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Critical considerations and effective assessment of extraction and recovery processes of RAP

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ABSTRACT

With the increasing shortage of resources, the reuse of recycled asphalt pavements (RAP) in pavement engineering is considered as a sustainable technology. Challenges posed by common extraction and recovery methods may result in misjudgment of asphalt pavement performance. In this study, we investigate the optimization of extraction and recovery processes in recycled asphalt pavement (RAP) recycling, aiming to promote sustainable development within the pavement engineering sector. We prepared eleven asphalt samples to simulate common extraction and recovery scenarios, using virgin SBS-modified asphalt as a reference. Employing Fourier Transform Infrared (FTIR) analysis, thermogravimetric analysis (TGA), and Dynamic Shear Rheometer (DSR) testing, we assessed the samples' rheological and chemical properties. We pointed out three common but easily overlooked problems in the extraction and recovery process, namely residual mineral powder, residual trichloroethylene, and incomplete extraction. Residual mineral powder and trichloroethylene greatly influence extraction recovery accuracy; high-speed centrifugation effectively addresses trichloroethylene, but completely removing mineral powder remains challenging. Accurate evaluation of residual substances in recycled asphalt is achievable through FTIR, TGA, and rheological tests, providing valuable insights for material selection and processing. Additionally, it is crucial to fully recover the binder from RAP for precise performance evaluation, as the binder's interior exhibits lower aging levels compared to the surface. This aging heterogeneity should be considered when assessing RAP performance and developing effective rehabilitation strategies. Our findings hold significant implications for enhancing the efficiency and effectiveness of extraction and recovery processes in RAP recycling, ultimately contributing to sustainable development in pavement engineering.

1. Introduction

The recycling of waste building materials can significantly reduce energy consumption during reproduction, decrease greenhouse gas emissions, and facilitate sustainable development [18,14]. During road construction, reclaimed asphalt pavement (RAP) is extensively employed in various forms to conserve nonrenewable asphalt and aggregate resources, protect the environment, and minimize investment [17,5,9,28]. Throughout its service life, RAP's binder may undergo physical or chemical reactions such as volatilization, oxidation, degradation, and polymerization due to adverse factors like high temperature, oxygen, water, and ultraviolet light. These reactions can alter the binder's chemical components, ultimately deteriorating road performance [6,7,13,15]. To comply with specification requirements, the binder must be extracted from RAP to assess its properties and content, enabling the determination of appropriate recycling agent types and doses [26,8,21]. Thus, it is crucial to separate the binder and aggregate in RAP using

effective methods [29–31].

Centrifugal extraction and rotary evaporation are common techniques for asphalt recovery. These methods involve dissolving the binder on the aggregate surface in an organic solvent and separating it from the solvent through rotary evaporator to obtain the RAP binder. Various solvents are used in the extraction and recovery process, such as trichloroethylene, dichloromethane benzene, toluene, a mixture of toluene and ethanol, or trichloroethane [4,22]. Trichloroethylene is a widely utilized and highly effective solvent for asphalt extraction and recovery due to its powerful solvent properties [11].

Although established procedures for extraction and recovery tests exist (ASTM-D D5404M-21), the type of asphalt, the proportion of mineral powder, and the degree of aging are all different in different RAP [2,3], so there may still be some problems during the extraction and recovery process. Three common issues can affect the results of extraction and recovery during actual operation. Firstly, inadequate centrifugation or filtration during the centrifuge extraction process can result in residual mineral powder in the asphalt and trichloroethylene solution,

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Nomenclature

RAP	Recycled asphalt pavements
SBS	Styrene butadiene styrene block copolymer
TGA	Thermogravimetric analysis
DSR	Dynamic shear rheometer
FTIR	Fourier transform infrared spectroscopy
MA	Modified asphalt
TCE	Trichloroethylene
PB	Polybutadiene

impacting the physical properties of the final recycled asphalt and leading to an inaccurate assessment of asphalt aging [12]. Furthermore, even though the residual mineral powder to asphalt ratio during the extraction and recovery process is often lower and does not reach 1:1, the presence of these minerals can still affect the FTIR spectrum between 600 and 1600 cm^{-1} . This, in turn, influences the area of carbonyl groups, polybutadiene, and other functional groups, which may potentially lead to inaccuracies in the evaluation [23,20]. Secondly, the number of centrifugal extraction cycles affects the recycled asphalt's performance. Trichloroethylene cannot dissolve all bitumen in RAP in one cycle, and the process usually requires multiple iterations to meet requirements [19]. Insufficient asphalt recovery can lead to improper evaluations of asphalt aging [1]. Lastly, during rotary evaporation, trichloroethylene solvent may remain in the recycled asphalt binder due to improper oil bath temperature and rotation time, softening the asphalt binder and underestimating the actual stiffness of the asphalt binder [25,1,16]. These frequent errors during extraction and recovery may result in inaccurate conclusions regarding the extent of binder aging [10]. Owing to the absence of effective judgment methods, such errors sometimes go undetected, which can significantly impact the efficient utilization of RAP and negatively affect sustainable development.

In this study, we aimed to simulate the common situations encountered during the extraction and recovery process by preparing a series of samples. We utilized Fourier Transform Infrared (FTIR) analysis, thermogravimetric analysis (TGA), and Dynamic Shear Rheometer (DSR) testing to examine the rheological and chemical properties of these samples. Our goal was to analyze the influence of various factors (i.e. residual mineral powder, residual trichloroethylene and only a portion of the asphalt has been recycled) present during the extraction and recovery process on the samples' rheological properties, functional group distribution, and thermal stability. Following this analysis, we aimed to establish technical indicators that can effectively assess the accuracy of extraction and recovery processes, so that experimenters can quickly identify problems and make targeted adjustments in a timely manner. Additionally, we endeavored to provide recommendations for enhancing the efficiency and effectiveness of these processes in the context of reclaimed asphalt pavement recycling and the promotion of sustainable development.

2. Experimental design

2.1. Methods of extraction and recovery

2.1.1. Centrifugal extraction method

Asphalt extraction was done according to Standard Test Methods for Quantitative Extraction of Asphalt Binder from Asphalt Mixtures (ASTM D2172). Extraction is to separate the asphalt from the mineral aggregates in RAP. The purpose of extraction is to provide the mixture of asphalt and trichloroethylene for asphalt recovery in the next step. Extraction was divided into the following steps:

- (1) 1000 g of RAP was placed into the bowl of the extraction apparatus.
- (2) Trichloroethylene was added into the bowl, and the RAP was completely immersed in trichloroethylene. The trichloroethylene and RAP were mixed with filter rod, then stewing one hour so that the bitumen in RAP could be fully dissolved by the trichloroethylene.
- (3) A piece of filter paper was covered on the bowl to filter the mineral powder. A beaker was placed under the drain to collect the extract. The machine was started and maintaining at a certain speed until no liquid flows out of the drain.
- (4) The procedure (2) and (3) were repeated until the extract was nearly transparent.
- (5) High-speed centrifuge was used to process the asphalt extract, and the centrifugal speed was set to 3000 r/min for 15 min.

2.1.2. Rotary evaporator method

Asphalt recovery was done according to Standard Practice for Recovery of Asphalt Binder from Solution Using the Rotary Evaporator (ASTM D5404 M-21). Rotary evaporator was divided into the following steps:

- (1) The asphalt extract (120 ~ 180 ml) was put into a 500 ml rotating flask and the vacuum pump was started to form a negative pressure in the whole system, with a vacuum degree of 94.7 kPa.
- (2) The rotating flask was started, rotating for 5 ~ 10 min at the speed of 50 r/min without immersing in the hot oil bath, and then the flask was immersed in the oil bath at 50 °C, and the solvent in the flask evaporated into gas. Then heating area of the flask immersed in the oil bath was increased until there was no trichloroethylene distilled out.
- (3) As the viscosity of aged modified asphalt was higher than unaged, the oil bath was heated to 165 °C, and then the flask was rotated continuously for 20 min (Standard time is 15 min).
- (4) When no trichloroethylene was distilled, the carbon dioxide valve was opened for 2 min at the flow rate of 1000 ml/min, then the carbon dioxide valve was closed. The device was restored to normal pressure gradually, the rotating flask was stopped and left the oil bath, and then the device was disassembled.
- (5) The residual asphalt in the flask was poured out for subsequent test.

2.2. Sample preparation

In this investigation, reclaimed asphalt pavement (RAP) was sourced from the surface asphalt mixture layer of the Cheng'ya highway pavement in Sichuan Province, China, which had been in service for over nine years. The thickness of asphalt layer is 4 cm and the core sample was obtained from the wheel path in the pavement. The asphalt mixture utilized in the upper layer incorporated a styrene-butadiene-styrene (SBS) modified binder. The SBS modified binder is produced by Sinopec, and we use this asphalt as the test object. It should be noted that we are using virgin asphalt, not RAP binder. The properties of the virgin SBS modified binder are detailed in Table 1. The indexes of the mineral powder used in this study are presented in Table 2. A comprehensive overview of the asphalt binder samples used in this study is presented in Table 3. Each binder sample was tested three times to ensure the

Table 1
Indexes of virgin SBS modified asphalt binder produced by Sinopec.

Item	Unit	SBS modified asphalt binder
Penetration (25 °C, 100 g, 5 s)	0.1 mm	45
Softening point ($T_{R\&B}$)	°C	73.9
Ductility (5 °C, 5 cm/s)	cm	56.9
Viscosity (135 °C)	Pa·s	2.378

Table 2
Indexes of the mineral powder.

Item	Unit	The mineral powder
Density	t/m ³	2.73
Water content	%	0.37
Passing rate of 0.075 mm	%	93.5
Plasticity index	%	2.6

Table 3
A summary of all asphalt binder samples tested in this study.

No.	Code name	Description and sampling location	Purpose
1	MA-Virgin	SBS modified asphalt	Provide a reference substance.
2	MA-2.5 % Mineral	2.5% Mineral powder was added in SBS modified asphalt (the mass ratio of mineral powder and asphalt is 1:40, the mass ratio of asphalt).	Compare with MA-Virgin to discuss the effect of residual mineral powder.
3	MA-5 % Mineral	5% Mineral powder was added in SBS modified asphalt (the mass ratio of mineral powder and asphalt is 2:40, the mass ratio of asphalt).	Compare with MA-Virgin to discuss the effect of residual mineral powder.
4	MA-7.5 % Mineral	7.5% Mineral powder was added in SBS modified asphalt (the mass ratio of mineral powder and asphalt is 3:40, the mass ratio of asphalt).	Compare with MA-Virgin to discuss the effect of residual mineral powder.
5	MA-10 % Mineral	10% Mineral powder was added in SBS modified asphalt (the mass ratio of mineral powder and asphalt is 4:40, the mass ratio of asphalt).	Compare with MA-Virgin to discuss the effect of residual mineral powder.
6	MA-TCE	SBS modified asphalt dissolved by trichloroethylene (TCE), the mass ratio is 1:3, and then recovered. Compared to the normal operation process, the rotary evaporation time is reduced by 5 min.	Compare with MA-Virgin to discuss the effect of residual TCE.
7	MA-TCE-Mineral	5% Mineral powder was added in SBS modified asphalt dissolved by trichloroethylene (the mass ratio of mineral powder and asphalt is 1:20, the mass ratio of asphalt and trichloroethylene is 1:3), and then filtered for recovery.	Compare with MA-Virgin to discuss the effect of residual mineral powder and residual TCE.
8	MA-Centrifuge	Mineral powder was added in SBS modified asphalt dissolved by trichloroethylene (the mass ratio of mineral powder and asphalt is 1:20, the mass ratio of asphalt and trichloroethylene is 1:3), and then centrifuged, filtered and recycled.	Compare with MA-TCE-Mineral to discuss the effect of centrifuge.
9	MA-Mixture	SBS modified asphalt was used to mix AC-13 hot asphalt mixture.	Compare with MA-Centrifuge to discuss the effect of centrifugal extraction.
10	RAP	Extracted from the pavement which endured a 10-year long-term aging.	Provide a reference substance.
11	RAP-surface	The process of extraction was carried out twice, and then the extract was recovered. At this time, the extract was black.	Compare with RAP to discuss the effect of the times of centrifugal extraction.
12	RAP-inside	After two times of rap extraction, two more times of extraction shall be carried out, and then the extract was recovered. At this time, the extract was nearly transparent.	Compare with RAP to discuss the effect of the times of centrifugal extraction.

reliability of the results and the steps involved in sample preparation are illustrated in Fig. 1.

2.3. DSR tests

2.3.1. Complex shear modulus test

The complex shear modulus tests were carried out on the extracted and recovered binders using the TA dynamic shear rheometer (DSR) AR1500ex. The complex shear modulus (G^*) and phase angle (δ) of the samples were measured at a temperature of 76 °C. According to the AASHTO T 315, the test was performed with 25 mm parallel plate and a gap of 1 mm at a frequency of 10 rad/s. Two replicates were measured for each binder sample.

2.3.2. Multiple stress creep recovery (MSCR) test

The MSCR test was employed to determine the rutting resistance and elasticity of extracted and recovered binders according to AASHTO T 350. The sample geometry and testing temperature for complex shear modulus tests was used for the MSCR tests. In each creep and recovery cycle, a shear load was applied to the sample for 1 s, followed by a rest period of 9 s. The average values of non-recoverable creep compliance Jnr3.2 and percent recovery R3.2 from two replicates were reported.

2.4. Fourier Transform infrared spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) was conducted to observe the impurity in the recovered asphalt, i.e. mineral powder and trichloroethylene. In this study, the obvious absorption peaks appear near 871 cm⁻¹ (Si—O, the silicon-oxygen bond) and 930 cm⁻¹ (trichloroethylene) due to impurities in the recovered asphalt in the extraction and recovery process [10,20,27]. The change of carbonyl group at a wavenumber of 1700 cm⁻¹ in the spectrum, which is the most commonly used functional group for identifying binder aging, was employed to quantify the aging extent of extracted and recovered binders. Besides, the change of polybutadiene peak (PB peak at 966 cm⁻¹) was measured to determine the degradation of SBS polymer in the extracted and recovered binders [15,24]. Since the testing sample's thickness and infrared radiation path length both have influence on the intensity of absorption peaks of FTIR spectra, it is better to use the index for comparison reason than their absolute values of peak area or peak height. The index of Si—O bond (I_{Si-O}), the carbonyl index $I_{C=O}$ and PB index I_{PB} are calculated by using the equations (1)–(3),

$$I_{Si-O} = \frac{\text{Area of the spectral band centered around } 871 \text{ cm}^{-1}}{\sum \text{Area of the spectral bands between } 4000 \text{ and } 600 \text{ cm}^{-1}} \quad (1)$$

$$I_{C=O} = \frac{\text{Area of the spectral band centered around } 1070 \text{ cm}^{-1}}{\sum \text{Area of the spectral bands between } 4000 \text{ and } 600 \text{ cm}^{-1}} \quad (2)$$

$$I_{PB} = \frac{\text{Area of the polybutadiene band centered around } 966 \text{ cm}^{-1}}{\sum \text{Area of the spectral bands between } 4000 \text{ and } 600 \text{ cm}^{-1}} \quad (3)$$

2.5. Thermal gravity analysis (TGA)

Thermal gravity analysis (TGA) is a simple analytical method to measure the change of material weight with the change of temperature. The tests were conducted on Perkin Elmer TGA 4000 thermogravimetric analyzer. Asphalt samples were 5 ~ 10 mg and all tests were conducted under the protection of nitrogen (40 ml·min⁻¹). The test temperature range was 50 ~ 700 °C and the heating rate was 15 °C /min. The percentage weight loss and corresponding temperatures were recorded by TGA.

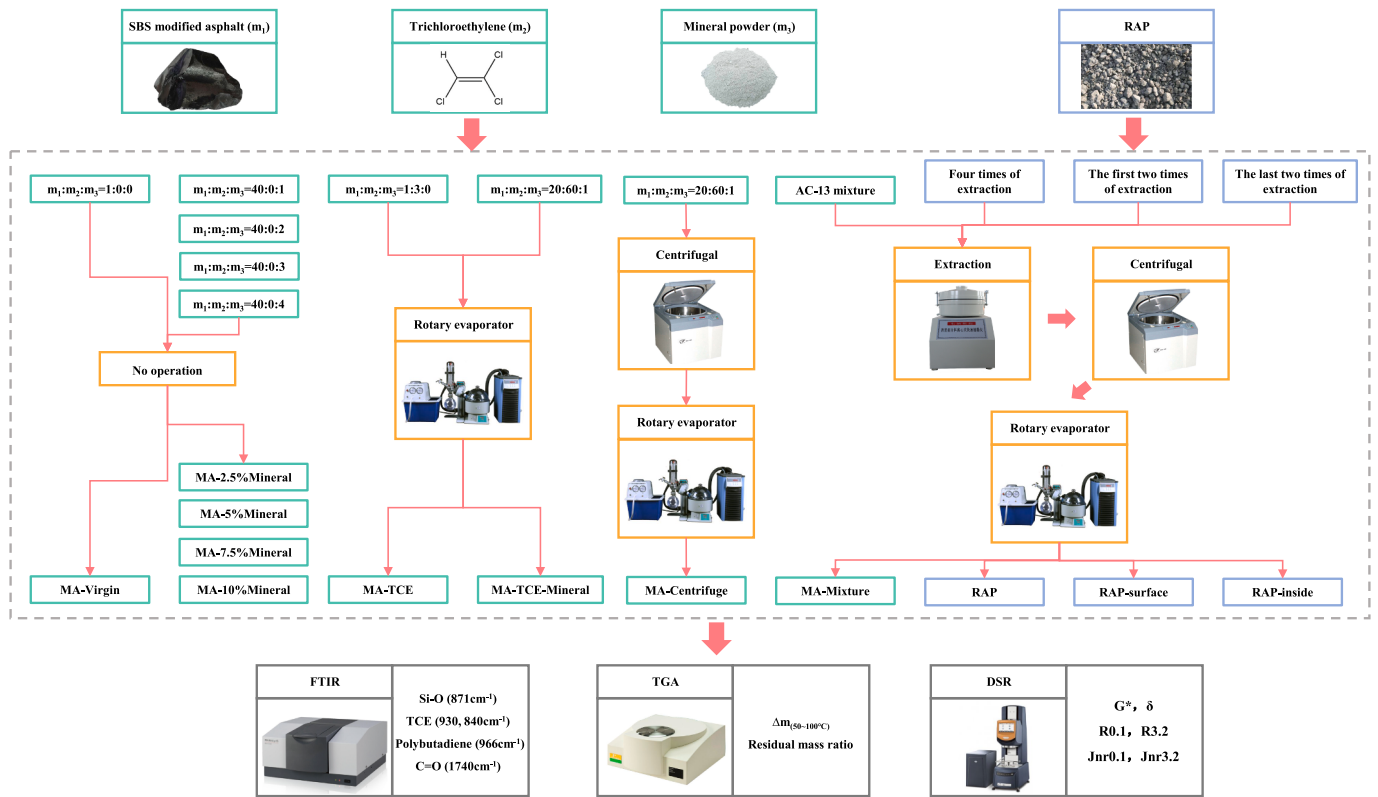


Fig. 1. Summary of the sample preparation process.

3. Results and discussion

3.1. FTIR result analysis

The Fourier Transform Infrared (FTIR) spectroscopy results are depicted in Fig. 2. A comparative analysis of MA-Virgin, MA-2.5% Mineral, MA-5% Mineral, MA-7.5% Mineral, and MA-10% Mineral reveals a prominent absorption peak at 871 cm^{-1} upon the addition of mineral powder to the asphalt. Furthermore, the area of the absorption peak progressively expands as the mineral powder content increases.

The absorption peaks of MA-Centrifuge and MA-Virgin coincide, with no discernible differences observed between the two. This suggests that no chemical reaction occurs between trichloroethylene and virgin asphalt, confirming the suitability of trichloroethylene as a solvent for extraction and recovery. Moreover, the 871 cm^{-1} absorption peak in the centrifuged samples closely resembles that of MA-Virgin, implying that centrifugation effectively eliminates residual mineral powder. Relative to MA-Virgin, the FTIR spectra of MA-TCE exhibit two conspicuous absorption peaks at 840 cm^{-1} and 930 cm^{-1} due to residual trichloroethylene. This indicates that these peaks can be utilized to identify the

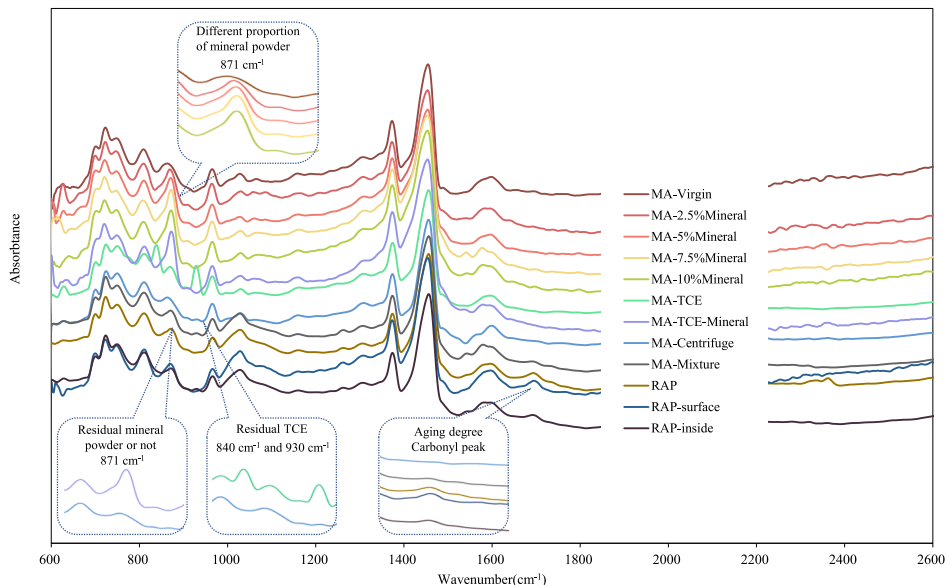


Fig. 2. Summary of the infrared spectra.

presence of residual minerals.

For the Reclaimed Asphalt Pavement (RAP), the peak around 1740 cm^{-1} is attributed to the carbonyl group in the SBS modified asphalt binder spectrum. Long-term aging leads to the formation of ketones, aldehydes, esters, carboxylic acids, and other oxygenates, which consequently increases the carbonyl group content. In comparison to MA-Virgin, the asphalt binder extracted from RAP exhibits a significantly higher carbonyl peak, indicating severe aging in the field. Additionally, the differences between RAP-surface and RAP-inner samples illustrate the gradation of aging in the thickness of the aged asphalt binder on the RAP surface.

In order to quantitatively analyze the influence of mineral powder, trichloroethylene, and the number of extraction iterations, the area of the absorption peak is quantified and depicted in Figs. 3–6, respectively.

As illustrated in Fig. 3, due to the presence of mineral powder, prominent absorption peaks emerge at 871 cm^{-1} in the spectrum. In comparison to MA-Virgin, the addition of 2.5%, 5%, 7.5%, and 10% mineral powder into the asphalt results in an increase in the absorption peak area by 89%, 115%, 152%, and 162%, respectively. The absorption peak area decreases by 73% after centrifugation. The peak area at 871 cm^{-1} , situated between 900 and 837 cm^{-1} , is attributed to silicon-oxygen-silicon (Si-O-Si) or silicon-oxygen-aluminum (Si-O-Al) bonds. Thus, centrifugation proves to be an effective method for reducing the mineral powder content in the extract.

Fig. 4 demonstrates that the absorption peak area around 840 cm^{-1} and 930 cm^{-1} are zero for all samples except MA-TCE, indicating that the modified rotary evaporation process is a feasible approach for removing residual TCE from the extracted asphalt binder and ensuring the accuracy of subsequent experimental characterizations.

Carbonyl index (I_{CA}) and PB index are examined for all samples and presented in Figs. 5 and 6, respectively. The carbonyl index typically indicates the oxidation degree of asphalt, while the PB index represents the SBS polymer degradation level. Both indices can be utilized to evaluate the aging degree of asphalt binder.

Irrespective of the inclusion of centrifugation or rotary evaporation processes, and regardless of the addition or removal of mineral powder or trichloroethylene, the carbonyl and PB indices display minimal fluctuations. This indicates that the rotary evaporation process and centrifugation during extraction do not substantially affect the aging of

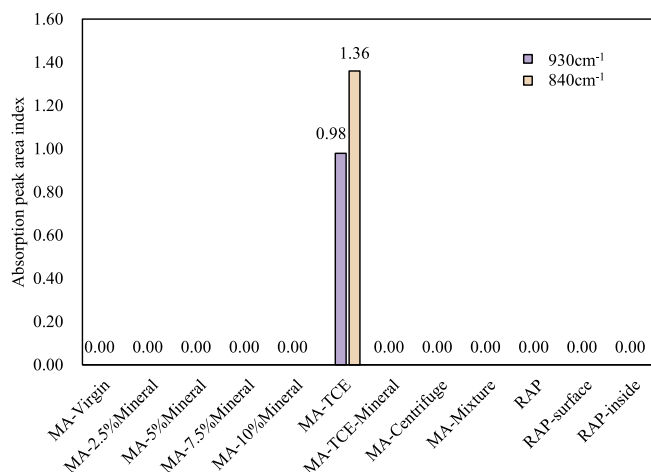


Fig. 4. Summary of absorption peak area index at 840 cm^{-1} and 930 cm^{-1} .

the extracted asphalt binder.

Notably, MA-Mixture exhibits a considerably higher carbonyl index and a markedly lower polybutadiene index. The primary difference between MA-Mixture and MA-Virgin is the short-term aging of MA-Mixture introduced during asphalt mixture preparation. This indicates that the short-term aging process significantly degrades the SBS polymer and causes substantial oxidation of the asphalt phase. Consequently, it is recommended to evaluate the performance of SBS modified asphalt after aging.

The carbonyl index of RAP-surface is 1.86 times greater than that of RAP-inside, suggesting a higher carbonyl content in asphalt near the outer layer due to increased exposure to sunlight, air, and water. In comparison to RAP-surface, the PB index of RAP-inside increases by 9.6%, which is expected since RAP-inside experiences less exposure to ultraviolet rays and oxygen during long-term service.

In summary, from the perspective of infrared spectroscopy, mineral powder and trichloroethylene have minimal impact on the evaluation of asphalt aging degree, while the number of extraction iterations plays a more significant role.

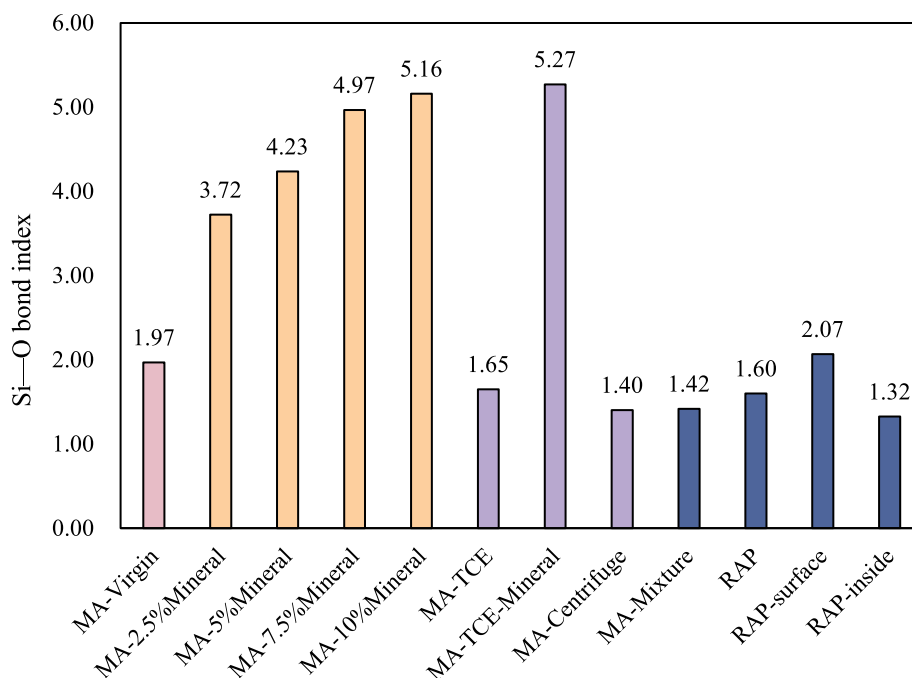


Fig. 3. Summary of Si-O bond index calculated from FTIR analysis.

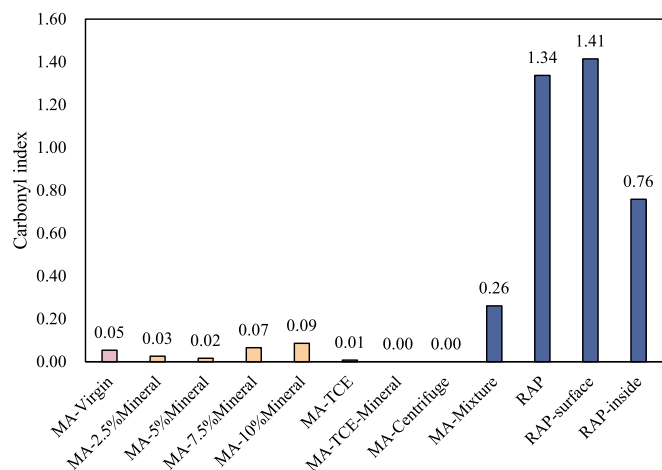


Fig. 5. Summary of carbonyl index calculated from FTIR analysis.

3.2. TGA result analysis

A summary of the TGA curves for the samples under a nitrogen atmosphere is presented in Table 4. A subtle mass loss occurs within the 50 °C ~ 100 °C range, which can be attributed to the volatilization of residual trichloroethylene in the asphalt samples. The boiling point of trichloroethylene is 87 °C. The residue of MA-RAP at 700 °C is higher than that of the Virgin sample. As mineral powder remains in the final residue, samples containing mineral powder exhibit a greater residual mass ratio compared to those without mineral powder. This difference is approximately equivalent to the proportion of mineral powder in the samples.

3.3. Complex modulus and phase angle analysis

3.3.1. Complex modulus result analysis

The complex modulus of samples is summarized in Fig. 7. The presence of 2.5% mineral powder significantly increases the complex modulus of asphalt; however, further increases in mineral powder

proportion do not lead to additional increases in the complex modulus. This indicates that mineral powder increases the binder's stiffness, but further addition of filler has limited effect. Compared to virgin asphalt, asphalt with trichloroethylene exhibits a 20.8% reduction in complex modulus at 70 °C, attributed to the incomplete evaporation of trichloroethylene. The complex modulus of MA-Centrifuge decreases by 24.7% compared to that of MA-TCE-Mineral. After the centrifugation of the solution, the mineral powder content decreases, and the complex modulus of MA-Centrifuge approaches that of MA-Virgin.

In contrast to virgin asphalt, MA-Mixture exhibits a 20.7% increase in complex modulus after high-temperature mixing, while RAP displays a substantial complex modulus growth of 71.9% following long-term aging. This is due to the oxidation and hardening of the asphalt during service. The complex modulus of RAP-surface is twice that of RAP-inside. RAP-surface represents the sample extracted during the first two iterations, where the recovered sample is the outer part of the asphalt membrane, which has been in direct contact with air and experienced more severe aging. RAP-inside is the sample extracted during the last two iterations, where the recovered part is the inner part of the asphalt membrane, which has had less exposure to oxygen and experienced less aging. Incomplete extraction can lead to deviations in experimental results.

Table 4
Summary of TGA curves.

Sample	Loss mass ratio from 50 to100 °C (%)	Residual mass ratio at 700 °C (%)
MA-Virgin	0.05	17.21
MA-2.5 %Mineral	0.07	19.72
MA-5 %Mineral	0.04	21.16
MA-7.5 %Mineral	0.07	23.68
MA-10 %Mineral	0.06	25.1
MA-TCE	1.83	15.7
MA-TCE-Mineral	0.05	25.4
MA-Centrifuge	0.06	18.07
MA-Mixture	0.13	17.86
RAP	0.16	18.39
RAP-surface	0.03	17.81
RAP-inside	0.09	18.42

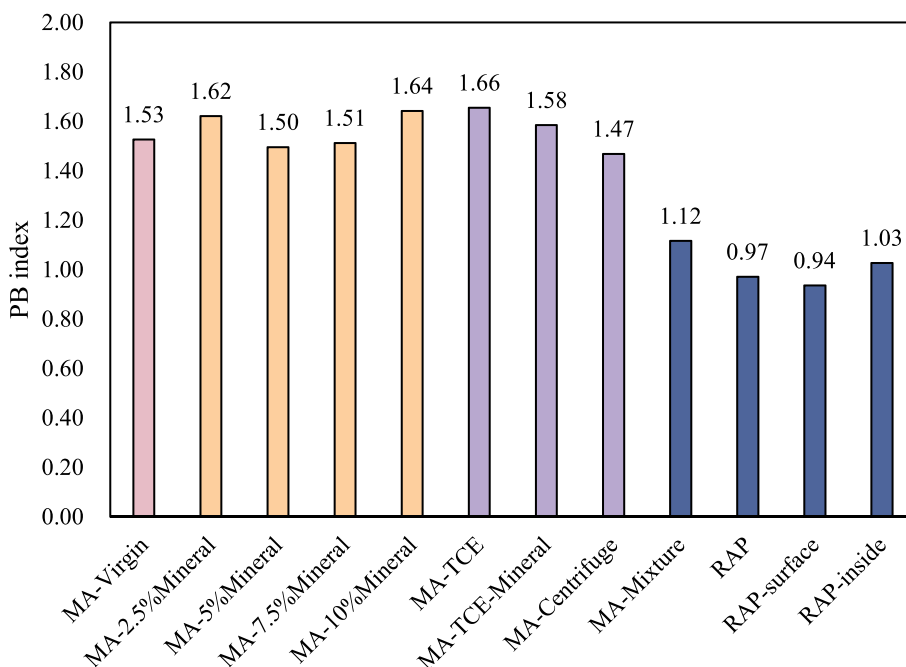


Fig. 6. Summary of PB index calculated from FTIR analysis.

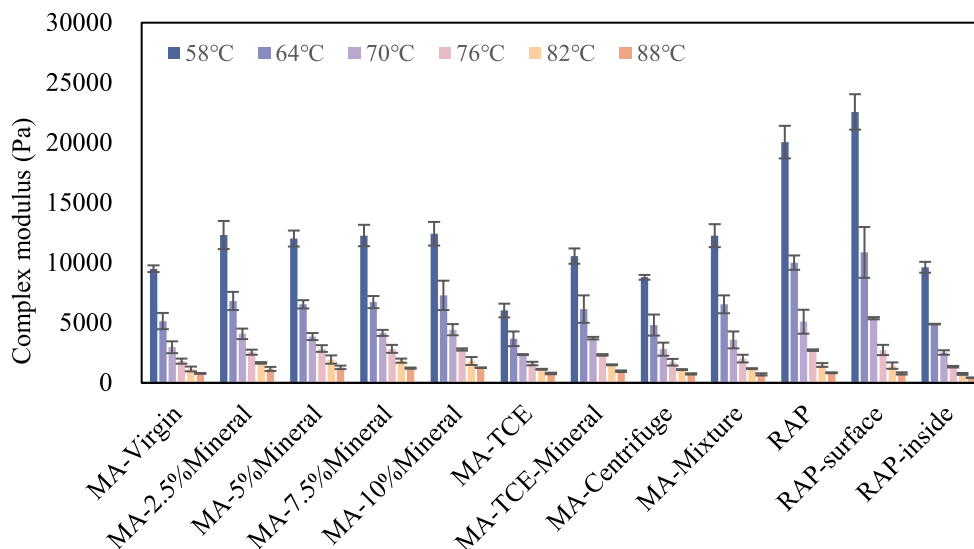


Fig. 7. Summary of modulus measurements (58 °C ~ 88 °C).

3.3.2. Phase angle result analysis

The phase angle characterization results of different samples is presented in Fig. 8. Mineral powder reduces the phase angle of asphalt, but there is no significant change in phase angle with an increase in the mineral powder ratio, which follows the same trend observed for complex modulus. The mineral powder enhances the binder’s elasticity. After centrifugation, the asphalt binder exhibits its original viscoelastic behavior. The presence of trichloroethylene increases the asphalt’s viscosity (e.g., increased phase angle).

The phase angle of asphalt significantly increases after long-term aging, indicating a decrease in the asphalt’s elasticity. SBS polymer degrades during aging, causing the asphalt binder to display increased viscosity behavior. The phase angle of RAP-surface is smaller than that of RAP-inside, indicating that the surface part of the asphalt membrane exhibits greater elasticity.

It is worth noting that, for the sample before aging, the phase angle decreases with increasing temperature. This is because the existence of the polymer network plays a dominant role in providing elasticity at high temperatures. Simultaneously, the stiffness of the bitumen phase is very low at high temperatures, so the polymer network dominates,

resulting in a decreased phase angle for SBS modified bitumen. However, for SBS modified bitumen after aging, the polymer network cannot provide elasticity at high temperatures, and the bitumen phase plays the dominant role, leading to an increased phase angle.

In summary, the complex modulus and phase angle analyses demonstrate that mineral powder content, trichloroethylene presence, and aging significantly impact asphalt’s rheological properties. Mineral powder increases the stiffness and elasticity of the binder, but its effects plateau with increasing filler proportions. Trichloroethylene, when not fully evaporated, reduces asphalt’s complex modulus and increases its viscosity. Aging leads to oxidation and hardening, decreasing the elasticity of asphalt and increasing its viscosity. These findings emphasize the importance of accurately controlling the extraction and recovery processes, as well as considering the influence of various factors during reclaimed asphalt pavement recycling. This will help ensure the efficient utilization of RAP and promote sustainable development in the road construction industry.

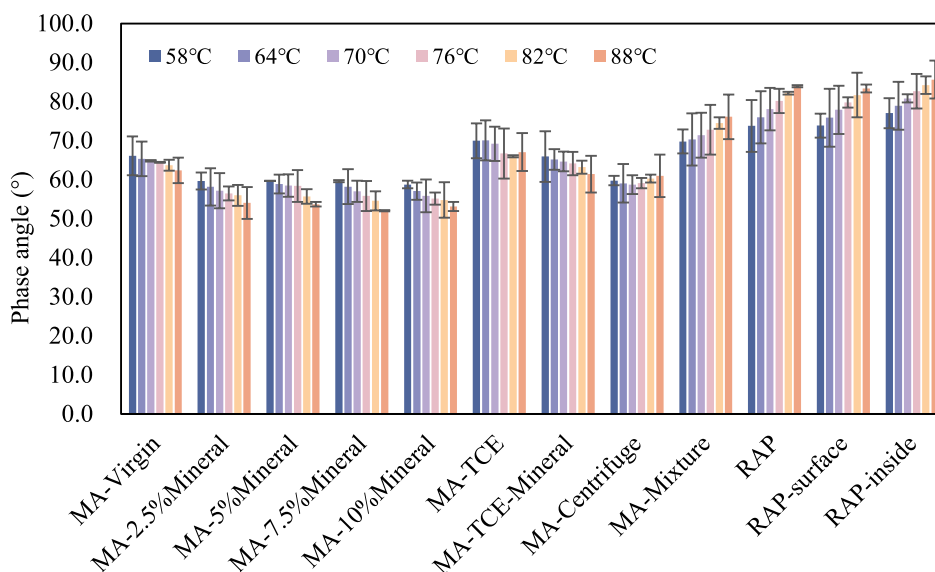


Fig. 8. Summary of phase angle measurements (58 °C ~ 88 °C).

3.4. MSCR test results analysis

3.4.1. Elastic recovery (R) results analysis

The result of R0.1 testing is presented in Fig. 9. The R0.1 testing results reflect the asphalt's elasticity. Under lower stress levels (0.1 kPa), the effects of mineral powder and trichloroethylene on asphalt's R0.1 are negligible. This is because the elastic recovery property of SBS modified bitumen binder primarily stems from the SBS polymer network, limiting the influence of minerals and trichloroethylene. After ten years of service, the R0.1 of asphalt significantly decreases due to reduced elasticity caused by SBS polymer degradation. Interestingly, the R0.1 of RAP-surface is higher than that of RAP-inside, indicating that oxidation of the bitumen phase also plays a crucial role in increasing elasticity. Consequently, the aging degree of the inside and surface parts of the asphalt membrane differs.

Fig. 10 displays the R3.2 testing results, which exhibit a trend similar to R0.1 but with significantly lower values. Under higher stress levels, more unrecoverable deformation occurs, leading to higher measured asphalt viscosity. As temperature rises, the R3.2 of MA-Virgin decreases, but the R3.2 of the sample with mineral powder remains relatively stable, suggesting that mineral powder can influence the temperature sensitivity of asphalt. Residual trichloroethylene increases asphalt viscosity, while mineral powder enhances its elasticity. These conclusions align with the phase angle results.

Regarding RAP, the differences in R3.2 under various extraction conditions are not pronounced. To facilitate the assessment of extraction accuracy, it is recommended to perform high-stress tests (R3.2) on asphalt with high elasticity, such as virgin asphalt. For asphalt with low elasticity, like RAP, lower stress tests (R0.1) are more suitable.

3.4.2. Non-recoverable creep compliance (Jnr) results analysis

The non-recoverable creep compliance (Jnr) results analysis for Jnr0.1 and Jnr3.2 tests are shown in Figs. 11 and 12, respectively. Under lower stress conditions, the Jnr0.1 results for virgin asphalt are very close, making it difficult to discern the influence of mineral powder and trichloroethylene. However, the Jnr3.2 of MA-TCE is higher than that of MA-Virgin, indicating that residual trichloroethylene impairs the asphalt's rutting resistance. Jnr3.2 is well-suited for determining whether trichloroethylene is entirely removed.

For RAP, Jnr0.1 and Jnr3.2 exhibit similar trends. The Jnr of RAP-surface is notably higher than that of RAP-inside. Therefore, a complete extraction process is essential for accurately evaluating aged asphalt performance.

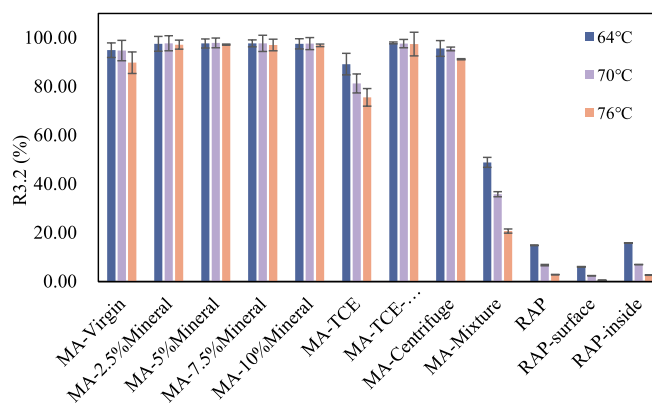


Fig. 10. Summary of R3.2 measurements from MSCR (64 °C, 70 °C and 76 °C).

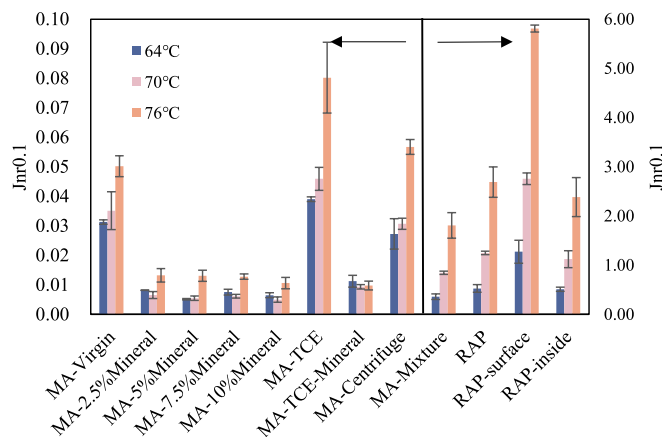


Fig. 11. Summary of Jnr0.1 measurements from MSCR (64 °C, 70 °C and 76 °C).

4. Conclusion

Centrifugal extraction and rotary evaporation are essential methods for obtaining and evaluating the binder in reclaimed asphalt pavement (RAP). This study aims to propose indicators for assessing the accuracy of extraction and recovery processes. Eleven distinct asphalt samples were prepared to simulate common scenarios encountered during

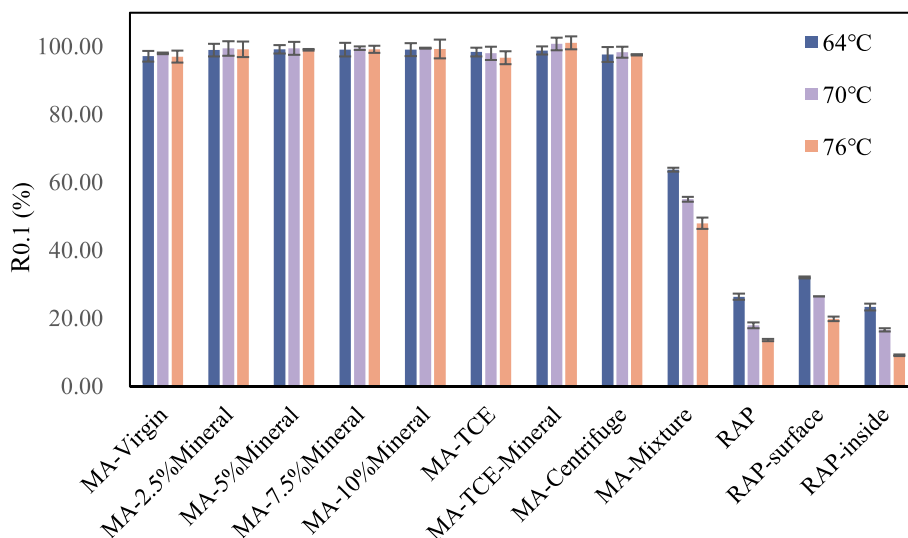


Fig. 9. Summary of R0.1 measurements from MSCR (64 °C, 70 °C and 76 °C).

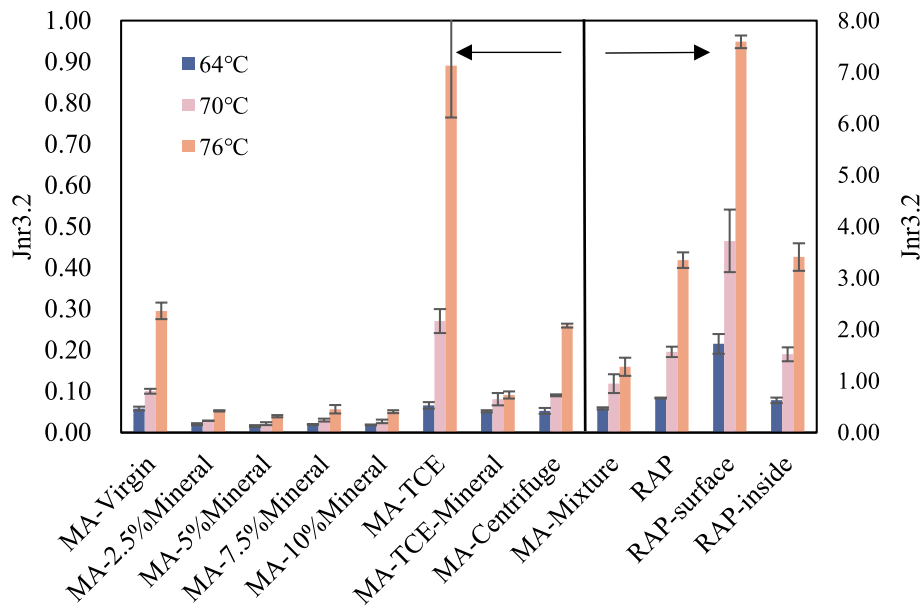


Fig. 12. Summary of $J_{nr3.2}$ measurements from MSCR (64 °C, 70 °C and 76 °C).

extraction and recovery. Additionally, virgin SBS-modified asphalt was tested to serve as a reference point. The conclusions drawn from the test results are as follows:

- Residual mineral powder, trichloroethylene, and insufficient extraction can affect the performance evaluation of recycled asphalt. This article provides some technical means to quickly determine the accuracy of recycled asphalt. The experimenter can adjust the experimental plan based on the discovered problems to improve the accuracy of extraction and recovery.
- Issues and causes: The accuracy of extraction recovery can be significantly affected by residual mineral powder, residual trichloroethylene and insufficient extraction. Residual mineral powder and trichloroethylene can change the physical and chemical properties of recycled asphalt. The inside part of the binder, which is less exposed to sunlight and oxidation, shows a significantly lower aging level compared to the surface part. Insufficient extraction can lead to overestimation of the aging degree of recycled asphalt in the evaluation process.
- Effective evaluation techniques: Employing FTIR (Fourier-transform infrared spectroscopy), TGA (thermogravimetric analysis), and rheological tests (oscillatory test, MSCR) allows for accurate evaluation of residual trichloroethylene and residual mineral powder in recycled asphalt. These techniques provide detailed information about material properties, such as viscoelasticity, aging, and phase transitions, facilitating better decision-making for material selection and processing.
- Suggestions: If any deviation is found in the extraction and recovery test using the above method, the test plan should be adjusted in time. High-speed centrifugation can effectively address residual mineral powder by separating it from the asphalt binder. Extending the rotary evaporation time can reduce the residue of trichloroethylene. Researchers and professionals should recycle all the asphalt in RAP as much as possible when assessing RAP performance and designing rehabilitation strategies.

5. Limitation and recommendation

Our research only focuses on one type of asphalt and the conclusions need to be further validation.

CRediT authorship contribution statement

Haobai Zhong: Data curation, Investigation. **Weidong Huang:** . **Peng Lin:** . **Lu Zhou:** . **Quan Lv:** Conceptualization, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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References

- [1] Y. AbuHassan, M. Alin, T. Iqbal, M. Nazzal, A.R. Abbas, Effect of extraction solvents on rheological properties of recovered asphalt binders, *J. Transp. Eng. Part B-Pavements* 145 (1) (2019), 04018064.
- [2] O.Y. Bayraktar, G.S. Citoglu, C.M. Belgin, M. Cetin, Investigation of the mechanical properties of marble dust and silica fume substituted portland cement samples under high temperature effect, *Fresen. Environ. Bull.* 28 (5) (2019) 3865–3875.
- [3] O.Y. Bayraktar, G.S. Citoglu, C.M. Belgin, S. Cetin, M. Cetin, Investigation of effect of brick dust and silica fume on the properties of portland cement mortar, *Fresen. Environ. Bull.* 28 (11) (2019) 7823–7832.
- [4] B.F. Bowers, B.S. Huang, Q. He, X. Shu, X.Y. Jia, B.C. Miller, Investigation of sequential dissolution of asphalt binder in common Solvents by FTIR and binder fractionation, *J. Mater. Civ. Eng.* 27 (8) (2015).
- [5] M. Cetin, Consideration of permeable pavement in landscape architecture, *J. Environ. Prot. Ecol.* 16 (1) (2015) 385–392.
- [6] Z. Chen, H. Zhang, C. Zhu, B. Zhao, Rheological examination of aging in bitumen with inorganic nanoparticles and organic expanded vermiculite, *Constr. Build. Mater.* 101 (2015) 884–891.
- [7] Z.-G. Feng, H.-J. Bian, X.-J. Li, J.-Y. Yu, FTIR analysis of UV aging on bitumen and its fractions, *Mater. Struct.* 49 (4) (2016) 1381–1389.
- [8] A. Forton, S. Mangiafico, C. Sauzéat, H. Di Benedetto, P. Marc, Properties of blends of fresh and RAP binders with rejuvenator: experimental and estimated results, *Constr. Build. Mater.* 236 (2020), 117555.

- [9] J. Gao, J. Yang, D. Yu, Y. Jiang, L. Luo, Reducing the variability of multi-source reclaimed asphalt pavement materials: A practice in China, *Constr. Build. Mater.* 278 (9) (2021), 122389.
- [10] D.D. Ge, Z.P. You, S.Y. Chen, C.C. Liu, J.F. Gao, S.T. Lv, The performance of asphalt binder with trichloroethylene: Improving the efficiency of using reclaimed asphalt pavement, *J. Clean. Prod.* 232 (2019) 205–212.
- [11] Geng, J., J. Dai and J. Yuan (2010). "Analysis of Sedimentation Theory and Elimination Method of Residual Filler in Extraction of Asphalt Cement." *Journal of Highway and Transportation Research and Development*.
- [12] J. Geng, J. Dai, J.A. Yuan, Analysis of sedimentation theory and elimination method of residual filler in extraction of asphalt cement, *J. Highway Transp. Res. Dev.* 27 (9) (2010) 23.
- [13] X.D. Hou, F.P. Xiao, J.G. Wang, S. Amirkhanian, Identification of asphalt aging characterization by spectrophotometry technique, *Fuel* 226 (2018) 230–239.
- [14] J. Li, F. Xiao, L. Zhang, S.N. Amirkhanian, Life cycle assessment and life cycle cost analysis of recycled solid waste materials in highway pavement: A review, *J. Clean. Prod.* 233 (OCT.1) (2019) 1182–1206.
- [15] P. Lin, C.Q. Yan, W.D. Huang, Y. Li, L. Zhou, N.P. Tang, F.P. Xiao, Y. Zhang, Q. Lv, Rheological, chemical and aging characteristics of high content polymer modified asphalt, *Constr. Build. Mater.* 207 (2019) 616–629.
- [16] L. Liu, H. Cheng, L. Sun, L. Xu, Quantifying physical and rheological properties of trichloroethylene-asphalt system to improve performance evaluation of recycled asphalt, *J. Clean. Prod.* 367 (2022), 133018.
- [17] McDaniel, R. S. and A. Shah (2003). "Use of Reclaimed Asphalt Pavement (RAP) Under Superpave Specifications: A Regional Pooled Fund Project." *Asphalt Paving Technology: Association of Asphalt Paving Technologists-Proceedings of the Technical Sessions* 72.
- [18] Mehmet, C. (2013). *Landscape Engineering, Protecting Soil, and Runoff Storm Water. Advances in Landscape Architecture*. Ö. Murat. Rijeka, IntechOpen: Ch. 27.
- [19] N. Pierard, S. Vansteenkiste, A. Vanelstraete, Effect of extraction and recovery procedure on the determination of PmB content and on the properties of the recovered binder, *Road Mater. Pavement Des.* 11 (2010) 251–279.
- [20] X. Qian, X. Hou, Z. Zhao, J. Prozzi, F. Xiao, Comparative evaluation of methods for removing residual mineral fillers during bitumen extraction and recovery based on FTIR, *J. Mater. Civ. Eng.* 32 (12) (2020).
- [21] S. Ren, X. Liu, H. Wang, W. Fan, S. Erkens, Evaluation of rheological behaviors and anti-aging properties of recycled asphalts using low-viscosity asphalt and polymers, *J. Clean. Prod.* 253 (2020) 120048.
- [22] D. Sun, Y. Song, F. Chen, The influence of extractant composition on the asphaltenes extracted from asphalt, *Coatings* 12 (10) (2022) 1600.
- [23] X. Xie, S. Tong, Y. Ding, H. Liu, L. Liang, Effect of the amount of mineral powder on the ultraviolet aging properties of asphalt, *Adv. Mater. Sci. Eng.* 2016 (2016) 5207391.
- [24] C.Q. Yan, W.D. Huang, P. Lin, Y. Zhang, Q. Lv, Chemical and rheological evaluation of aging properties of high content SBS polymer modified asphalt, *Fuel* 252 (2019) 417–426.
- [25] J. Yu-hong, M.A. Tao, S. Peng-cheng, Study on extraction and recovery experiments of stone matrix asphalt, *J. Hefei Univ. Technol. Nat. Sci.* 32 (2) (2009) 252–255.
- [26] M. Zaumanis, R.B. Mallick, R. Frank, 100% recycled hot mix asphalt: A review and analysis, *Resour. Conserv. Recycl.* 92 (2014) 230–245.
- [27] Y. Zhang, S. Han, Z. Shao, Test and improvement of recovering asphalt with rotary evaporator method, *J. Dalian Univ. Tech.* 60 (2) (2020) 192–199.
- [28] H. Zhong, W. Huang, C. Yan, Y. Zhang, L. Liu, Investigating binder aging during hot in-place recycling (HIR) of asphalt pavement, *Constr. Build. Mater.* 276 (1) (2021), 122188.
- [29] F. Yurong, H. Yong, Research on Macro Road Maintenance Standard of Asphalt Pavement by Comprehensive Method, *J. of Muni.Tech.* 40 (12) (2022) 14–18, <https://doi.org/10.19922/j.1009-7767.2022.12.014>.
- [30] D. Lingling, Analysis of Mechanical Properties of Recycled Asphalt Pavement based on ABAQUS Model, *J. of Muni.Tech.* 40 (09) (2022) 85–91+121, <https://doi.org/10.19922/j.1009-7767.2022.09.085>.
- [31] Y. Xudong, Discussion on Maintenance Schemes of Carbon Reduction of Asphalt Pavements, *J. of Muni.Tech.* 40 (02) (2022) 17–19, <https://doi.org/10.19922/j.1009-7767.2022.02.017>.