difference. Since the difference of the means was only 0.013% for Asphalt #6 and 0.105% for Asphalt #7, the small values indicate that for all practical purposes, there was really very little difference between the different solvents.

One final analysis was performed on the solubility data to see if the differences fell within the allowable limits specified in the precision statement of the ASTM D 2042. In order to perform this analysis, the standard deviation value from both within the laboratory variability and between laboratory variability was taken from the standard and this value was used to calculate the maximum allowable difference between the means of the data. The differences were within the allowable limits for within laboratory variations in 6 out of 10 cases, while they were within the allowable limits for between laboratory variations in 10 out of 10 times.

Conclusions from Solubility Testing

The results of the testing indicate that EnSolv is not statistically different from trichloroethylene in 7 out of 10 sets of tests. In the sets of tests that were statistically different, the maximum difference between the means was 0.114%. The difference was so small that it really has no practical significance in this test. From the data, it seems that EnSolv is a suitable replacement for trichloroethylene in the ASTM test method D 2042. In addition, this test method does not need any modifications when using EnSolv.

EXTRACTION TESTING

Testing Method

The Reflux extraction was performed in accordance with ASTM D 2172 Method B to extract the binder from each of the asphalt mixtures. For simplicity, all extractions were performed using only one basket in the glass jar. The solvents tested in this procedure included trichloroethylene, EnSolv, and recovered EnSolv. The sample size for testing was 1500 grams, and 800 ml of solvent was for each extraction. This sample size was chosen to ensure that there would be enough binder after the recovery to perform further testing. Observations of this testing included time required for extraction, ease of method, and asphalt content of the mixture.

Results

The requirement for performing the extraction and recovery procedures is that all testing must be completed within an 8-hour time limit (based on the Abson Method). When running the extraction test, the time required for a full extraction was recorded to help determine if the entire extraction and recovery procedure could be completed in the time allowed. It was noted that the extraction of the crumb rubber modified mixture took a long time to complete. Initial extraction tests on the crumb rubber modified mixture took an average time of 7 hours for the extraction of a 500-gram sample (which was smaller than the amount chosen for testing). It was apparent that the entire extraction and recovery procedure could not be completed within the 8-hour time limit.

One of the problems noted when performing the extraction of crumb rubber modified mixture was that the rubber mixture was causing a clogging problem and an excess amount of solvent was accumulating in the cone resulting in a very small amount of solvent remaining in the bottom of the jar. In addition, any rubber that was floating on top of the solvent in the cone splashed out when the solvent dripped down from the condenser. This caused an additional amount of rubber to be introduced into the solvent-binder solution.

In order to complete the extraction of the crumb rubber modified mixture within the time requirement, it was necessary to make some modification to the test procedure. The part of the test that seemed to be causing the problems was the filter paper. To resolve this problem, a filter paper with a faster flow rate was used. This new filter paper worked for the smaller sample size as well as the 1500-gram sample planned for the study.

The means and standard deviations of the times required for the extraction tests are displayed in Table 2. A statistical analysis was performed on all the data using the student t test for paired data. The first part of the analysis was performed on the length of time required to for extraction of the mixtures to see if there was any statistical difference between trichloroethylene, EnSolv, and recovered EnSolv. The results from the analysis are displayed in Table 2. The student t test revealed that at a 95% confidence level, there was a significant difference between trichloroethylene and EnSolv for all mixtures tested. The results from testing showed that EnSolv worked faster than trichloroethylene for the reflux extraction method. In addition, it was also noticed that the recovered EnSolv worked faster than the trichloroethylene and when comparing the means of the data, it was just as effective as new EnSolv for the extraction procedure.

The Student t test was also performed on the asphalt content data collected in this part of the study. These results are displayed in Table 3. The results indicate that at a 95% confidence level, the asphalt content as determined by using EnSolv is not statistically different from that determined by using trichloroethylene. Also, the asphalt content that was determined by using the recovered EnSolv is statistically no different from the asphalt content determined by using trichloroethylene.

Conclusions from Extraction Testing

The results from the extraction test indicate that from the standpoint of asphalt content determination and extraction time, EnSolv or recovered EnSolv would be a suitable replacement for trichloroethylene for ASTM D 2172 Method B. The testing methods are applicable for the use of EnSolv as well as recovered EnSolv. The use of EnSolv and recovered EnSolv actually reduced the time required to complete the test.

ASPHALT RECOVERY TESTS

Testing Method

For the recovery of asphalt from a solution, the rotavapor apparatus was used in accordance with ASTM D 5404. For each of solvents investigated, the three replicates that were used in the extraction procedure were recovered. The time required for the recovery procedure was recorded to see if there were any differences among the solvents.

Results

The means and the standard deviations for the times required for the asphalt recoveries are displayed in Table 4. Student t test was performed on the time data to determine if there were any significant differences among the different solvents. With the exception of the Superpave mixture, there was a significant difference in the time required to recover binders from trichloroethylene as compared with EnSolv or recovered EnSolv. From the data it can be seen that for the most part, EnSolv and recovered EnSolv required less time for the completion of the recovery than trichloroethylene.

Conclusions from Recovery Tests

The results of the recovery tests indicated that binders recovered from EnSolv and recovered EnSolv were recovered faster than those from trichloroethylene. The results also indicate that EnSolv and recovered EnSolv can be used in ASTM D 5404 without modifications to the method.

EVALUATION OF RECOVERED BINDERS

Testing Methods

The main purpose for testing the recovered binders was to determine if each solvent would yield the same recovered binders as trichloroethylene. The binders included in the testing were the recovered binders from the three replicates from the Marshall, Superpave and crumb rubber modified mixtures. The tests performed on these binders included Penetration at 25°C and viscosity at 60°C using the Brookfield viscometer.

Penetration Results

The penetration test was performed in accordance with ASTM D 5 [11] at a temperature of 25°C. The means and standard deviations of the penetration data are displayed in Table 5. The Student t test was performed on the penetration data and the results are also displayed in Table 5. The results from the student t test indicated that there was no significant difference between EnSolv and recovered EnSolv.

In addition to the Student t test, another analysis was performed on the penetration data to see if the differences in penetration values fell within the allowable limits specified in the precision statement for the penetration of recovered binders in ASTM D 5404. The precision statement for the penetration test shows that the acceptable range between two results was 27.2%. The differences in penetration values fell within this allowable limit in all cases.

Viscosity Results

The Brookfield viscosity test was performed at 60°C on all recovered binders. The means and standard deviations of the viscosity data are displayed in Table 6. The Student t test was performed on the viscosity data. The analysis results for the Marshall, Superpave and crumb rubber modified mixtures are displayed in Table 6. In all cases, there was no significant difference between the viscosity of recovered binder due to the use of EnSolv, recovered EnSolv and trichloroethylene.

Something unrelated to the viscosity was noted when heating the binder for the tests. The binders that were recovered from EnSolv and recovered EnSolv had a slight odor that resembled the solvent. This was indication that there was most likely some residual solvent remaining in the binder from the recovery procedure. Future investigations, such as the FTIR analysis of the binder would be necessary to see how this solvent might be affecting the binder.

The viscosity data were also analyzed to determine if the differences fell within the allowable limits specified by the precision statement for viscosity of the recovered binders in ASTM D 5404. The precision statement for the penetration test shows that the acceptable range between two results was 23.8%. The differences in viscosity of the recovered binders fell within this allowable limit in all cases.

Conclusions from Testing of Recovered Binders

The results from the penetration and viscosity tests on the recovered binders indicate that EnSolv and recovered EnSolv are not significantly different from trichloroethylene.

From the tests on the recovered binders, it was apparent that EnSolv and recovered EnSolv could be suitable replacements for trichloroethylene.

CONCLUSIONS

The research performed indicated that EnSolv could be a suitable replacement for trichloroethylene for the solubility test (ASTM D 2042). In addition, EnSolv and recovered EnSolv could be suitable replacements for trichloroethylene in the Reflux Extraction procedure (ASTM D 2172 Method B).

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LIST OF TABLES

Table 1 Results of Solubility Tests Using Different Solvents

Asphalt Type	Solvent	Sample Size	Mean (%)	t Value Calculated	t Critical for 95% Confidence	Significantly Different
(1) AC-30	TCE	5	99.952	0.000	2.306	NO
	EnSolv	5	99.952			
(2) AC-20	TCE	5	99.944	1.110	2.306	NO
	EnSolv	5	99.890			
(3) RA-1225	TCE	5	99.975	1.416	2.306	NO
	EnSolv	5	99.936			
(4) RA-700	TCE	5	99.980	0.180	2.306	NO
	EnSolv	5	99.982			
(5) AC-30	TCE	5	99.980	2.214	2.306	NO
	EnSolv	5	99.918			
(6) AC-30	TCE	5	99.966	2.667	2.306	YES
	EnSolv	5	99.950			
(6) AC-30 (Repeat)	TCE	5	99.968	1.925	2.306	NO
	EnSolv	5	99.958			
(7) AC-30	TCE	5	99.974	3.281	2.306	YES
	EnSolv	5	99.876			
(7) AC-30 (Repeat)	TCE	5	99.974	5.053	2.306	YES
	EnSolv	5	99.862			
(8) AC-30	TCE	5	99.988	0.316	2.306	NO
	EnSolv	5	99.986			

Table 2 Time Required for Extraction Tests Using Different Solvents

Mixture Type	Solvent	Mean (Minutes)	Standard Deviation	Sample Size	t Calculated	t Critical for 95% Confidence	Significantly Different
Marshall	TCE	207	18	6	10.287	2.228	YES
	EnSolv	120	9	6			
	TCE	207	18	6	8.413	2.228	YES
	Rec-EnSolv*	124	16	6			
Superpave	TCE	152	12	6	9.278	2.228	YES
	EnSolv	95	9	6			
	TCE	152	12	6	5.846	2.365	YES
	Rec-EnSolv*	106	8	3			
Crumb Rubber Modified	TCE	220	16	6	8.792	2.228	YES
	EnSolv	138	16	6			
	TCE	220	16	6	7.832	2.365	YES
	Rec-EnSolv*	138	12	3			

* Represents Recovered EnSolv

Table 3 Asphalt Content Obtained from Extraction Tests Using Different Solvents

Mixture Type	Solvent	Sample Size	Mean (%)	Standard Deviation (%)	t Calculated	t Critical for 95% Confidence	Significantly Different
Marshall	TCE	6	6.61	0.105	0.277	2.228	NO
	EnSolv	6	6.63	0.161			
	TCE	6	6.61	0.105	0.174	2.228	NO
	Rec-EnSolv*	6	6.62	0.127			
Superpave	TCE	6	4.35	0.287	1.634	2.228	NO
	EnSolv	6	4.55	0.086			
	TCE	6	4.35	0.287	1.278	2.365	NO
	Rec-EnSolv*	3	4.57	0.104			
Crumb Rubber Modified	TCE	6	5.04	0.215	1.549	2.228	NO
	EnSolv	6	5.22	0.186			
	TCE	6	5.04	0.215	0.071	2.365	NO
	Rec-EnSolv*	3	5.05	0.361			

* Represents Recovered EnSolv

Table 4 Time Required for Binder Recovery Tests Using Different Solvents

Mixture	Solvent	Sample Size	Mean (Minutes)	Standard Deviation	t Calculated	t Critical for 95% Confidence	Significantly Different
Marshall	TCE	3	103	6	3.128	2.776	YES
	EnSolv	3	89	5			
	TCE	3	103	6	4.976	2.776	YES
	Rec-EnSolv*	3	85	3			
Superpave	TCE	3	92	7	1.715	2.776	NO
	EnSolv	3	83	6			
	TCE	3	92	7	1.274	2.776	NO
	Rec-EnSolv*	3	86	3			
Crumb Rubber Modified	TCE	3	115	5	8.060	2.776	YES
	EnSolv	3	91	3			
	TCE	3	115	5	3.629	2.776	YES
	Rec-EnSolv*	3	101	5			

Table 5. Penetration of Recovered Binders From Different Solvents

Mixture	Solvent	Sample Size	Mean	Standard Deviation	t Calculated	t Critical for 95% Confidence	Significantly Different
Marshall	TCE	3	39.33	2.89	1.569	2.776	NO
	EnSolv	3	36.67	0.58			
	TCE	3	39.33	2.89	2.229	2.776	NO
	Rec-EnSolv*	3	35.00	1.73			
Superpave	TCE	3	37.00	1.00	1.835	2.776	NO
	EnSolv	3	34.33	2.31			
	TCE	3	37.00	1.00	2.530	2.776	NO
	Rec-EnSolv*	3	34.33	1.53			
Crumb Rubber Modified	TCE	3	46.33	3.21	0.487	2.776	NO
	EnSolv	3	47.33	1.53			
	TCE	3	46.33	3.21	0.169	2.776	NO
	Rec-EnSolv*	3	46.67	1.15			

* Recovered EnSolv

Table 6 Viscosity of Recovered Binders From Different Solvents

Mixture	Solvent	Sample Size	Mean (Poises)	Standard Deviation	t Calculated	t Critical for 95% Confidence	Significantly Different
Marshall	TCE	3	12002	792	0.503	2.776	NO
	EnSolv	3	12262	418			
	TCE	3	12002	792	1.104	2.776	NO
	Rec-EnSolv*	3	13376	2005			
Superpave	TCE	3	13060	424	0.982	2.776	NO
	EnSolv	3	14177	1924			
	TCE	3	13060	424	0.407	2.776	NO
	Rec-EnSolv*	3	13198	403			
Crumb Rubber Modified	TCE	3	9015	484	0.361	2.776	NO
	EnSolv	3	8913	79			
	TCE	3	9015	484	0.393	2.776	NO
	Rec-EnSolv*	3	9376	1512			

* Recovered EnSolv