Survey of Current Asphalt Binder Extraction and Recovery Practices

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ABSTRACT

In the context of a literature study for the Ministry of Transportation of Ontario, a survey was conducted on asphalt binder extraction and recovery, with government agencies and research laboratories in the United States, Canada and Europe participating, receiving 40 responses. Respondents from United States Departments of Transportation (DOTs) comprised the majority of responses. The survey covered three categories of test evaluation: apparatus type, performance and safety. The Centrifuge was found to be the most common extraction method, the Rotary Evaporator was found to be the most common method of recovery and Trichloroethylene was found to be the most common solvent. Only two respondents mentioned using bio-sourced solvents. The most common uses for extraction and recovery were for the analysis of RAP binder and the determination of binder content. The determination of binder content was found to be consistent by all of the respondents. About half of the respondents found that the recovered binder properties were modified during extraction and recovery process in a significant way while the others found issues with binder aging and solvent remaining in the binder. Concerns were raised about the testing of RAP and PMA binders, in terms of difficulty in breaking them down. The average extraction and recovery took around 5 hours. Operator safety concerns focused mostly on volatiles and handling at hot temperatures, while environmental safety concerns focused on toxic chemicals and waste disposal. A number of recommendations were provided by the respondents for improving the test methods.

Keywords: asphalt binder, extraction, recovery, solvent, HMA, RAP, safety.

1. INTRODUCTION

The mixing process of asphalt mixtures combines aggregates with asphalt binder. For quality control (QC), quality assurance (QA) and research purposes, the binder needs to be evaluated for its properties. The determination of the asphalt binder content from hot mix asphalt (HMA) requires its extraction (1). For a number of tests such as Direct Shear Rheology (DSR, AASHTO T 315), softening point (ASTM D36), penetration (ASTM D5), bending beam rheology (AASHTO T 313) and others, the binder needs to be separated from the aggregates, or in other words, recovered from the mixture (2).

The need to extract and recover asphalt binder is increasing with evolving environmental concerns and technological innovations, which has resulted in widespread use of Reclaimed Asphalt Pavement (RAP) (3). While RAP allows for the reduction of the environmental footprint of asphalt pavement, the variability of the RAP depending on the source presents challenges (4), and this variability manifests itself especially in the binder (5). The RAP binder needs to be tested to determine its condition and this requires the use of extraction and recovery techniques (6,7).

There are a number of combinations of methods currently in use by government agencies, universities and industry for the extraction and recovery of asphalt binder. Among these combinations of extraction methods, recovery methods and solvents, there are a number of different mechanisms that may act on the asphalt binder, and can impact or modify the binder characteristics in a variety of ways (8). The various setups for the extraction and recovery apparatuses provide different testing times, use of laboratory resources, ease of use and performance results. Finally, binder extraction and recovery pose challenges from the perspective of operator (9) and environmental safety.

In the context of the literature review conducted by the Centre for Pavement and Transportation Technology (CPATT) at the University of Waterloo for the Ministry of Transportation Ontario, an extraction and recovery survey was conducted in order to ascertain the practices of asphalt laboratories in government ministries, academia and industry.

2. Methodology

Firstly, an email list was developed, using industry, academia and ministry contacts available to the authors. It was further filled in by searching the transportation ministry websites of US states, Canadian provinces and a few European countries where the contact information of labs having extraction equipment could be identified (Belgium, Sweden, France and Britain).

The survey was constructed using Google Forms. It is free online software that allows the survey to be developed fairly quickly and sent out. Nevertheless, it did not allow for much analysis in terms of linking results from one question to another. This had to be done 'manually' by the authors using an excel sheet and sifting through individual responses. The survey was made to be detailed but also to not so long as to encourage participation.

The 10 survey questions were divided into three aspects of asphalt extraction and recovery: i) apparatus type, ii) testing performance and iii) safety. The survey question were developed with the knowledge from a literature review completed on this topic by the authors for MTO. For the apparatus type, the various extraction apparatuses, recovery apparatuses and solvent types were examined. The testing performance section covers the primary use of the testing, testing time, consistency of binder quantification, and binder modification. Finally, the safety portion covers operator and environmental safety concerns.

Asphalt laboratories in Canada, the United States and Europe were solicited for their responses, with mostly government agencies, some university researchers and a few asphalt mix producers being contacted. United States DOTs were consulted in particular as they are known to generally have well developed asphalt testing practices. In hindsight, the survey needed to ask for the contact details and origins of the person filling it out, as it would have been useful to identify the techniques geographically.

3. Survey and Responses

A survey was prepared covering various aspects of the extraction and recovery procedure, including apparatus type, testing performance along with safety and environmental concerns. An internet search was conducted to determine who the authors could forward the survey to. The survey was sent electronically to more than 150 email addresses, and after about a month, 41 completed surveys were received, the findings of which will be discussed in this section.

3.1. Experimental Setup

The questions on experimental setup were used to determine the prevalence of each type of extraction method, recovery method and solvent used in asphalt laboratories.

a. Which Extraction Method(s) is used in your laboratory?

Of the 41 survey respondents, 38 were using extraction methods, with 12 of those employing more than one type in their laboratory. The Centrifuge (Figure 1) is by far the most common extraction method with 84% of respondents (Table 1), Automatic Extraction, Reflux and Vacuum each being employed by a handful of users.

FIGURE 1 Extraction Centrifuge Bowl

The centrifuge was most commonly employed with a trichloroethylene solvent with 72% of users and nPB second with 31% of users. The recovery method used with the centrifuge is about equally Rotary Evaporator with 59% of users and Abson Method with 53% users. In terms of quantifying the binder content, 18 respondents said it was consistent while 13 called it very consistent.

Six of the respondents used Automatic Extraction, commonly with trichloroethylene as the solvent in 5 cases, followed by toluene and tetrachloroethylene with 2 each and 1 user mentioned xylene. The recovery methods used following extraction included Rotary with 6 and the Abson Method with 3. It should be noted that users tended to use more than one solvent/ recovery method in their labs. For binder content, 4 users found it very consistent and 2 consistent.

Reflux Extraction was employed by 5 users, all using trichloroethylene as the solvent, with one also using nPB. This method was used in combination with the Abson recovery method, with one user also employing the Rotary Evaporator. Four users found the binder content results very consistent with one finding it consistent. Only one user reported an issue with aging the binder, which was surprising given that the Reflux method is known to use elevated heat.

Vacuum extraction was reported by 4 users with 3 of them using trichloroethylene, one nPB and one case of the bio-solvent terpene was noted. Three of the users found the determination of binder content consistent with one finding it very consistent.

TABLE 1 WHICH EXTRACTION METHOD (S) IS USED IN YOUR LABORATORY?

Method	#/38	% of total[1]	Recovery		Solvent		
			Rotary	Abson	Trichloro-ethylene	nPB	Toluene
Centrifuge	32	84%	59%	53%	72%	31%	0%
Automatic Extractor	6	16%	100%	50%	83%	0%	33%
Reflux	5	13%	20%	100%	100%	20%	0%
Vacuum	4	11%	25%	25%	75%	25%	0%
Ignition Oven	3	8%	-	-	-	-	-
SHRP	1	3%	100%	0%	0%	0%	100%
Ultrasonic bath	1	3%	-	-	-	-	-

[1] 32% of respondents used multiple methods

One user reported using the Strategic Highway Research Program (SHRP) method – confirming its rarity in terms of used – with a toluene/ethanol mixture for the solvent and rotary evaporation for the recovery. They noted that determination of the binder content was found to be very consistent. Three used the Ignition method (10) to determine binder content which of course does not require using a solvent and does not allow for recovery. MTO reported used an ultrasonic bath for extraction in the past.

b. Which Recovery Method(s) is used in your laboratory?

The recovery methods for binder extraction were less diverse consisting of mostly the Abson and Rotary Evaporator methods (Table 2). Of the 40 respondents, 35 employed binder recovery, 7 of those employed multiple methods and the remaining 6 of the 40 did not recover binder. Two of the respondents mentioned ceasing to perform recovery procedures due to the danger volatile chemicals posed to laboratory technicians.

TABLE 2 Which Recovery Method (s) is used in your laboratory?

Method	#/35	% of total[1]	Extraction	Solvent			Concerns	
			Centrifuge	Trichloro-ethylene	nPB	Toluene	Aging	Residual Solvent
Rotary Evaporator	22	63%	86%	45%	0%	9%	36%	18%
Abson Method	18	51%	94%	94%	28%	11%	44%	33%
Other	2	6%	-	-	-	-	-	-

[1] 20% of respondents used multiple methods

The Rotary Evaporator (Figure 2) was employed by 22 respondents, 86% of whom used it after Centrifuge, 23% after Automatic extraction and one each for Reflux and Vacuum. For the solvent, half of Rotovap users used trichloroethylene, 14% toluene, 9% tetrachloroethylene and 1 user xylene. In terms of quality concerns, 36% users found that the sample was aged and 18% found that the solvent remained in the sample. One respondent commented on "the cumbersome nature of the Rotary Evaporator" and developed an alternative method to reduce the burden on technicians.

The Abson method was employed by 18 of the respondents, 94% of whom employed it after Centrifuge extraction, 28% after Reflux, 17% after Automatic and 11% after Vacuum. Trichloroethylene was the most common solvent used with 94% of cases, 28% responses for nPB and 11% for toluene. Binder aging was a concern for 44% of these users, 33% were concerned with residual solvent, and one mentioned residual fines, although this does not likely have to do with the Abson Method itself. Overall there was more concern in terms of binder modification than with the Rotovap. The MTO reported that they have recently ceased using the Abson method in favour of the Rotovap in their laboratory.

FIGURE 2 Rotary Evaporator Recovery Setup

In terms of alternative procedures, the FHWA reported developing an alternative method to the Rotary Evaporator that yielded half as many recoveries per day, but was much less demanding of the laboratory technicians. This technique involved "nitrogen blanket" distillation (11).

c. Which Solvent(s) is used in your laboratory for the extraction and recovery process?

From the 36 respondents who used solvents, Trichloroethylene was by far the most commonly used solvent with two thirds of responses mentioning it (Table 3). This was followed by nPB with 31%, Toluene with 22%, Tetrachloroethylene with two responses and a number of other solvents mentioned as single responses, including two that could be characterized as bio-solvents. Multiple solvents were reported by 9 respondents, and 2 reported not using a solvent (in the case of the Ignition Oven method that burns the asphalt, which is not technically an extraction method but serves the same purpose in determining the binder content).

TABLE 3 Which Solvent (s) is used in your laboratory for the extraction and recovery process?

Method	#/36	% of total[1]	Concerns		Safety	
			Aging	Resid. Sol.	Volatiles	Corrosive Chemicals
Trichloroethylene	24	67%	46%	33%	88%	29%
N-Propyl Bromide	11	31%	27%	45%	64%	36%
Toluene	8	22%	63%	13%	75%	0%
Tetrachloroethylene	2	6%	50%	0%	100%	50%
Other	6	17%	-	-	-	-

[1] 25% of respondents used multiple methods

In terms of determining the binder content, 46% of the 24 users of Trichloroethylene found it consistent while 54% found it very consistent. In terms of binder modification, 46% had concerns about binder aging while 33% had concerns about the solvent remaining in the samples after recovery. Volatiles were a major in terms of safety, with 88% of respondents, while 29% found the corrosive chemicals concerning. This correlates well with the fact that chlorinated solvents in general are very volatile. One lab stopped performing binder recovery to minimize technician exposure to trichloroethylene.

For nPB, 55% of the 11 users found the determination of the binder content consistent while 45% found it very consistent. About 18% had concerns about binder aging while 45% had concerns about the solvent remaining in the sample. From a safety point of view, 64% of nPB users were concerned about volatiles while 36% were concerned about corrosive chemicals. The concerns are similar to the ones reported by users of Trichloroethylene.

Toluene was found by 88% of 8 users to be very consistent in the determination of binder content while 1 found it merely consistent. About 63% users mentioned concerns about binder aging, with only 1 mentioning the solvent remaining in the sample.

Tetrachloroethylene was reported to be used by two users with one of them raising concerns about binder aging. Other non-organic solvents included dichloromethane, ethanol and xylene. Alcohol was employed by a user for extraction of binder with Elvaloy polymer.

It terms of bio-solvents, Orange Bioact and terpene extract were mentioned by users. Nevertheless the fact that it was only two (5%) of respondents shows that bio-solvents need further development. With the advent of solvent recycling machines and solvent recovery systems perhaps more research and development with bio-solvents could lead to increased usage.

3.2. Testing Performance

a. What is your asphalt binder extraction and recovery setup primarily used for?

Of the 38 respondents who performed extraction and recovery of binder, two thirds used it in the analysis of RAP binder (Table 4). This shows the importance of extraction and recovery setups in the control of RAP mixes and vice versa. The binder in RAP mixes requires considerably more QC/QA and analysis than virgin binder, due to the source of the binder often being unknown. More importantly, the conditions of its aging (production temperatures, additives, silo storage, long hauls to site, climate, etc.) will be variable road to road, and even at different layers in the asphalt pavement. As explained in the introduction, the increased use of RAP has made the extraction and recovery of binder more important for asphalt research and QC/QA.

TABLE 4 What is your asphalt binder extraction and recovery setup primarily used for?

Method	#/38	% of total
Analysis of RAP Binder	26	68%
Determination of Binder Content	23	61%
Asphalt Mix QC/QA	16	42%
Research	7	18%
Forensic Analysis	4	11%

The determination of binder content was a common task among respondents with 61% performing it. It is a common test for asphalt mixture analysis and can be performed with only binder extraction. The determination of binder content can even be performed without solvent, with the ignition oven, where the binder is burned off and so cannot be recovered.

General asphalt mix QC/QA was identified as a task by 42% of respondents. QC/QA could comprise the analysis of binder content, asphalt mix gradation or the analysis of the binder after it has been recovered. General research was mentioned by 18% of respondents and forensic analysis was at 11%, keeping in mind that they were both write-in responses. Forensic analysis usually occurs when there are issues with the pavement performance or durability and can involve the analysis of both the binder and the aggregates.

b. How consistently is your setup able to determine the asphalt binder content?

Of the 35 respondents that used or have used the extraction apparatus to determine binder contact, half find it consistent with the other half find in very consistent (Table 5). This indicates that the accuracy of determining the binder content is not generally a concern, and the users have confidence in the accuracy of their equipment. This could be attributed to regular equipment correlation testing among the various transportation agencies, such as the correlation program at MTO.

TABLE 5 How consistently is your setup able to determine the asphalt binder content?

Method	#/35	% of total
Very Consistently	18	51%
Consistently	17	49%
Somewhat Unreliably	0	0%
Unreliably	0	0%

c. In what way, if any, would you say that the recovered binder is modified by the extraction and recovery process?

Out of the 35 respondents that perform binder recovery, a significant number (54%) had concerns about the binder being modified by the process (Table 6). Aging was the primary concern at 40%, with solvent remaining in the binder being a concern for 29% of users. Binder aging is a risk, especially for processes that use heat to make the binder viscous. On the other hand, if the solvent does not evaporate sufficiently, some will remain in the recovered binder and make the recovered binder softer.

TABLE 6 In what way, if any, would you say that the recovered binder is modified by the extraction and recovery process?

Method	#/35	% of total
Binder not Modified in Significant Way	16	46%
Binder Aged	14	40%
Solvent Remains in Binder	10	29%
Other	3	9%

Other concerns included 2 users mentioning trouble with extraction and recovery of PMAs. This is understandable given that the solvents are designed to dissolve normal binder and not binder with more complex polymers. Users who work with polymers often had to change their solvent to alcohol for Elvaloy for example. One of the respondents claimed that their DOT stopped using recovery due to inconsistencies with PMAs. Another user was concerned about sand remaining in the binders, but this was noted in only one case.

d. How much time does the extraction and recovery process take?

Due to the variety of techniques employed by the users of extraction and recovery, there was a wide variety of estimates for the time to complete the process. The test duration depends not only on the apparatus, but on the type of mix, and the number of passes needed to complete the extraction. RAP and PMA mixes were reported to take somewhat longer than ones with only straight binder. The amount of asphalt needed to be recovered impacts the length of the process as well.

From the respondents that performed both extraction and recovery, a time as short as 1 hour was recorded, although this value was given as per liter and thus could only be applicable for very small sample sizes. The average time was around 5 hours with as much as 8 hours (or a full workday) reported by several respondents. The most frequent test duration was however, between 2 and 4 hours (Figure 3).

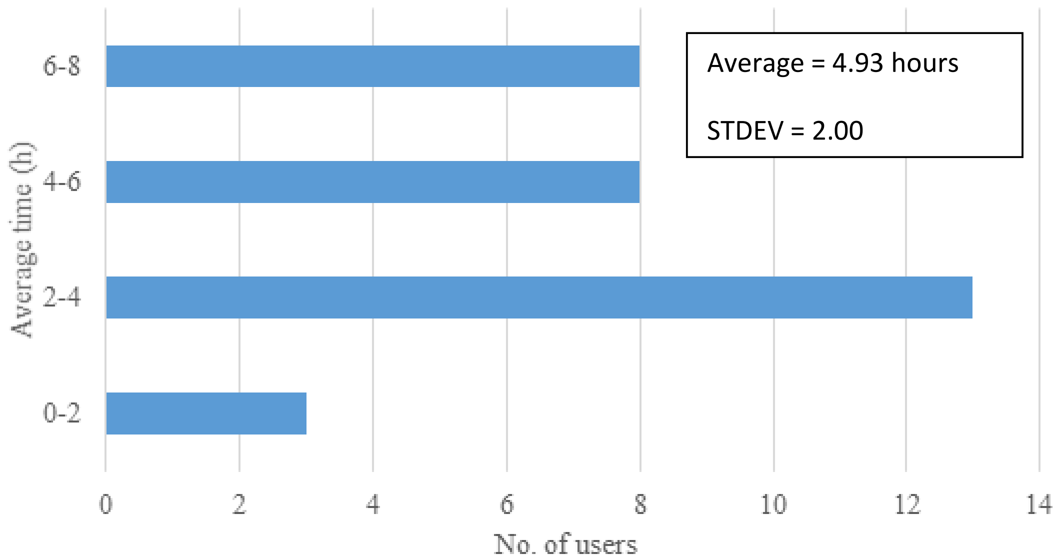

FIGURE 3 Total Extraction and Recovery Time Range

Among the shorter times, the Automatic Extractor with the Abson Method was reported to take 2 hours by one user compared to 4 hours with the Reflux Extraction and 6 hours with the Centrifuge.

e. Are there any ways you could suggest improving the methods?

The survey participants were solicited to provide their practical insights on improving the extraction and recovery methods. A number of interesting responses were received and will be discussed in this section.

A couple of users commented on the cumbersome nature of the Rotary Evaporator. As mentioned previously, this resulted in one lab developing a custom “nitrogen blanket” distillation method (11).

With regards to the Abson Method, one user mentioned difficulty getting the trichloroethylene out of RAP samples, recommending the temperature be increased above 163℃ for these types of samples. It should be noted however, that this risks further aging the sample and that perhaps a more effective solvent would be a better modification to the method.

In addressing higher volume extractions, one user recommended using larger flasks than recommended by the standards for the distillation process (AASHTO T 170), commenting that there would be no adverse effects from using larger flasks.

One user recommended using Fourier transform infrared spectroscopy (FTIR) to verify that there is no solvent remaining in the binder, which would be a relatively simple control procedure for labs that have the device available to them.

3.3. Health, Safety and Environment

a. Are there any safety or health concerns for the operator or the lab environment with regard to extraction, recovery or the solvents?

Among the 40 users who commented on safety concerns for the operators, three-quarters were concerned about the volatiles (Table 7), which come from not only the solvent, but the binder itself. Handling at hot temperatures was the second largest safety concern at 40%, a risk that exists for methods that heat the sample to liquefy the binder. Only 15% of users did not have major safety concerns with regards to these methods.

TABLE 7 Are there any safety or health concerns for the operator or the lab environment with regard to extraction, recovery or the solvents?

Method	#/40	% of total
Volatiles	30	75%
Handling at Hot Temperatures	16	40%
Corrosive chemicals	14	35%
Carcinogens	6	15%
Other	6	15%
No Major Concerns	6	15%

Corrosive chemicals was a concern for 35% of users, while carcinogens (as a write-in response) was a concern for 15%, although this can be considered related to concerns about volatiles. Other concerns included flammability and the potential exposure to silica dust.

b. In regards to the preceding question, what precautions are taken (or need to be taken) given these concerns?

There were a number of suggestions for improving the user safety of extraction and recovery procedures. Foremost, they suggested the use of fume hoods, which are able to reduce the exposure of the user to volatile chemicals. Personal protection equipment (PPE) was cited as important including using proper ventilation masks, eye goggles, latex and nitrile gloves, full length clothes etc. Avoiding binder recovery was also mentioned as improving safety conditions.

c. Are there any environmental concerns in preforming asphalt binder extraction and recovery?

For environmental concerns, toxic chemicals and waste disposal were the most common, shared by 2/3 of respondents each (Table 8). Extraction and recovery requires the use of solvents which can be harmful to the environment if not properly contained. Waste disposal is an issue not only for solvents, but the asphalt binder as well. Two of the users mentioned that their labs have contracts with a specialized waste disposal companies to deal with this aspect.

Volatiles were an environmental concern for 45% of the respondents, two users mentioned the importance of fume hoods in mitigating this problem. Only 23% had no major concerns in this regard, indicating that this remains a major concern with the practice and one respondent mentioned that their lab stopped performing extraction and recovery due to environmental issues.

TABLE 8 Are there any environmental concerns in preforming asphalt binder extraction and recovery?

Method	#/40	% of total
Toxic Chemicals	27	68%
Waste Disposal	27	68%
Volatiles	18	45%
No Major Concerns	9	23%

4. Discussion and Conclusions

The use of extraction and recovery methods consists of mostly analysis of RAP binder and determination of binder content. Asphalt mix QC/QA, scientific research and forensic analysis also commonly employ these methods. In terms of improving the methodology, the survey should have for the contact details and origins of the person filling it out, as it would have been useful to identify the techniques geographically. Additionally, survey software that included more data analysis option could be considered.

The responses to the extraction and recovery survey indicated a variety of extraction, recovery methods along with solvents used throughout different asphalt labs. Nevertheless, there was an overwhelmingly popular choice, or in the case of recovery two main choices. Multiple methods were also used in a number of labs, making deciphering the information somewhat more difficult.

For extraction methods, the Centrifuge is far and away the most common one, likely having to do with its affordability and ease of use. Automatic Extraction seemed to be the easiest to operate, although with some concerns about binder aging. Reflux extraction seemed to give the least concern in terms of aging and the SHRP method warrants consideration, although it appears to be seldom used by labs.

The choice of recovery methods was much less diverse than for extraction, comprising almost entirely of the Rotary Evaporator and Abson Method. The Rotovap had less binder modification concerns than the Abson in terms of aging and residual solvent.

For the solvent, trichloroethylene was by far the most common solvent used, with two-thirds of users. N-Propyl Bromide and Toluene also have significant occurrences. Unfortunately, all of these solvents are volatile, dangerous to humans and the environment. There was very little in terms of bio-sourced solvent options as the only ones identified were Orange Bio-Act and Terpene Extract. Given the risks that come from solvents used in these procedures, a lot more work could be done in developing bio-sources and environmentally friendly solvents for asphalt binder.

The determination of binder content was found to be consistent or very consistent, indicating that this was not a major concern. The most common concerns about the performance of extraction and recovery procedures was regarding the aging of the binder and solvent remaining in the binder, although about half of respondents said they did not have major concerns. However there clearly is a need for more research in binder aging and solvent distillation with these procedures (12). The recovery of RAP and PMAs were also identified as a challenge as found in previous studies (13), as due to the stiffer nature of the binders, it is more difficult to break them down in extraction.

The average time to perform extraction and recovery procedures was around 5 hours, with a significant amount of respondents indicating 2-4 hours. In terms of improving the methods, suggestions included the use of larger flasks as well as the verification of solvent presence in the binder by FTIR spectrometry.

Volatiles were the biggest concern in terms of operator health. Handling of samples at hot temperatures as well as corrosive and carcinogenic chemicals were also significant safety concerns. The use of fume hoods, ventilation masks, eye goggles, latex and nitrile gloves, full length clothes were among the suggestions provided for improving safety, although these are already in most laboratory safety protocols.

Toxic chemicals and waste disposal were the most common environmental concerns, with volatiles also being significant. A more environmentally as well as health and safety solvent would go a long way in this regard (14).

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