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Investigation on Adhesion Characteristics of Virgin-Aged Composite Binder and Binder-Aggregate System

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Abstract: In recycled asphalt mixtures, the RAP binder often becomes stiff and brittle due to aging, making it difficult to achieve a complete integration with the added virgin binder, which could provide a negative impact on the long-term performance of the recycled asphalt mixtures. In an attempt to identify the influence of the integration degree on the adhesion characteristics of virgin-aged composite binder and the corresponding binder-aggregate system, a series of experiments, including the modified boiling test and pull-off test and corresponding formation methods for the binder samples with various interfusion states, were designed and carried out in the present study. The two-layered composite binder (CB), consisting of a layer of virgin binder and a layer of aged binder was fabricated for the experiments, and the testing results were analyzed and compared with those of the virgin binder (VB), the aged binder (AB), and the blended binder (BB). The thermostatic treatments with various durations were subjected to the binder samples to attribute them with various integrations, and the wetting conditioning was also imposed on them to introduce the impact of moisture erosions. The rheological properties, creep recovery capacity, and fatigue resistance of the binders were first investigated based on a dynamic shear rheometer. The adhesion characteristics and failure behaviors of the binder-aggregate system were furthermore explored with the modified boiling test and pull-off test, and the percentage of residual binder (*%_residual binder*), pull-off test strength (*POTS*), and moisture damage index (*MDI*) were adopted to assess or quantify their adhesion characteristics and moisture susceptibility. The results found that the rheological properties and creep recovery capacity of the CB were similar to those of the VB, and its fatigue life exhibited an increasing trend with the extension of the thermostatic-treatment duration. In contrast to the modified boiling test, the pull-off test was more effective at evaluating the integration and distinguishing the failure behaviors of the binder-aggregate system. As the thermostatic-treatment duration prolonged, the interfusion between the virgin and the aged binder was facilitated, the cohesion within the binder and the adhesion between the binder and the aggregate was also enhanced, and thus the adhesion performance and moisture-damage resistance of the system were improved.

Keywords: virgin-aged composite binder; adhesion characteristics; moisture susceptibility; modified boiling method; pull-off test

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1. Introduction

The usage of recycled asphalt mixtures could consume a large amount of reclaimed asphalt pavement (RAP) material, which has the major advantages of saving resources, protecting the environment, reducing costs, and improving economic efficiency [1–4]. However, the presence of RAP may lead to large uncertainties and deviations in the performance of recycled asphalt mixtures. The influence of the RAP binder on the performance of the final designed recycled mixtures is not yet clear due to multiple factors such as the integration degree between the aged binder and virgin binder, the use of the rejuvenator, its compatibility with the aged binder, the mixing temperature and mixing time, and so on [4–6]. Therefore, a more profound study related to the effect of RAP (especially the aged binder in the RAP) on the long-term performances of recycled mixtures are of great significance.

Moisture susceptibility is one of the crucial issues with great concern for recycled asphalt mixtures. In general, moisture could deteriorate the adhesion between the asphalt binder and aggregates, and the cohesion of the asphalt binder could be negatively affected by the erosion of water especially if the binder has already suffered the aging process, which consequently leads to the functional degradation of the asphalt mixture of the pavement, showing loosening, stripping, cracking, and raveling [6–8]. Many studies have found that the addition of RAP could increase the water sensitivity of recycled mixtures [9]. The aged binder in RAP with high stiffness exhibits a relatively high cracking potential, which made it easier for water to penetrate into the mixtures containing RAP and deteriorate the cohesion of the asphalt binder and its adhesion to aggregates [10–12]. However, some research has suggested that the moisture-damage resistance of mixtures could be enhanced to some extent with the incorporation of RAP due to the increased adhesive strength of the aged binder [13,14]. For example, Fakhri et al. [14] showed that the usage of RAP and nano-clay had a beneficial effect on both crack and moisture stability of warm mix asphalt (WMA) mixtures. By using the surface free energy method, Ghabchi et al. [15] found that RAP could increase the acidic component of the regenerated binder, and the moisture-damage resistance between the regenerated binder and various types of aggregates was improved as the content of RAP increased. In general, the conclusion is remains controversial with regards to the influence of RAP on the moisture stability of recycled asphalt mixtures. Therefore, it would be essential to further understand the performance of regenerated binders containing a RAP binder (aged binder) in the presence of moisture, and quantify the impact of interfusion quality of virgin and aged asphalt binders on the moisture susceptibility of recycled mixtures.

Caro [16,17] provided a comprehensive analysis of the definition, identification, and research methods of moisture damage, and a detailed summary of the mechanisms, characterization, and modeling of moisture damage was also proposed in the study. It was reported that moisture damage generally occurs in two steps: firstly, the moisture in liquid or vapor state penetrates into the asphalt mixture and reaches the interface of binder-aggregate; then, the internal structure of the mixture changes due to the intrusion and erosion of the moisture, resulting in an insufficient adhesion and cohesion for the system. As well known, the cohesion of asphalt binder and the adhesion of asphalt binder to aggregates are the crucial factors determining the moisture-damage resistance of the asphalt mixture. Therefore, the evaluation of the cohesion and adhesion characteristics of the regenerated binder could be used to evaluate the magnitude of moisture-damage resistance for the mixture.

For recycled asphalt mixtures, a destruction of the RAP aggregate and hardening of the RAP binder could lead to inadequate adhesion properties of the binder-aggregate system. Currently, most of the experimental studies in this regard have been conducted by mixing the aged and virgin binders (or rejuvenator, if needed) thoroughly and assuming that a complete integration has been researched between them [15,18]. Obviously, this is consistent with the actual situation in the plant. In recycled mixtures, the aged binder and virgin binder coated on aggregates is hard to reach a fully interfusion state, even if they have been mixed with sufficient time. This could produce a non-negligible effect on the adhesion and bonding between the asphalt binder and aggregates in the recycled mixtures, which, in turn, affects the long-term performance of the recycled mixtures, especially the moisture susceptibility. Many studies have been presented on the interfusion efficiency and integration degree of the aged and virgin binders, but the adhesion properties and moisture-damage resistance of the regenerated binders considering the effect of integration degree between the aged and virgin binders need to be further explored.

There are many methods for the evaluation of the adhesion characteristics of asphalt binder to aggregates. The boiling method and water-immersion method are the most widely employed ones. The boiling method involves the immersion of asphalt-coated aggregate particles in slightly boiling water for 3 min and the percentage of the stripping area for the asphalt film wrapped on the aggregate was observed for the evaluation. This method is simple and practicable but features a lot of limitations. For instance, it is difficult to control the boiling condition accurately as the water has to be maintained at a slight boiling condition and no boiling bubbles is allowed during the process. In addition, the estimation based on artificial recognition is highly subjective and the quantitative results also lack precision. Accordingly, some researchers have suggested that the modified boiling method is more applicable be used instead of the boiling method [4,19], in which the water was maintained at a full boiling condition, and the impact of water on the binder-aggregate system was intensified. The duration for the asphalt film is all stripped or the mass loss of the asphalt film after boiling for a certain time is adopted to evaluate the adhesion properties between the asphalt binder and aggregate.

The pull-off test method is also widely used for the investigation of adhesion performance of asphalt binder to aggregates. Moreover, based on the principle of pull-off test, the binder bond strength (BBS) test specified in the AASHTO TP 91 [20] is used to evaluate the bond strength between asphalt binder and aggregates. Many studies have been reported using the pull-off test and shearing test to analyze the adhesion characteristics of asphalt-aggregate systems [21–23]. Compared to the other methods, Gan [24] pointed out that the pull-off test method could visually reflect the true adhesion strength with the advantages of simple and easy operations. Other studies [25,26] have also shown that the pull-off test is easy to observe and it could be used to determine the failure state of the asphalt-aggregate system by recording the magnitude of the bond strength. Considering the effect of aged asphalt and aggregates in RAP, Yan et al. [27] used the coating treatment to simulate the RAP aggregate state and evaluated the adhesion of fresh asphalt binder to RAP aggregate by BBS test. It was found that there was always cohesive damage in the diffusion zone and a significant reduction in bonding strength after water immersion treatment. The same method has been used by Cardone et al. [28] to prepare the artificial RAP aggregate substrate and tested its adhesion to the emulsified asphalt and asphalt mastic. The results showed that the adhesion failures were observed dominantly with or without the water-immersion treatment when asphalt mastic was used. But when the pure emulsified asphalt binder was used, the failure state was changed with the water immersion treatment. In addition, the bond strength may be reduced or increased after the water immersion treatment. Similar studies have been carried out by other researchers [29], but the effect of the integration degree of the virgin binder and the RAP binder on the adhesion characteristics was not discussed.

It is clear that, according to the previous researches discussed above, the integration states between the virgin binder and the aged binder exhibit significant influence on the performances of the composite binder and the adhesion characteristics of binder-aggregate system. An experimental study based on specially designed sample preparation methods considering various treatment conditions for the samples were performed, herein, to investigate the adhesion characteristics of the virgin-aged composite binders and the binder-aggregate system.

2. Objective and Scope

The main objective of this study was to investigate the effect of the interfusion degree between the virgin binder and aged binder on adhesion characteristics to aggregates. To achieve this goal, two-layered composite samples made with a layer of virgin binder and a layer of aged binder were fabricated and treated under a thermostatic condition with various durations to obtain different integration degrees between the composite binders. The dynamic shear rheometer (DSR), multiple stress creep recovery (MSCR), and linear amplitude sweep (LAS) tests were performed to understand the fundamental properties and mechanical performances of the binders, and the modified boiling tests and pull-off tests were conducted to investigate the adhesion characteristics of the binder-aggregate system.

3. Materials and Sample Preparation

3.1. Materials

The experimental materials used in the study include the aged binder (RAP binder), the RAP aggregate, the virgin binder, the virgin aggregate (new aggregate), and rock (the substrate for pull-off test). The RAP was sampled through a milling process from the surface layer of a highway in Hunan, China, and the aged binder was extracted from the RAP mixture through the centrifuge extraction and rotary evaporation methods (Figure 1). The DLC-5 Asphalt Extractor was produced by Meiyu Instrument Technology Co., LTD, Shanghai, China, and the RV-211A Evaporator was produced by Yihen Yiqi Co., LTD, Shanghai, China The virgin binder is a commonly used SBS modified asphalt binder provided by Hunan Baoli Asphalt Co., LTD, Changsha, China. The fundamental properties of the binders are presented in Table 1.

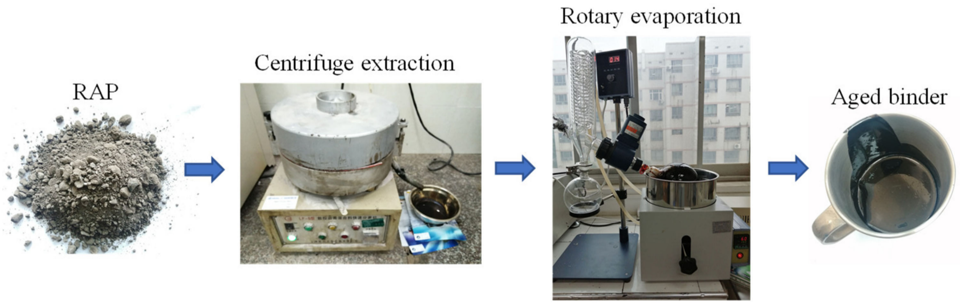


Figure 1. Extraction process of aged binder from RAP.

Table 1. Fundamental properties of asphalt binders.

Properties	Unit	Virgin Binder	Aged Binder
Penetration (25 °C, 100 g, 5 s)	0.01 mm	55.6	23.5
Ductility (5 cm/min, 5 °C)	cm	30.2	5
Softening point	°C	78.9	86.3
Kinematic viscosity (135 °C)	Pa·s	1.98	-
Elastic recovery (25 °C, 10)	%	95	-
Residue after RTFOT			
Mass loss	%	0.024	-
Penetration ratio (25 °C)	%	75	-
Ductility (5 cm/min, 5 °C)	cm	17.2	-

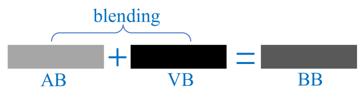
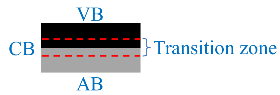
The virgin aggregate and RAP aggregate used for the modified boiling test and the rock substrate used for the pull-off test are all basalt, as shown in Figure 2.

Figure 2. Aggregates and rock substrate for testing. (**a**) aggregate; (**b**) rock substrate.

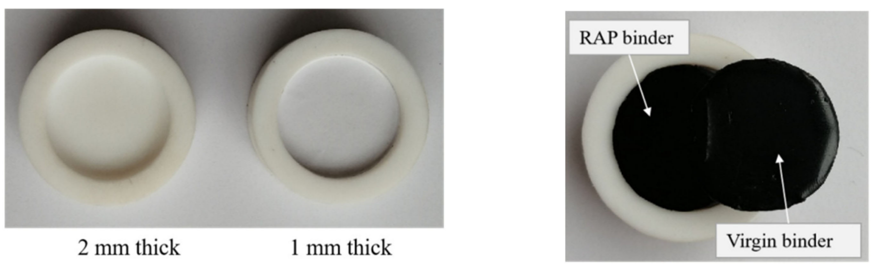
3.2. Sample Preparation and Pretreatment

There are four formations of binders used for the study: the virgin binder (VB), the aged binder (AB), the blended binder (BB), and the composite binder (CB), shown in Table 2. The virgin binder and the aged binder are the new SBS binder and the old binder extracted from the RAP, respectively. The blended binder was obtained by mixing the virgin binder and the aged binder thoroughly with a mass ratio of 1:1 at a high temperature, which could be considered as a uniform mixture that is often referred to as rejuvenated binder. The composite binder is a two-layered combination made with a layer of virgin binder and a layer of aged binder, which was used in the study as a simulation for considering the various integration degrees between the virgin binder and aged binder.

Table 2. Various formations of binders considered in the study.

Binder Type	Formation	Schematic Description
Virgin binder (VB)	New SBS asphalt	
Aged binder (AB)	Extracted from RAP	
Blended binder (BB)	A mixture of virgin and aged binders	
Composite binder (CB)	A two-layered combination of virgin binder and aged binder	

For the fabrication of the composite binder sample, the virgin and aged binders were first separately poured into a cylindrical rubber mold with 25 mm in diameter and 1 mm in thickness. After the binders were hardened, the virgin binder sample was stacked on top of the aged binder sample and placed in a rubber mold with 2 mm in thickness (Figure 3) to form the composite sample.



(**a**) (**b**)

Figure 3. Preparation of virgin-aged composite sample. (**a**) rubber molds; (**b**) composite sample.

The thermostatic treatment was performed by preheating the samples in an oven for a certain duration, to attribute the composite binder (CB) with various integration degrees. After the treatment, the composite sample with different degrees of integration in the interfusion zone was achieved. In order to accelerate the interfusion between the virgin binder and the aged binder and, meanwhile, prevent the binders from aging, 120 °C was selected for the thermostatic treatment, and the duration was set to 0, 20, 40, 60, 90, 120, and 150 min for the samples.

Drying and wetting conditioning were also carried out on the samples used for the modified boiling and pull-off tests to reveal the impact of moisture on the adhesion properties of the binder-aggregate system. For the drying conditioning, the samples were placed in a dry environment around 20 °C for 24 h, and an immersion of samples in a water bath at 40 °C for 24 h was followed for the samples with wetting conditioning.

4. Methodology and Experiments

4.1. Tests for Fundamental Properties

Temperature-frequency sweep tests were conducted using the dynamic shear rheometer (DSR, SmartPave 102, manufactured by Anton-Paar Inc., Ashland, VI, USA) to investigate the rheological characteristic of the binders. To ensure that the binder samples were tested within the liner viscoelastic (LVE) limits, a strain level of 1% was maintained for the tests. The test temperature was designed from 10 to 70 °C with an interval of 10 °C, and the sweep frequency was 0–30 Hz. The multiple stress creep recovery (MSCR) tests and the linear amplitude sweep (LAS) tests were also performed based on the DSR to explore the creep recovery property and fatigue resistance of the binders at 64 °C and 25 °C, respectively.

4.2. Modified Boiling Test

Based on the conventional boiling method, a modified boiling test was employed for the evaluation of the adhesive performance of the asphalt-aggregate system. During the test, the sample was boiled in the water at a full boiling condition in 2 h, and in this condition, the intrusion of water on the interface between asphalt film and aggregates was enhanced and the stripping in the sample is more prone to occur. In order to reduce the influence of deviation in the thickness of the asphalt film on the testing results, the total asphalt content was controlled as 5.5% to provide full and uniform covers on aggregates, and the gradation of aggregates was maintained identical for all mixtures. The treatment effects were considered after the preparation of samples to evaluate the time dependency of the quality of binder coating. The samples before and after the boiling tests are shown in Figure 4.

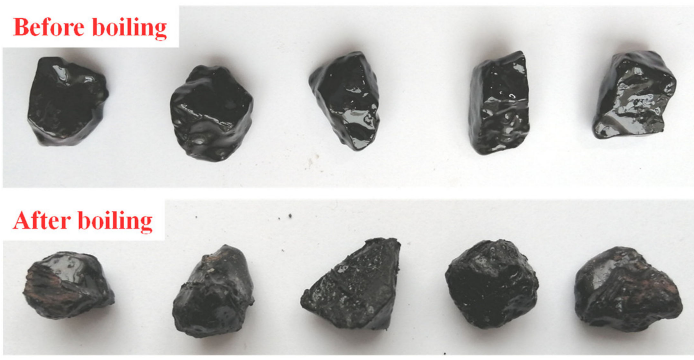


Figure 4. Samples before and after boiling test.

The percentage of residual binder on the aggregate was used to quantify the adhesion performance of the binder-aggregate system, as in Equation (1), and a greater *%_residual binder* usually represents a better adhesion of the binder to the aggregate.

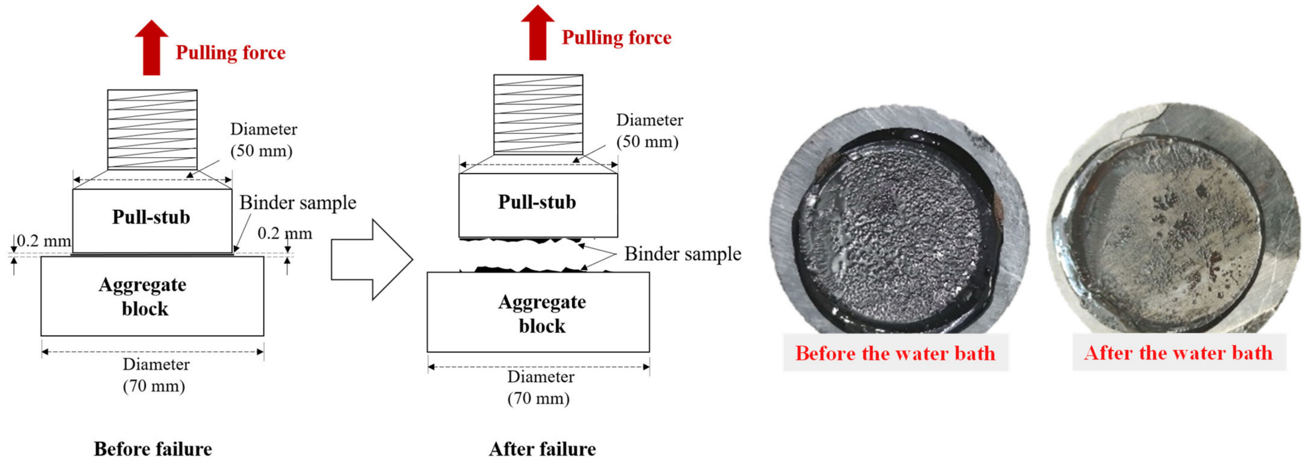
$$\%\_residual\ binder = \frac{m_2 - m_0}{m_1 - m_0} \times 100\% \quad (1)$$

where, *%_residual binder* is the percentage of residual binder on the sample, %; m_0 is the mass of the aggregate; m_1 is the mass of the sample before boiling; m_2 is the mass of the sample after boiling.

4.3. Pull-Off Test

The adhesion characteristics between asphalt binders and aggregates were evaluated with the bonding strength and failure states from the pull-off tests. By comparing the changes in bonding strength and failure state before and after water immersion treatment, the moisture susceptibility of the recycled asphalt mixture could be analyzed. In this study, the CH-LB-02 intelligent pavement interlayer pull-off tester (manufactured by, Hunan

Chenhao Technology Co., LTD, Hunan Province in China) was used to conduct the test. The setups for the pull-off test are presented in Figure 5.



(**a**) (**b**)

Figure 5. Setups for the pull-off test. (**a**) Schematic diagram of pull-off test; (**b**) Failure interface before and after wetting conditioning.

Figure 6 demonstrates the setups of the pull-off test. The cylindrical sample with a height of 25 mm and a diameter of 70 mm was cored and cut from a rock used as the substrate, and the surface of samples was roughened with sandpapers to enhance the adhesion. The aged binder, virgin binder, and blended binder were heated and molded into thin slices with 50 mm in diameter, while the aggregate substrate and pull-stub were heated in an oven at 170 °C for 1 h. The aged binder and virgin binder were placed on the aggregate substrate and pull-stub, respectively. A copper coil with 0.2 mm in diameter was placed on top of the substrate to ensure the thickness of the binder sample was controlled as 0.2 mm. The pull-stub was then pressed onto the substrate, and at that time the aggregate block and pull-stub were both at a sufficiently high temperature to soften the binder, and the excess binder was squeezed out by applying sustained pressure. Hence, a virgin-aged composite binder-aggregate sample with a film thickness of 0.2 mm was produced for the pull-off test, as shown in Figure 6c.

Prior to testing, the thermostatic treatment and wet conditioning were subjected to the binder samples by placing them in an oven at 120 °C for a certain duration and immersing them in the water with 40 °C in 24 h, as specified previously. After the pretreatments on the samples, the aggregate substrate was glued to a stable base and the samples and pull-stub were then mounted on the pulling device, as shown in Figure 6e. After initiating the test, the device applied a loading rate of 10 kPa/s for the test and recorded the data automatically. The testing parameters and corresponding treatment and conditioning methods for the binders are summarized in Table 3.

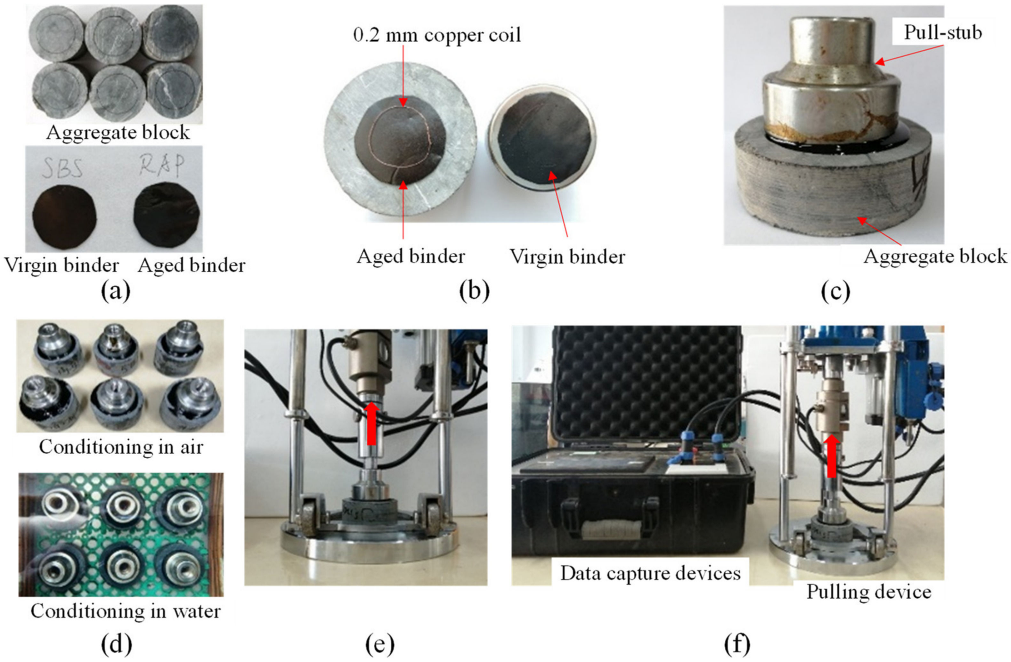


Figure 6. Main procedures for the pull-off test. (**a**) Samples of binder and aggregate; (**b**) Placement of binder sample; (**c**) The sample of binder-aggregate system; (**d**) Sample conditioning; (**e**) Sample installation; (**f**) The testing process.

Table 3. Test parameters and pretreatment methods for the samples.

Test	Objective	Thermostatic Treatment		Conditioning		Test Setting
		Temperature	Duration	Dry	Wet	
DSR	Rheological characteristics	120 °C	0, 20, 40, 60, 90, 120, 150 min	Not applicable	Not applicable	10–70 °C, 0–30 Hz
MSCR	Creep resilience					64 °C
LAS	Fatigue resistance					25 °C
Modified boiling test	Moisture susceptibility					Full boiling
Pull-off test	Adhesion performance			24 h in air at 20 °C	24 h in water at 40 °C	20 °C

The pull-off test strength (*POTS*), which is the ultimate tensile strength obtained from the test, was used to characterize the bond strength between asphalt binder and aggregate. In conjunction with *POTS* and the type of interfacial damages (failure states), the effect of the thermostatic treatment duration on the adhesion performance of the virgin-aged composite binder-aggregate system could be derived. The residual strength ratio after water-immersion conditioning was defined as the moisture damage index (*MDI*) to characterize the water damage resistance of asphalt-aggregate system [30]. The *POTS* and *MDI* were calculated according to the following equations.

$$POTS = \frac{F}{\frac{\pi}{4}d^2} \tag{2}$$

$$MDI = \frac{POTS_{wet}}{POTS_{dry}} \times 100 \tag{3}$$

where, *POTS* is the ultimate tensile strength, MPa; *F* is the maximum tensile load, N; *d* is the diameter of the pull-stub, *d* = 50 mm; *MDI* is the moisture damage index, %; $POTS_{dry}$ and $POTS_{wet}$ are the ultimate tensile strength before and after water immersion conditioning, MPa.

5. Results and Discussions

5.1. Fundamental Performances of the Binders

5.1.1. Rheological Properties

The master curve of the modulus (phase angle) of the binder, which describes the rheological properties of the asphalt material, can be obtained by the Time-Temperature Superposition (TTS) principle. In a complex shear modulus (phase angle)-frequency diagram, a selected test temperature was taken as the reference temperature, and the curves measured at other temperatures were shifted towards the curve at reference temperature to construct the viscoelastic master curve over a wider temperature range or a wider frequency range. The WLF empirical formula was used to determine the shift factor of the curve for each temperature, as in Equation (4).

$$lg\alpha_T = \frac{-C_1(T - T_r)}{C_2 + (T - T_r)} \quad (4)$$

where, T and T_r is test temperature and reference temperature, respectively, °C; C_1 and C_2 are constants related to material properties and can be derived by fitting to experimental data.

In this study, 40 °C was selected as the reference temperature to build the master curve. As illustrated in Figure 7, the virgin-aged composite binder (CB) was taken as an example, when the shear modulus $|G^*|$ was given a certain value, for each curve the corresponding frequency value could be obtained according to the curve variation law, and thus the shift factor at the reference temperature (40 °C) could be calculated. The shift factors for each curve from 10 °C to 70 °C were 2.414, 1.451, 0.661, 0, −0.785, −1.479 and −2.070. The results were fitted by WLF formula, as shown in Figure 8, which reveals that the shift factors were well correlated with the temperature, and the constants C_1 and C_2 were 37.2 and 503.3, respectively. Similarly, shift factors and master curves for other binder samples could be obtained.

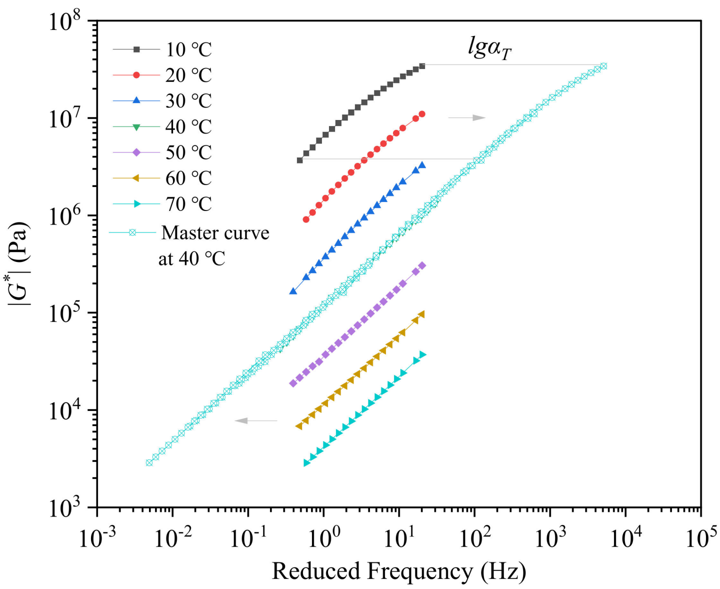


Figure 7. Shear modulus vs. frequency.

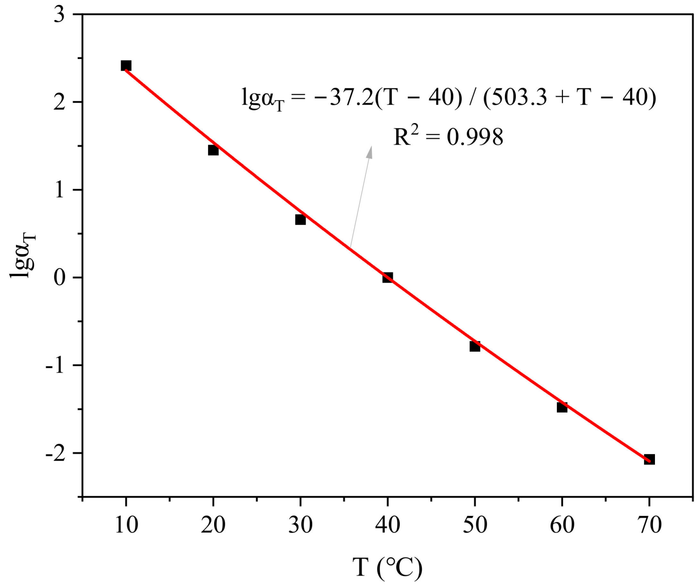


Figure 8. Shift factor-temperature.

The master curve of shear modulus and the phase angle of binders are shown in Figure 9. The modulus of binders gradually increased with the increasing frequency. At the same frequency, the modulus of AB was the highest, followed by the BB, and the VB, while the moduli of CB with various treatment durations all fell in between the VB and BB. In the low frequency region, more noticeable difference could be observed between the moduli of the binders, while the difference became smaller in the high frequency region. Theoretically, when the frequency increases to a certain level, the modulus would not keep increasing with the frequency, but tend to reach a constant value, which is the glassy modulus. It could be inferred from the results that the glassy modulus of AB was smaller than that of other binders, while the glassy modulus of VB, BB and CB were relatively close to each other. The differences between the results of CB at various thermostatic durations were insignificant, and ranged between the VB and the BB. The phase angle of AB and BB decreased gradually with the increasing frequency. Parabolic curves, which first increase, then decrease, were observed for the VB and CB varying with the increased frequency, and the phase angles of CB were basically lower than that of VB. The master curve of the phase angle of CB did not show a significant pattern with the various thermostatic treatment durations, which is similar to the results of the modulus.

Apparently, from the results of DSR, the effect of extending the thermostatic-treatment duration on the rheological properties of the virgin-aged composite binder was insignificant at 120 °C for the range from 0 min to 150 min. This was possibly due to the fact that the thickness of the virgin binder and the aged binder film (both 1 mm) in the laboratory experiments was too thick compared to the actual practice (lower than 10 μm), and although the interfusion between the virgin binder and the aged binder has been generated, the degree of integration was relatively low, and the aged binder in the lower layer behaved like the "fixed bottom plate" due to its relatively high stiffness. In this case, due to the limitations of the test method, the rheological properties of the virgin-aged composite binder were more dominated by the virgin binder.

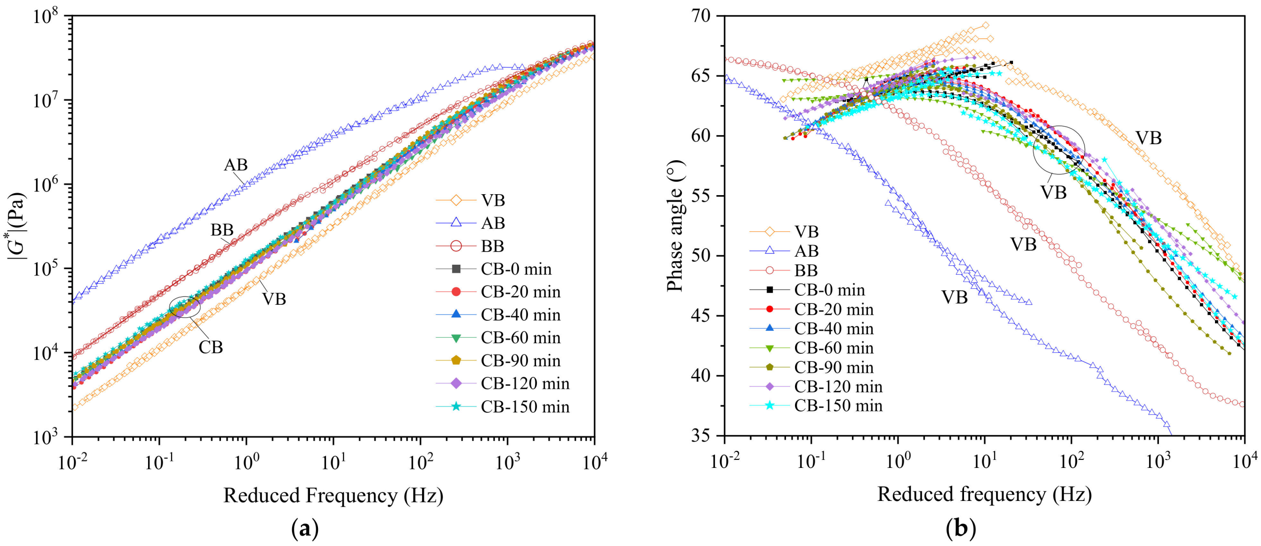


Figure 9. Shear modulus and phase angle of various binders. (**a**) Master curve of shear modulus; (**b**) Master curve of phase angle.

5.1.2. Creep Recovery Capacity

As illustrated in Figure 10, the percent recovery, *R*, and non-recoverable creep compliance, *Jnr*, of the binders were obtained from the MSCR tests. The *R3.2* represents the percent of elastic recovery at 3.2 kPa calculated by the ratio of recoverable strain to unrecoverable strain, and it reflects the recovering capacity of the binders after being subjected to loads or stresses. The *Jnr3.2* is the non-recoverable creep compliance at 3.2 kPa calculated by unrecoverable strain and applied stress, and the lower the *Jnr3.2*, the stiffer and the better rutting resistance of the binder. With the extension of the duration of thermostatic treatment, the creep recovery capacity of the CB gradually decreased, but the reduction was insignificant. The creep recovery capacity of CB was overall similar to that of VB, but much higher than that of the AB and BB. The creep recovery capacity of CB under various thermostatic treatment durations was about 4.5 times as that of AB and about 2.3 times as that of BB. The non-recoverable creep compliance of CB fluctuated between 0.07 and 0.11 kPa^{-1}, which was slightly less than that of VB, but much lower than that of the AB and BB.

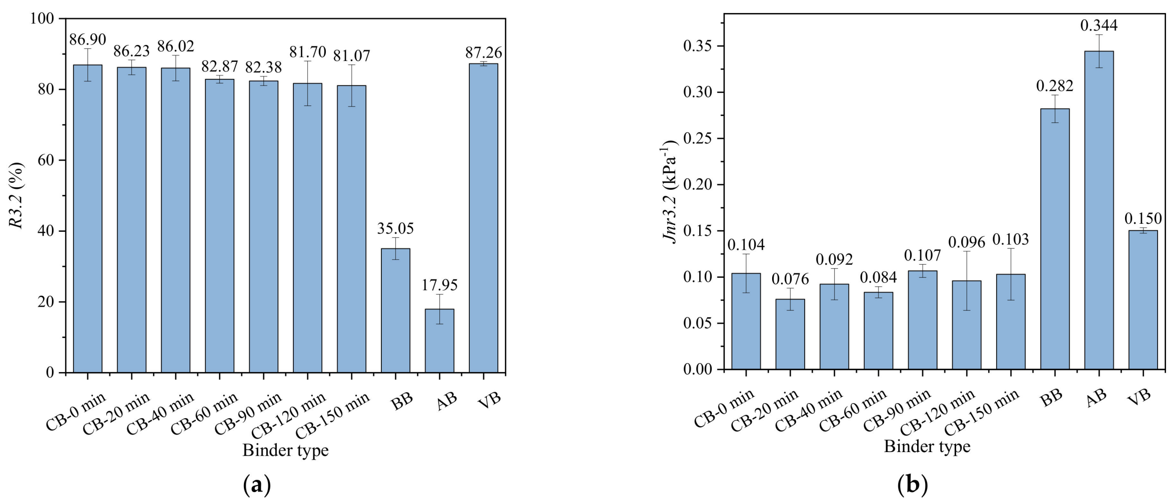


Figure 10. Results from MSCR tests for various binders. (**a**) *R3.2*; (**b**) *Jnr3.2*.

In terms of those results, it was observed that the creep deformation and recovering capacity of the CB were mainly determined by the virgin binder and the binder in the interfusion zone between the virgin binder and the aged binder, to some extent. Obviously, if the construction process is not well controlled in the actual practice, it is possible that the integration degree between the virgin binder and the aged binder would be inadequate and the aged binder does not interfuse well with the virgin binder. In this case, the role of the RAP in the recycled mixtures is more like a "black stone" with a poor adhesion to the virgin binder.

Based on the specification of AASHTO R 92-18 [31] and some references [12,32], the curve of *R* versus *Jnr* could be used to identify the elastic characteristic of the binder. If the results of *R3.2* versus *Jnr3.2* fall above the curve, the binder is considered to have a significant elastic response for the associated value of creep, indicating that the binder has been successfully enhanced and its elastic characteristic has been improved. In terms of the results illustrated in Figure 11, the results of CB were very close to that of the VB, implying that the virgin-aged composite binder exhibited similar elastic properties as the virgin binder, which is consistent with the results from the DSR and MSCR tests, whereas, the results of BB and AB fell below the curve (beneath the red-dash line in Figure 11), and correspondingly they displayed relatively weaker elastic characteristics. Furthermore, with the extension of the thermostatic-treatment duration (from 0 min to 150 min), the results of CB gradually moved downwards, which manifests that the gradual increasing of the integration degree between the virgin binder and the aged binder would lead to the reduction of elasticity of the whole composite binder, but the integration degree was still much lower than the completely miscible state, as in the BB.

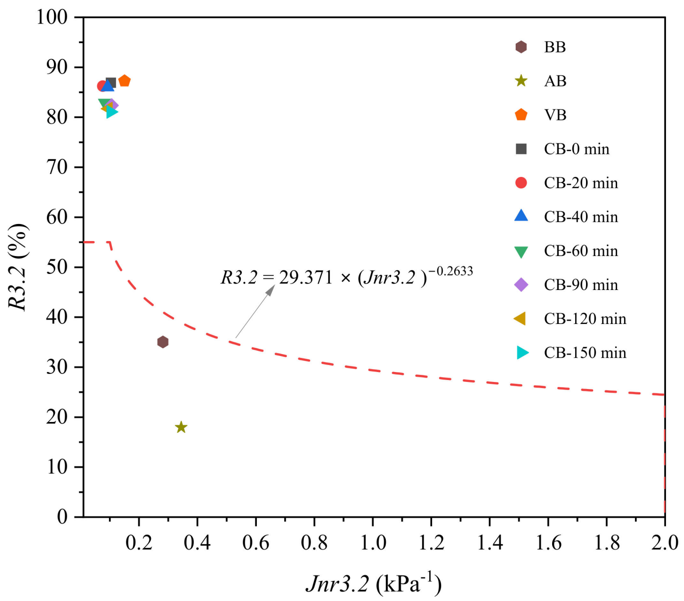


Figure 11. The results of *R3.2* vs. *Jnr3.2*.

In this study, we expected to obtain different interfusion degrees of virgin-aged composite binder through various thermostatic treatment durations and establish a connection between the interfusion degree and the adhesive performance or moisture-damage resistance. However, the DSR and MSCR tests showed no significant differences in the rheological properties and creep recovery capacity of virgin-aged composite binder at various durations. In fact, in the plant production, the thickness of asphalt film is usually below 10 μm, where the interfusion state between the virgin binder and aged binder has a significant impact on its performance. However, the binder sample used in the DSR tests (1 mm aged binder + 1 mm virgin binder) was too thick, compared to plant practices in which, although integration and diffusion occurred at the contact surfaces during the thermostatic treatment, the interfusion zone was insignificant in relation to the thickness of the two-layered composite binder sample, as shown in Figure 12. During testing, the lower layer (RAP binder) played a role of a "fixed plate or hard plate" in part due to its higher stiffness. The properties exhibited by the samples, at this point, were basically determined by the upper layer (virgin binder), and no significant difference could be observed on the properties of the binder samples in the effect of the thermostatic treatment process. This implies that the DSR test could not effectively recognize and differentiate the integration state of the composite binder samples, nor could it recognize the effect of the thermostatic treatment on the interfusion degree between the two layers of binders.

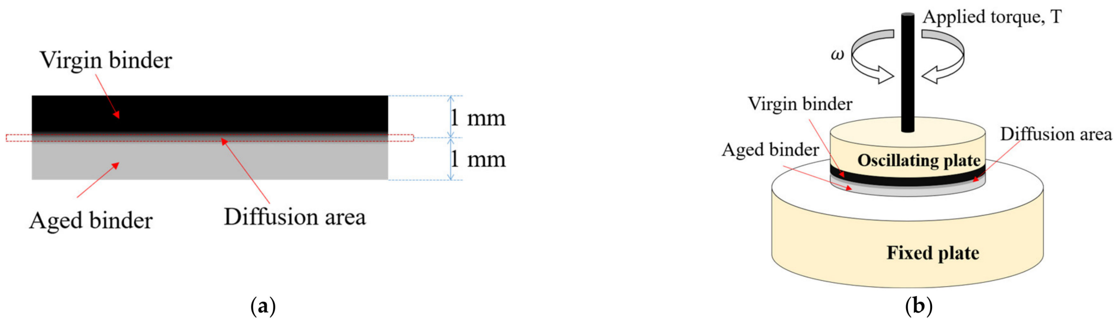


Figure 12. Schematic diagram of the composite binder in the DSR test. (**a**) two-layered composite binder; (**b**) Schematic diagram of DSR test.

5.1.3. Fatigue Resistance

Figure 13 illustrates the fatigue life, N_f, of the asphalt binders at 2.5% strain level from the LAS test. It can be observed that the fatigue resistance of AB was much lower than that of the other binders due to the hardening and the reduction of resilience induced by the aging of the material. The fatigue life of BB was in between that of the VB and AB, which manifests a relatively intermediate state of the binder being integrated. However, the fatigue life of CB with thermostatic treatments (from CB-0 min to CB-150 min) were all in between the AB and BB, and in general showed a tendency to increase with the increased treatment duration. This suggests that by promoting the interfusion between the virgin binder and aged binder through the thermostatic treatment, the fatigue resistance of the virgin-aged composite binder could be improved to some extent, and the higher the degree of integration, the longer the fatigue life of the composite binder.

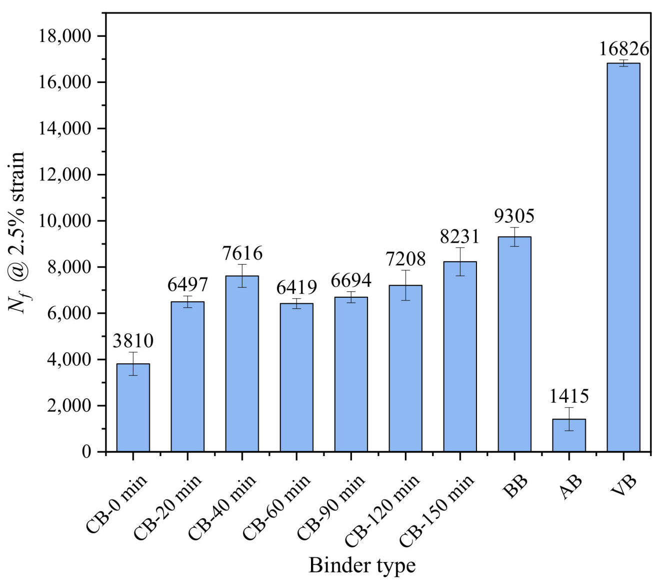


Figure 13. Fatigue life of binders at 2.5% strain level.

5.2. Adhesion Characteristics of Aggregate-Binder

5.2.1. Results from Modified Boiling Tests

The results from the modified boiling test are presented in Figure 14. The %_*residual binder* values for the virgin aggregates were overall higher than those of the RAP aggregates, which implies that the virgin aggregates exhibited better bonding performance to the asphalt binder under the action of water damages. Due to the variability of RAP materials, the coefficient of variation for the results of RAP aggregate was much greater than those for virgin aggregate. It was clear that although the same rock was used for testing, both basalts, the virgin aggregate exhibited stronger bonds with the asphalt binders with lower variabilities.

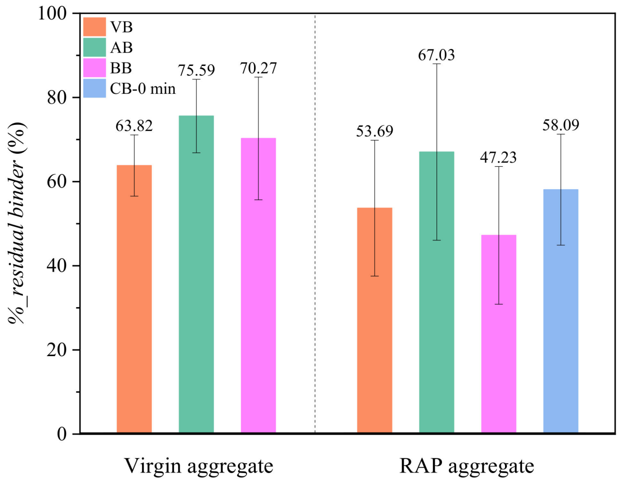


Figure 14. Results from the modified boiling tests.

The AB exhibited better adhesion performance to the aggregates than those of the VB and BB. This is because the asphalt binder that has undergone aging, on the one hand, the average molecular weight and viscosity of the binder increased due to the increased content of asphaltene and pectin, which results in an enhanced cohesive strength of the asphalt binder itself. On the other hand, the aged binder contains more asphaltene; thus, the number of hydrogen bonds between the binder and the silicon dioxide increased, which could furthermore strengthen the interaction between the aged binder and siliceous aggregate.

In the production of recycled asphalt mixtures, the asphalt coated on the RAP aggregate was a compound of virgin and RAP binder. Therefore, a combination of RAP aggregate and virgin-aged composite binder was selected for the boiling test to explore the moisture susceptibility of the binder-aggregate system (Figure 15). It was observed that the CB samples with a certain duration of the thermostatic treatment (from the CB-20 min to CB-150 min) presented a higher than those without the thermostatic treatment (the CB-0 min, BB, AB, and VB). The overall trend of increasing %_*residual binder* value for CB with increasing the thermostatic treatment duration indicated that the thermostatic treatment was beneficial in improving the adhesion properties.

Furthermore, it is worth mentioning that the stripping of the asphalt film usually did not occur on the binder-aggregate interface, due to the softening of the asphalt binder under high temperature and the divulsion of sustaining boiling bubbles during the testing process. This also reveals that the boiling method, to some extent, has a lack of accuracy and validity in regard to the evaluation of the adhesion performance between asphalt binder and aggregate.

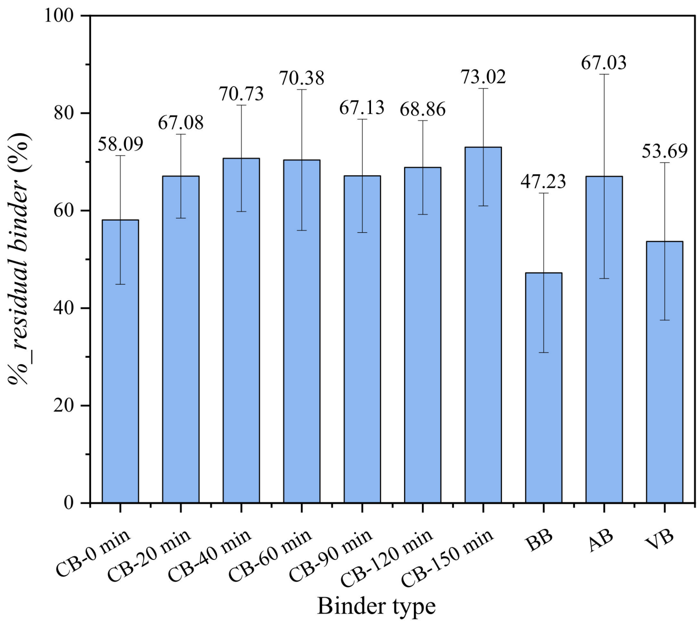


Figure 15. Results of binders-aggregate system with various thermostatic treatments.

5.2.2. Failure Behaviors of the Binder-Aggregate System

As shown in Figure 16, there are four typical debonding failure states of binder-aggregate system in the pull-off tests: cohesive failure (denoted as C), adhesive failure (denoted as A), rock failure (denoted as R), and hybrid failure (denoted as C/A or C/B).

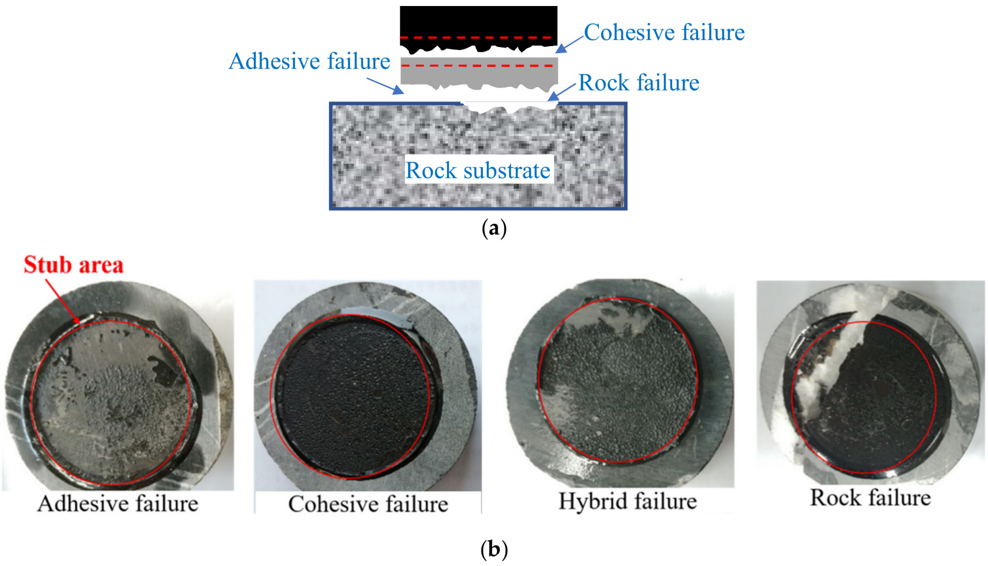


Figure 16. Various failure states of asphalt-aggregate system. (**a**) schematic diagram of various failure states; (**b**) various failures observed from tests.

The results of pull-off tests for all samples were summarized in Table 4. It was noticed that the binder-aggregate system mainly suffered cohesive failure and hybrid failure in dry conditions. In contrast, the hybrid failure and adhesive failure mainly occurred for the samples that had suffered water-immersion conditioning. This confirms the conclusion that water invasion could deteriorate the bonding strength of the asphalt binder to the aggregate due to the better affinity of the aggregate to water; thus, the failure state would be more inclined from cohesive failure to hybrid failure and adhesive failure. This was consistent with the findings in Moraes et al. [25,27]. In addition, occasionally if there was damage on the surface of the rock substrate, the rock failure or hybrid failure could be observed partially on the bonding interface between the aggregate and the binder.

Table 4. Summarized results from pull-off tests.

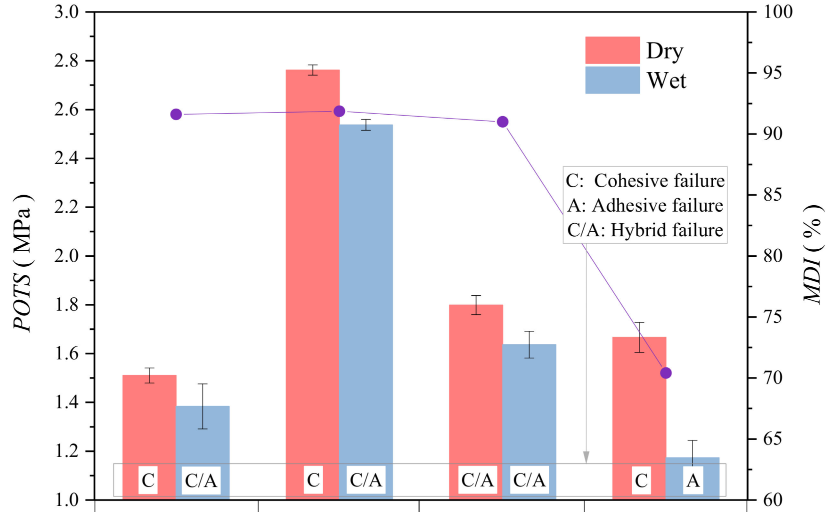
Binder Type	Thermostatic Treatment	Drying Conditioning			Wetting Conditioning		
		POTS		Failure State	*POTS*		Failure State
		Average (MPa)	Cov (%)		Average (MPa)	Cov (%)	
Virgin binder (VB)	-	1.51	3.07	C	1.38	9.26	C/A
Aged binder (AB)	-	2.76	2.10	C/R	2.54	2.25	C/A
Virgin-aged composite binder (CB)	0 min	1.67	6.14	C	1.17	7.10	A
	20 min	1.96	1.44	C	1.42	7.06	A
	40 min	2.23	4.48	C	1.72	2.58	C/A
	60 min	2.28	1.28	C/A	2.25	0.94	C/A
	90 min	2.37	3.37	C/A	2.38	1.83	C
	120 min	2.51	0.51	C	2.54	2.25	C
	150 min	2.65	3.41	C	2.69	3.59	C
Blended binder (BB)	0 min	1.80	3.90	C/A	1.64	5.49	C/A
	20 min	2.08	2.65	C/A			
	40 min	2.27	6.58	C/A			
	60 min	2.53	1.29	C			
	90 min	2.56	8.54	C			
	120 min	2.61	4.26	C			
	150 min	2.65	4.29	C			

5.2.3. Adhesion Characteristics and Moisture-Damage Resistance

The bonding strength obtained from pull-off tests (*POTS*) for binder-aggregate samples without thermostatic treatment are shown in Figure 17. In the dry condition, the AB exhibited a higher *POTS* than the BB and CB binders due to the increased internal cohesion of the asphalt binder and enhanced adhesion of the binder to the aggregate induced by aging, which is consistent with the results reported in the references [27,30,33]. The bonding strength of CB was in-between the virgin binder and aged binder due to the common influence of the virgin binder and the aged binder.

Combined with the failure states listed in Table 4, it was found that after the water-immersion conditioning, the adhesion strength of the binder to the aggregate was impaired and more hybrid failures could be observed in this case. The moisture damage index (*MDI*) for the VB, AB, and BB was 91.61%, 91.86%, and 91.00%, respectively, whereas the *MDI* for the CB was only 70.40%. Obviously, compared to the BB samples, the integration degree between the virgin and aged binders in the CB samples was relatively lower, no matter how long the thermostatic treatment duration is. Therefore, it could be inferred that the lower the integration degree, the weaker the bonding connections between the virgin binder and the aged binder. Moreover, water could intrude not only into the asphalt-aggregate interface, but also into the virgin and aged binders' interface, resulting in a weaker ability of the system to resist the moisture damage. Conversely, increasing the interfusion degree between the virgin binder and aged binder and making the binder integrated uniformly in the transition zone could improve the moisture stability of the recycled asphalt mixture.

The thermostatic treatment could not only promote the interfusion between the virgin and aged binder but strengthen the bonding strength of asphalt binder to aggregate. It could be interpreted by comparing the results of the VB-aggregate samples to the BB-aggregate samples after thermostatic treatments, as shown in Figure 18. As the components of the virgin binder and the aged binder in the BB were mixed and distributed fairly uniform, it could be assumed that the cohesion hardly changed with the thermostatic treatment duration, and the increase in *POTS* values was mainly determined by the adhesion between the binder and the aggregate. With the extension of the thermostatic-treatment duration, the bonding strength of both BB and CB increased. When the duration reached 60 min, the growth rate of the *POTS* of BB was retarded, and the failure state changed mostly from hybrid failure to cohesive failure. This implies that the adhesion between binder and aggregate was mainly enhanced in the early stage of the thermostatic treatment, and when the duration exceeded 60 min, the adhesion strength of the binder to the aggregate became higher than the internal cohesion of the binder, and after that the bonding strength for the system was then mainly determined by the cohesion of the binder. The cohesive strength of CB was enhanced more significantly by the continuous thermostatic treatment than the BB. With the extension of treatment, the bonding strength of CB increased much faster than that of the BB, and it became more remarkable after 60 min.



	VB	AB	BB	CB
$POTS_{Dry}$	1.5102	2.7621	1.7985	1.6664
$POTS_{Wet}$	1.3835	2.5373	1.6366	1.1732
MDI (%)	91.61	91.86	91.00	70.40

Figure 17. Results for various binder-aggregate systems.

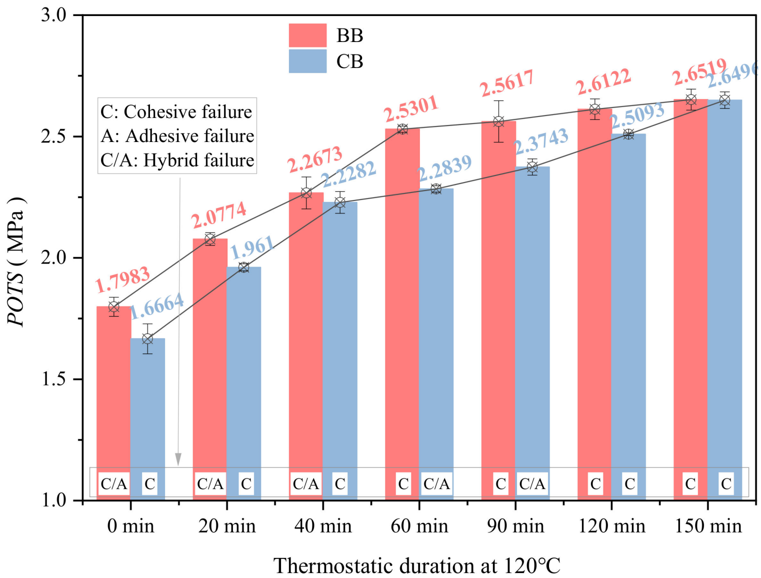
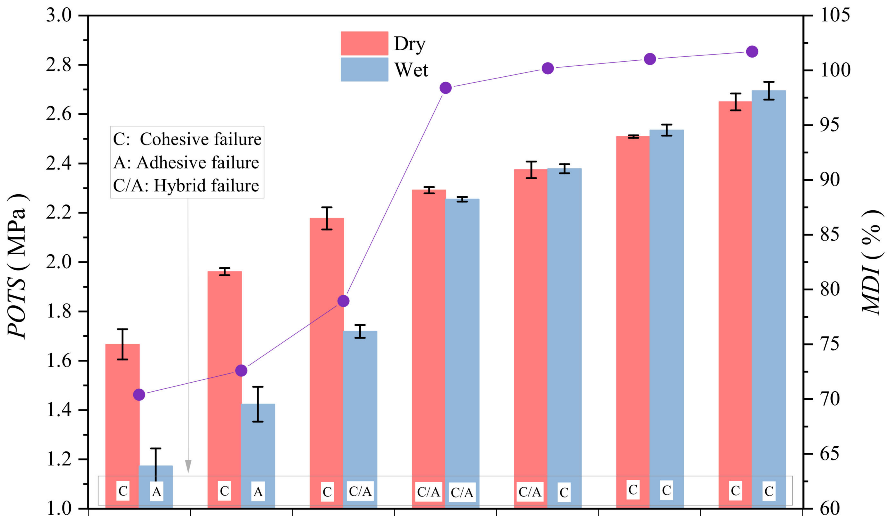


Figure 18. Bonding strength of BB and CB binder-aggregate systems.

The *POTS* and *MDI* results of the CB-aggregate system at various interfusion degrees are shown in Figure 19. The *POTS* and *MDI* both gradually increased with the thermostatic treatment from 0 min to 150 min, which proves that the interfusion degree and bonding strength between the virgin binder and aged binder could be improved by the thermostatic treatment. In conjunction with the failure states of the samples, in the early stage of the thermostatic treatment (from 0 min to 40 min), the adhesion between the binder and the aggregate was relatively weak. During the water-immersion treatment, moisture could easily intrude into the binder-aggregate and binder-binder interfaces and undermine their connections. When the duration was longer than 60 min, the asphalt binder and the rock substrate could bond more tightly with the increased adhesion and it was difficult for the moisture to invade the binder-aggregate interface. When the thermostatic-treatment duration reached 90 min, the $POTS_{wet}$ for the samples after wetting conditioning were greater than the $POTS_{dry}$ for the samples in drying condition, and at that time the *MDI* exceeded 100%.



	CB-0 min	CB-20 min	CB-40 min	CB-60 min	CB-90 min	CB-120 min	CB-150 min
$POTS_{Dry}$	1.6664	1.9610	2.2282	2.2839	2.3743	2.5093	2.6496
$POTS_{Wet}$	1.1732	1.4237	1.7189	2.2549	2.3787	2.5353	2.6947
MDI (%)	70.40	72.60	77.14	98.73	100.18	101.04	101.70

Figure 19. Results for CB-aggregate system under various treatment durations.

Based on the above analysis, it could be concluded that the thermostatic-treatment duration had a great influence on the interfusion degree between the virgin and the aged binders as well as the bonding strength (including adhesion and cohesion) of the binder-aggregate system, which further effected its moisture susceptibility. Extending the thermostatic-treatment duration could simultaneously improve the cohesion and adhesion of the binder-aggregate system and enhance its ability to resist moisture damage. Given this, it was suggested that in the actual practice the mixing time of the recycled asphalt mixture could be suitably prolonged to obtain a better integration for the materials, or a certain pre-fusion time could be provided for a part of the virgin asphalt or regenerating agent (if needed) to infiltrate into the RAP before the mixing process, such as set an atomizing-spray system in the RAP storage silo to moisture the material with the virgin binder or regenerating agent.

6. Conclusions and Recommendations

In this study, an experimental investigation was conducted on the adhesion characteristics and moisture susceptibility of the virgin-aged composite binder and the binder-aggregate system. Based on the results, the following conclusions can be drawn:

(1) The modulus of AB (aged binder) was much higher than that of the BB (blended binder) and VB (virgin binder), while the moduli of CB (virgin-aged composite binder) with various treatment durations all fell in between the VB and BB. No apparent difference could be observed on the rheological and creep properties of the CB with various thermostatic-treatment durations. The creep recovery capacity of CB was overall similar to that of VB, but much higher than that of the AB and BB. The fatigue resistance of AB was much lower than that of the other binders due to the aging. The fatigue life of CB with thermostatic-treatment durations were in-between the AB and BB, and in general showed a tendency to increase with the increased treatment duration.

(2) The bonding performance between the virgin aggregate and the binders was better than that of the RAP aggregate, whereas the adhesion of the aged binder (RAP binder) to the aggregate was stronger than that of the virgin binder. However, the modified boiling test was not so convincing as to distinguish the difference in adhesion for the CB binders under various interfusion degrees. For the aggregate coated with CB, the thermostatic treatment could promote the interfusion between the virgin binder and aged binder, as well as the adhesion of the binder to the aggregate.

(3) The bonding strength and the moisture damage index obtained from the pull-off tests were effective to reflect the adhesion characteristics and moisture susceptibility of the binder-aggregate system. With the extension of the thermostatic-treatment duration, the integration degree between the virgin binder and the aged binder in the CB was facilitated, and the cohesion of the asphalt binder and the adhesion between the binder and the aggregate could be both enhanced to some extent. There were four failure states of the binder-aggregate system observed in the tests, and the failure state changed along with the variation of cohesion and adhesion and affected by the actions of moisture.

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