Development of a simple RAP extraction and recovery method for smaller material quantities

Dheeraj Adwani
Department of Civil, Architectural and Environmental Engineering
The University of Texas at Austin, Texas, USA

Pravat Karki
Texas Department of Transportation (TxDOT), Austin, Texas, USA

Amit Bhasin
Department of Civil, Architectural and Environmental Engineering
The University of Texas at Austin, Texas, USA

ABSTRACT: As road networks expand globally and existing pavements are upgraded, the need for recycling in asphalt pavements continues to grow. However, the quality of RAP materials varies significantly, necessitating reliable assessment methods to determine their suitability for new pavements and ensure optimal performance. Rapid and efficient RAP quality assessment is especially critical in regions managing multiple RAP stockpiles, where informed decisions rely on accurate material evaluation. This study presents a novel microextraction procedure combined with a vacuum oven recovery method to extract small binder quantities for characterization. The vacuum oven recovery method was developed and refined through simulation trials using binder pellets, while various microextraction filtration techniques were evaluated with loose asphalt mixtures. The proposed procedure was validated through rheological characterization of binders and benchmarked against conventional extraction and recovery methods, demonstrating its efficacy and potential for widespread application.

1 INTRODUCTION

1.1 *Background and Motivation*

The incorporation of reclaimed asphalt pavement (RAP) in road construction has become nearly indispensable in today's world due to the need for recycling, sustainability, and cost considerations. However, the quality of RAP varies significantly, influenced by factors such as its source, location, age, and the original materials used in its composition. This variability results in a wide range of RAP binder quality across different stockpiles. For instance, the state of Texas alone exhibits substantial diversity in RAP characteristics, necessitating rapid and efficient methods for quality assessment to support informed decision-making.

Each RAP possesses unique properties, making proper evaluation critical for distinguishing between materials of differing quality. Among the key components of asphalt mixtures, the asphalt binder plays a crucial role in determining the long-term performance of the pavement. To assess RAP binder quality, the binder properties must be characterized, which requires its extraction from RAP materials. Conventional binder extraction and recovery methods typically involve the use of large quantities of organic solvents and specialized laboratory setups. Centrifuge-based methods are widely employed for binder extraction, where the RAP is soaked in an organic solvent to obtain a binder-solvent solution. For binder recovery, methods such as the Abson recovery technique and the use of a rotary evaporator (rotovap) are commonly utilized (McDaniel et al., 2000).

Although these conventional methods are well-established and widely used, they are inherently time-consuming, require large volumes of solvents, and have limited throughput. A single centrifuge and rotovap setup in a laboratory can process only one RAP binder sample at a time, making the evaluation process slow and inefficient, particularly for regions with numerous RAP stockpiles. Additionally, the proposed EPA ban on trichloroethylene (TCE) for laboratory use presses the urgent need for alternative methods that employ safer, less chlorinated solvents like toluene. As the asphalt industry faces the need to evaluate large volumes of RAP, faster, safer, and high-throughput methods are becoming essential.

Recent advancements in dynamic shear rheometer (DSR) technology have enabled the use of smaller diameter plates, such as 4 mm and 8 mm plates, for low temperature testing (Hajj et al., 2019, Filonzi et al., 2020). This allows comprehensive rheological characterization, including high, intermediate, and low temperature testing, to be performed exclusively on the DSR using minimal binder quantities. Consequently, RAP extraction and recovery methods that yield small binder quantities can be highly effective, provided they are fast, simple, and capable of high throughput.

1.2 *Scope*

This study focuses on the development of a microextraction and recovery method for RAP. To establish the vacuum oven recovery method using toluene as the solvent, initial trials were conducted with simulated binder pellets, without using RAP or asphalt mixtures. Through multiple iterations, key recovery parameters, such as temperature, duration, vacuum level, and other relevant conditions, were optimized. Subsequently, the refined procedure was applied to loose asphalt mixtures, simulating lab-produced RAP.

The rheological characteristics results for the recovered binders were compared with binders recovered through conventional extraction and recovery to validate the developed approach. The proposed method offers a simple, fast, and high throughput method to recover small binder quantities, making it highly suitable for RAP quality assessment.

2 METHOD DEVELOPMENT WITH BINDER PELLETS

2.1 *Simulated binder-solvent solution*

For method development, the researchers simulated the proposed approach using asphalt binders to establish extraction and recovery parameters. Instead of utilizing asphalt mixtures or RAP samples, asphalt binder pellets made from RTFO and PAV aged binders were dissolved in toluene inside a glass bottle. During the micro-recovery simulations, small 2 g binder pellets were dissolved in 20 ml solvent, as illustrated in Figure 1.

To ensure homogeneity, the binder-solvent solution was placed on a magnetic stirrer equipped with a chemical-resistant covered magnet inside the bottle. The solution was stirred at approximately 200 rpm for an extended duration, preferably overnight, to achieve complete dissolution.

Figure 1. Binder pellet dissolved in a solvent in a glass bottle stirred on a magnetic stirrer.

2.2 *Vacuum oven recovery procedure*

Binder recovery was conducted using a conventional vacuum oven. The binder-toluene solution was transferred to 4 oz steel cans, commonly used in asphalt laboratories for binder storage. Earlier trials employed glass dishes for this process, but to increase throughput, the glass dishes were replaced with disposable 4 oz steel cans (Filonzi et al. 2020). These cans containing the solutions were placed in the vacuum oven for recovery. The oven's initial temperature was adjusted based on the solvent, which in this study was toluene, a solvent with a relatively high boiling point. The temperature was gradually increased from 60°C to 165°C, with increments of 20–25°C every 30 minutes, making the entire recovery process approximately 5 hours long. Figure 2 illustrates a typical vacuum oven setup with samples undergoing the recovery procedure.

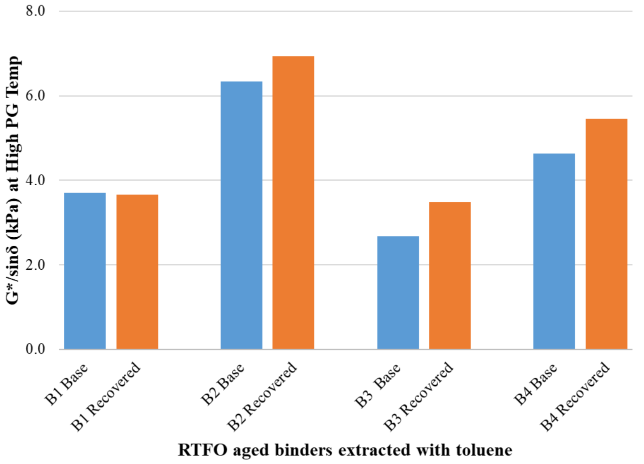
Figure 2. Vacuum oven used for recovery of binder from the binder-solvent solution.

A vacuum of 70 cm of Hg was applied in the oven using a chemical-resistant vacuum pump. This high vacuum level is critical for reducing the boiling point of the solvent, facilitating efficient removal of solvent vapors from the oven while minimizing binder aging at elevated temperatures. The amount of binder solution in the sample cans was carefully calibrated to ensure that the recovered binder quantity was sufficient for DSR measurements.

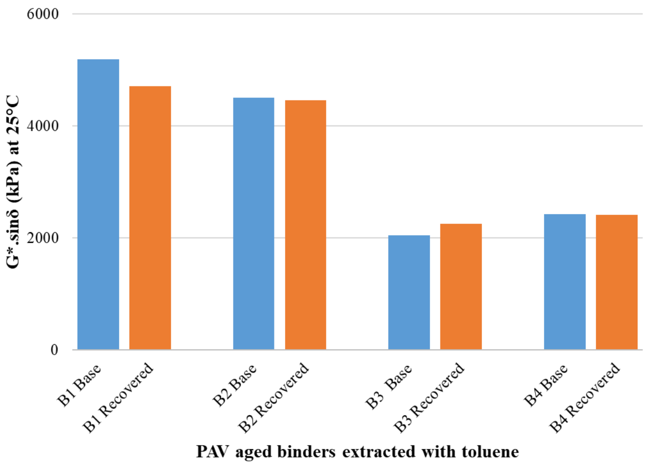
For these simulations, conventional high- and intermediate-temperature PG testing was performed, with complex modulus, phase angle, and stiffness parameters compared between the base binders and the recovered binders. Trials were conducted on four binders (designated B1, B2, B3, and B4), representing three different PG grades: two PG 64-22, one PG 70-22, and one PG 76-22, each in both RTFO and PAV aged conditions.

2.3 *DSR evaluation on recovered simulated binder*

The DSR results for 25 mm plate testing of RTFO aged base and recovered binders are presented in Figure 3 (a). As shown, the $G^*/sin\delta$ values for the base RTFO binders and binders recovered through vacuum oven recovery simulation were highly comparable, with negligible d2s% differences between the two measurements. These findings indicate that, from a rheological standpoint, toluene is an effective solvent for recovering the original binder using the vacuum oven micro-recovery process, with complete removal of the solvent during recovery. Similarly, Figure 3 (b) presents the $G^*.\sin\delta$ values for base and recovered PAV aged binders. The results further confirm the effectiveness of the toluene-based recovery process, demonstrating the success of the simulation trials in preserving the rheological properties of the original binders, and no evidence of solvent in the recovered binder.



(a)



(b)

Figure 3. Stiffness parameters at high and intermediate temperatures for RTFO and PAV aged original and recovered binders.

3 MICROEXTRACTION AND RECOVERY TRIALS WITH LOOSE ASPHALT MIXTURES

The previously described recovery trials simulated the recovery of binder from binder-solvent solutions using the vacuum oven procedure. The preceding step obtaining the binder-solvent solution involves extracting binder from the RAP, or lab simulated RAP in this study in the form of loose asphalt mixture. This section focuses on the efforts to develop a fast and simple microextraction method to obtain the binder-solvent solution ready for recovery process.

3.1 *Different filtration methods*

The binder microextraction process begins by soaking asphalt mixture or RAP in a solvent for a sufficient duration to dissolve the binder into the solvent. Approximately 50 g of loose mixture was soaked in 50 mL of toluene within a glass bottle, same as the one illustrated in Figure 1. The solvent amount was adjusted based on the container dimensions to ensure the mixture was adequately submerged. A magnetic stirrer was used to agitate the mixture at around 200 rpm overnight, ensuring effective binder dissolution.

The next step involved filtering the binder-solvent solution to separate aggregates and fine materials. Previous research utilizing 1-micron syringe filters effectively removed fine particles but required a significant number of filters when processing mixtures with higher fine content (Filonzi et al. 2020).

Two plant-produced loose asphalt mixtures were used for these trials. The first was produced using a PG 64-22 binder, and the second with a PG 70-22 binder, both incorporating 20% RAP. Several filtration methods were explored, summarized as follows:

3.1.1 *Method-1*

This trial introduced a two-stage filtration process to improve efficiency over earlier methods. The first stage utilized generic coffee filters with a pore size of 10–20 microns to remove larger particles, as shown in Figure 4. Vacuum flask assistance was tested to expedite this step. In the second stage, the filtrate was passed through a 1-micron syringe filter with vacuum assistance (Figure 4). While this method slightly reduced the number of filters needed, the additional effort did not justify the limited savings in efficiency.

Figure 4. Trial with coffee filter as the first stage in the two-step filtration and vacuum manifold with syringe filters

3.1.2 *Method-2*

In this method, a single-stage filtration using 1-micron syringe filters was employed. To reduce the number of filters required, the binder-solvent solution was left undisturbed overnight after a few hours stirring, allowing fine particles to settle. This waiting period reduced the load on the filters without compromising the homogeneity of the solution.

3.1.3 *Method-3*

This method eliminated magnetic stirring, instead manually stirring the solvent-mixture solution for a few minutes after a few hours of soaking time. The bottle was then left undisturbed overnight or around 16 hours depending on the operator's schedule, allowing fines to be settled. After the settling period, the supernatant was manually drawn into a syringe through a 1-micron filter directly attached to the syringe as shown in Figure 5, significantly reducing the number of filters needed and simplifying the process. This approach efficiently recovered the binder with minimal time and filter usage.

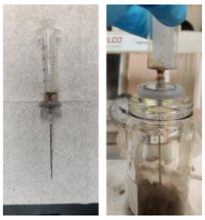
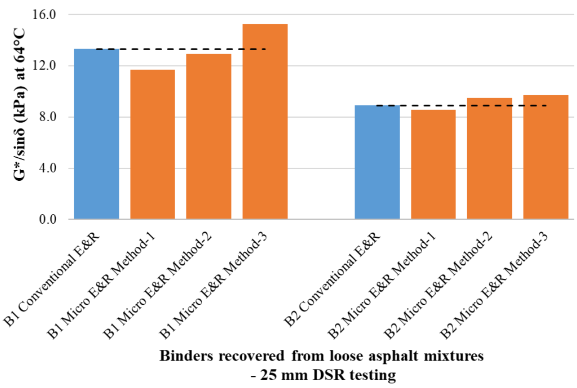


Figure 5. Manual method of drawing supernatant through a syringe filter from the undisturbed solution (without vacuum assistance and stirring)

The filtrates obtained through the methods discussed above were then subjected to the vacuum recovery procedure outlined earlier.

3.2 *DSR measurements on recovered binders*

The binders recovered from the filtrates were tested for DSR measurements and compared to binders recovered using conventional extraction and recovery with a centrifuge and rotovap. The recovered binders were tested for 25 mm high temperature and 8 mm intermediate temperature PG, and stiffness parameters $G^*/\sin\delta$ at 64°C and $G^*.\sin\delta$ at 25°C were compared. The results, shown in Figure 6, clearly demonstrate that all microextraction and recovery trials produced binders with stiffness values similar to those recovered by conventional methods. These findings confirm that the microextraction and recovery method is a suitable, efficient approach for recovering binder from RAP or asphalt mixtures in small quantities.



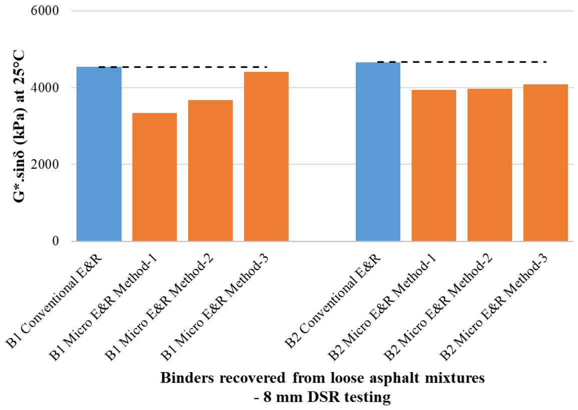


Figure 6. Summary of stiffness parameters for recovered binders from different extraction and recovery methods

4 CONCLUSIONS AND DISCUSSION

This study introduces a simple, rapid, and safer microextraction and recovery method for assessing RAP binder quality. The developed method uses just 50 g RAP and 50 ml of solvent in a glass bottle, syringes, filters, and a vacuum oven to extract and recover sufficient binder for DSR rheological testing.

The proposed method was validated through rheological characterization of the recovered binders and benchmarked against conventional extraction and recovery method. The proposed method provides a practical, efficient, and high-throughput solution for RAP quality assessment, and supports the asphalt industry in achieving sustainability goals.

5 ACKNOWLEDGEMENTS

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6 DISCLAIMER

This study does not constitute a standard or specification or represent views of TxDOT.

7 REFERENCES

McDaniel, R.S. and Anderson, R.M., 2001. Recommended use of reclaimed asphalt pavement in the Superpave mix design method: technician's manual (No. Project D9-12 FY'97). Transportation Research Board.

Hajj, R., Filonzi, A., Rahman, S. and Bhasin, A., 2019. Considerations for using the 4 mm plate geometry in the dynamic shear rheometer for low temperature evaluation of asphalt binders. Transportation Research Record, 2673(11), pp.649-659.

Filonzi, A., Lee, S.K., Ferreira, W., Hajj, R. and Bhasin, A., 2020. A micro-extraction method for use with 4 mm plate geometry in the Dynamic Shear Rheometer to evaluate asphalt binder rheology. Construction and Building Materials, 252, p.119024.