Performance of Rejuvenated Asphalt and Mixtures with Waste Engine Oil Bottom and Liquid Styrene-Butadiene Rubber

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1. Introduction

If waste engine oil bottom (WEOB) cannot be rejuvenated, it poses a serious threat to the environment. Considering that both WEOB and asphalt come from petroleum and WEOB contains large amounts of light components that can soften asphalt; using WEOB in rejuvenated asphalt not only avoids environmental pollution but also realizes recyclability.

The research on the application of WEOB in asphalt originated in the United States, and in 2017, the American Asphalt Association issued the relevant standard, that is, ASTM D8125-17 [1]. Arnold and Gibson [2] studied the surface micromorphology of modified asphalt with WEOB before and after aging using atomic force microscopy (AFM) and found that adding WEOB makes tiny pores appear on the surface of asphalt, which increases in number with an increase in the degree of asphalt aging. Ding et al. [3, 4] studied the low-temperature performance of different asphalt binders using WEOB as a modifier and the regenerator of SBS-modified asphalt, respectively, thereby distinguishing more precisely the relationship between low-temperature performance and physical hardening of asphalt; by extending the aging time of asphalt and using more demanding test conditions such as the double edge notch test (DENT) and an extended bending beam rheometer (Ex BBR), the effect of WEOB on the low-temperature classification and durability of asphalt was evaluated. Furthermore, it was found that WEOB increased the continuous grading temperature difference ($\Delta T_c$) of the modified asphalt. Through a DENT, Li et al. [5] found that WEOB was
detrimental to the tensile extension performance of aged asphalt. Li et al. [6] studied the effects of types and mixing amounts of WEOB on the low-temperature performance of asphalt and found that the PG grade of asphalt decreased with an increase in the WEOB admixture. Using the research results of other scholars [7–10], it can be deduced that the main problem when WEOB is applied to the regeneration of an asphalt pavement is that some of the low-temperature performance indexes of the rejuvenated asphalt and asphalt mix are slightly inadequate.

In this study, liquid styrene-butadiene rubber (liquid SBR) compounded with WEOB (hereinafter abbreviated as WEOB-S) was used to improve the low-temperature performance of rejuvenated asphalt. The mechanical properties of WEOB or WEOB-S rejuvenated asphalt binders were studied by penetration, ductility, softening point, dynamic shear rheometer (DSR), and bending beam rheometer (BBR) tests. In addition, the microstructure of the rejuvenated asphalt binders was characterized by the differential scanning calorimetry (DSC), Fourier-transform infrared spectroscopy (FTIR), and atomic force microscope (AFM). Finally, the performance of the corresponding mixtures was evaluated by a series of traditional tests.

2. Materials and Methods

2.1. Materials. The original asphalt is Shell No. 70 road asphalt. The WEOB is red-brown at room temperature and is a residue that is recovered by membrane separation technology from the waste engine oil (WEO) of Hopper Environmental Technology Co. in Shandong. The basic properties of WEOB are listed in Table 1. The liquid SBR is a colorless and translucent paste-like viscous substance at room temperature, a random copolymer of butadiene and styrene, and has a glass transition temperature lower than room temperature, a random copolymer of butadiene and styrene, and has a glass transition temperature lower than room temperature. SBR is Shell No. 70 road asphalt. The rejuvenated asphalt was obtained by adding WEOB or WEOB-S with a mass that is 10% of the aged asphalt at 135°C and mixing the product at 2000 rev/min to form a homogeneous liquid, which is stored at room temperature for 24 h to obtain the WEOB-S.

Aged asphalt is prepared by aging the original asphalt for 85 min and 20 h in a rotary thin-film oven and pressure aging vessel, respectively. The rejuvenated asphalt was obtained by adding WEOB or WEOB-S with a mass that is 10% of the aged asphalt at 135°C and mixing the product at 1200 rev/min.

2.2. Methods

2.2.1. Asphalt and Rejuvenator Preparation. The compound rejuvenator WEOB-S is prepared by physical mixing: first, the WEOB is placed in an oven at 150°C for 30 min to remove the water, the liquid SBR is added (10% of the mass of WEOB), and a mixer is used to stir the contents for 20 min at 800 rev/min to form a homogeneous liquid, which is stored at room temperature for 24 h to obtain the WEOB-S. Aged asphalt is prepared by aging the original asphalt for 85 min and 20 h in a rotary thin-film oven and pressure aging vessel, respectively. The rejuvenated asphalt was obtained by adding WEOB or WEOB-S with a mass that is 10% of the aged asphalt at 135°C and mixing the product at 1200 rev/min.

2.2.2. Rheological Properties Test. High-temperature rheological properties tests were conducted using a AR2000ex rheometer with a 25-mm fixture at 1-mm intervals, a loading strain of 1%, and a frequency of 10 rad/s. A temperature scan test was conducted to obtain the complex moduli and phase angles at four temperatures of 52°C, 58°C, 64°C, and 70°C.

Bending beam rheological tests were conducted using a TE-BBR tester, the cooling temperature was not higher than −36°C, the temperature control accuracy was within 0.03°C, the specimen deformation measurement accuracy was less than 0.1555 μm, the force accuracy was 0.147 mN, and the load range of the beam could be measured from 0–45 g.

2.2.3. Differential Scanning Calorimetry Test. DSC was used to test the asphalt samples in a temperature range of −60°C to 150°C, at a heating rate of 10°C/min, and a nitrogen flow rate of 20 mol/min.

2.2.4. FTIR and AFM Microscopic Analysis Tests. Fourier transformation infrared spectroscopy (FTIR) and atomic force microscopy (AFM) were used to analyze the change patterns of functional groups and the surface micromorphology of asphalt, respectively, before and after aging and regeneration. The FTIR was a Thermo Scientific Nicolet iS5 with a scan range of 650–4000 cm⁻¹. The AFM was a Bruker Dimension ICON with a 0.4 N/m probe in a light mode at room temperature, scan range of 50 μm × 50 μm, resolution of 512 × 512, and resonance frequency of 260 kHz.

2.2.5. Performance Tests of Rejuvenated Asphalt Mixtures. Three types of rejuvenated asphalt mixtures were prepared: an A-Mix (with only the original asphalt and reclaimed asphalt pavement (RAP), and without any rejuvenator), a B-Mix (with WEOB), and a C-Mix (with WEOB-S). The proportion of RAP in the rejuvenated asphalt mixtures was 30% of the total mass of the mixture, the asphalt-aggregate ratio was 4.5%, and the dose of the WEOB or WEOB-S was 10% of the weight of the old asphalt in the RAP.

In accordance with the “Standard Test Methods of Bitumen and Bituminous Mixtures for Highway Engineering” (JTG E20-2011), a rutting test at 60°C, bending test at −10°C, and

| Table 1: WEOB basic properties. |
| Testing index | Unit | Test data |
|----------------|------|-----------|
| Appearance    | —    | Clear liquid |
| Ash           | %    | 0.033     |
| Mechanical impurities | % | 0.094 |
| Asphaltenes | %    | 2.1       |
| Saturates     | %    | 18.1      |
| Aromatics     | %    | 71.3      |
| Resins        | %    | 8.5       |

| Table 2: Basic properties of liquid SBR. |
| Testing index | Unit | Test data |
|----------------|------|-----------|
| Color          | —    | White translucent viscous |
| Molecular weight | — | 50000 |
| Styrene content | % | 25–28 |
| Volatility     | %    | ≤0.1      |
| Ash            | %    | ≤0.1      |
| Elongation at tear | % | ≥500–650 |
freeze-thaw splitting test were conducted on the three types of rejuvenated asphalt mixtures to study the high-temperature performance, low-temperature performance, and water stability performance of the rejuvenated asphalt mixtures.

3. Test Results and Analysis

3.1. Three Major Indicators Test Results. From the data in Table 3, it can be seen that WEOB and WEOB-S can effectively recover the penetration and softening point of aged asphalt, but the recovery ability of the ductility at 10°C shows a large difference, and the recovery efficiency of WEOB-S on the ductility of aged asphalt is significantly greater than that of WEOB. This indicates that the light component in WEOB is effective in softening the aged asphalt and reducing its viscosity but is very unsatisfactory in recovering the ductility, with a recovery rate of only 26.3%. This is also an important reason why the low-temperature performance of the WEOB-rejuvenated asphalt and asphalt mixture is not ideal. Adding liquid SBR enables the WEOB-S-rejuvenated asphalt to effectively recover the ductility based on the effective recovery of the penetration and softening point, and the recovery rate can reach 92.42%, which is a very substantial improvement effect.

3.2. Dynamic Shear Rheology Test Results. The effects of WEOB and WEOB-S on the high-temperature rheological properties of aged asphalt are shown in Figure 1. After adding WEOB or WEOB-S rejuvenators to the aged asphalt, the rutting factors were reduced to different extents. This indicates that the addition of WEOB or WEOB-S rejuvenators can effectively reduce the hardness of aged asphalt and bring its high-temperature performance grade close to that of the original asphalt. The rutting factor of WEOB-S-rejuvenated asphalt was slightly larger than that of WEOB-rejuvenated asphalt and higher than that of the original asphalt, which indicated that liquid SBR rubber had a certain enhancement effect on the high-temperature performance of the rejuvenated asphalt.

3.3. Bending Beam Rheological Test Results. Table 4 lists the BBR test results of the original asphalt, aged asphalt, WEOB-rejuvenated asphalt, and WEOB-S-rejuvenated asphalt. As can be seen from the BBR test results, the low-temperature performance grade of the original asphalt is PG-22, and it is increased to PG-16 after aging. Adding WEOB or WEOB-S rejuvenators can make the low-temperature performance grade of the aged asphalt reach PG-28, which is lower than the original asphalt. This indicates that the WEOB and WEOB-S rejuvenators can effectively reduce the low-temperature performance grade of asphalt. In addition, the continuous grading temperature at an average creep rate of $m = 0.3$ was analyzed according to ASTM 7643 [11], and it was deduced that the continuous grading temperature of pure WEOB-rejuvenated asphalt was $-28.4°C$, and the continuous grading temperature of WEOB-S-rejuvenated asphalt was $-33.0°C$, which is reduced by $4.6°C$. This indicates that liquid SBR has a further apparent improvement effect on the low-temperature stress relaxation performance of rejuvenated asphalt.

3.4. Differential Scanning Calorimetry Test. The internal factors that cause low-temperature performance deterioration of asphalt mainly include the low-temperature crystallization process of wax in asphalt, the aggregation of asphaltenes, and the collapse of free volumes. The low-temperature crystallization of the wax changes the phase structure of the asphalt, resulting in a deterioration of its internal mechanical properties. The aggregation phenomenon of asphaltenes results in the phases separation of asphalt, and the asphaltenes as a dispersed phase tends to settle after
| Materials                  | Test temperature −6°C | Test temperature −12°C | Test temperature −18°C | Test temperature −24°C | Test temperature −6°C | Test temperature −12°C | Test temperature −18°C | Test temperature −24°C | Continuous grading temperature at m = 0.3 (°C) | Continuous grading temperature at m = 0.3 (°C) | Asphalt low-temperature performance grade |
|----------------------------|------------------------|-------------------------|-------------------------|------------------------|------------------------|-------------------------|-------------------------|------------------------|-----------------------------------------------|-----------------------------------------------|---------------------------------------------|
| Original asphalt           | —                      | 0.356                   | 0.291                   | 0.234                  | —                      | 156                     | 315                     | 554                    | −17.2                                         | −27.2                                         | PG-22                                       |
| Aged asphalt               | 0.348                  | 0.289                   | 0.266                   | —                      | 94.3                   | 228                     | 367                     | —                      | −10.9                                         | −20.9                                         | PG-16                                       |
| WEOB-recycled asphalt     | —                      | 0.34                    | 0.302                   | 0.269                  | —                      | 59.4                    | 119                     | 198                    | −18.4                                         | −28.4                                         | PG-28                                       |
| WEOB-S-recycled asphalt   | —                      | 0.376                   | 0.343                   | 0.291                  | —                      | 49                      | 91.9                    | 188                    | −23.0                                         | −33.0                                         | PG-28                                       |

Table 4: BBR test results.
aggregation, prohibiting the asphalt from forming a stable colloidal structure. According to the free volume theory, the internal structure of asphalt can be divided into free volume and occupied volume, and a volume of thermal expansion or contraction occurs with a change in the temperature. When the temperature continues to decrease, the ability of mutual movement between molecules continues to weaken until the asphalt reaches a glassy state. At this state, the free volume in the asphalt is frozen and no longer changes, which leads to a weakening of the asphalt deformation ability. Therefore, lower glass transition temperatures correspond to better low-temperature performance.

DSC was used to obtain the glass transition temperature of asphalt, and the DSC diagrams of the original asphalt, aged asphalt, WEOB-rejuvenated asphalt, and WEOB-S-rejuvenated asphalt are shown in Figure 2. The glass transition temperature of the WEOB-S-regenerated asphalt is smaller than that of the WEOB-regenerated asphalt, which indicates that liquid SBR can enhance the regeneration effect of WEOB on the low-temperature performance of aged asphalt. Liquid SBR has a low glass transition temperature, which can also reduce the glass transition temperature of asphalt, and the rubber has a strong elastic deformation ability, which can be added to asphalt to enhance its deformation ability in low-temperature conditions.

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3.5. Fourier-Transform Infrared Spectroscopy Test. The content of carbonyl and sulfoxide groups in the infrared spectrum of asphalt can characterize the aging degree to some extent. The infrared spectrum in Figure 3 shows that the content of carbonyl (C=O) and sulfoxide (S=O) groups increased after asphalt aging, mainly due to their carbon and sulfur elements reacting with oxygen during asphalt aging, respectively, which generated oxygen-containing
compounds such as aldehydes, ketones, and esters [12]. The characteristic peaks of carbonyl and sulf oxide groups were significantly weakened after WEOB and WEOB-S were added, indicating that the WEOB and WEOB-S can reduce the content of carbonyl and sulf oxide groups in the aged asphalt, thereby reducing the aging of the asphalt. Figure 3 also shows that there is no chemical reaction to generate new substances during the regeneration of aged asphalt by WEOB and WEOB-S, and the regeneration of aged asphalt is achieved by replenishing the oil lost during the aging process and dissolving the dispersed asphaltene. WEOB-S-rejuvenated asphalt showed vibration peaks of aromatic compounds and olefin $C=\text{C}$ bonds at $840 \text{ cm}^{-1}$ and $933 \text{ cm}^{-1}$, respectively, which were mainly caused by the addition of liquid SBR.

### 3.6. Atomic Force Microscopy (AFM) Test

The surface microscopic morphology of the original, aged, and two rejuvenated asphalts is shown in Figure 4. The surfaces of all four asphalts have bee-like-structure distributions with different characteristics. Some scholars believe that the main component of the bee-like structure is asphaltene [13–15], some believe that it is a type of wax crystal [16–20], and some consider that it is a product of the strong polar asphaltene that is attached to the wax as a nucleus. However, most scholars agree that the formation of this structure is related to crystallization and aggregation [13–20].

From Figure 4, it can be seen that the surface microscopic morphological characteristics of asphalt before and after aging or regeneration are considerably different. The number of bee-like structures decreased, the crests became higher, and the structures became longer after asphalt aging, indicating that the aging effect of asphalt intensified the aggregation of bee-like crystalline substances, which may be related to an increase in the molecular weight and polarity of the asphaltene after aging and the possibility of asphaltene aggregating into clusters. Adding WEOB and WEOB-S to the aged asphalt resulted in a slight increase in the number of bee-like structures, lower peaks, and shorter structures relative to the aged asphalt, which indicates that adding the two rejuvenators resulted in a better dispersion of the bee-like crystalline material in the aged asphalt and a more uniform distribution of the dispersed phase in the dispersion medium. Compared with the WEOB-rejuvenated asphalt, the bee-like structure of the WEOB-S-rejuvenated asphalt is more uniformly distributed and has a lower crest, which closer resembles the bee-like structure on the surface of the original asphalt, indicating that adding liquid SBR is conducive to the restoration of the uniformity of the surface structure of the WEOB-rejuvenated asphalt, which may be one of the reasons for the improvement in some low-temperature performance indexes of WEOB-S-rejuvenated asphalt [21].

### 3.7. Performance of Rejuvenated Asphalt Mixes

#### 3.7.1. Reclaimed Asphalt Mixture Ratio and Volume Index

To investigate the effect of the two rejuvenators on the performance of rejuvenated asphalt mixes, three types of rejuvenated asphalt mixture specimens were molded: an A-Mix (no rejuvenator), a B-Mix (with WEOB), and a C-Mix (with WEOB-S). The grade type of the three rejuvenated asphalt mixtures is AC-20°C, the grade composition of the mixtures is listed in Table 6.

#### 3.7.2. High-Temperature Performance

The rutting test results of the three types of rejuvenated asphalt mixes are listed in Table 7. The dynamic stability of rejuvenated asphalt mixture A-Mix, without the addition of rejuvenator, was the highest, which is related to the higher hardness of the aged asphalt in the RAP. The dynamic stabilities of the B-Mix and C-Mix are close and slightly lower than that of the A-Mix but much higher than the technical requirements for dynamic stability in Technical Specifications for Construction of Highway Asphalt Pavements [22]. This indicates that adding WEOB-S or WEOB effectively reduces the hardness of the aged asphalt while maintaining a better high-temperature stability.

#### 3.7.3. Low-Temperature Performance

From the bending test results of three kinds of recycled asphalt mixes in Table 8, it can be seen that, compared with the recycled asphalt mixes without regenerant, the addition of WEOB makes the recycled asphalt mixes reduce the modulus of stiffness, increase the maximum bending tensile strain, and improve the low-temperature crack resistance to a certain extent, while the addition of Liquid SBR makes the low-temperature crack resistance of WEOB-recycled asphalt mixes with more significantly improved. This is mainly due to the fact that the addition of regenerant can restore the properties of aging asphalt and improve the integration of old and new asphalt, and the addition of liquid SBR improves the stress relaxation capacity of asphalt binder and asphalt mixture, resulting in better low-temperature performance.
Figure 4: Asphalt AFM scan results: (a) original asphalt, (b) aged asphalt, (c) WEOB-rejuvenated asphalt, (d) and WEOB-S-rejuvenated asphalt.
3.7.4. Water Stability Performance. From the data in Table 9, it can be seen that the splitting strength ratio of the rejuvenated asphalt mixture with WEOB significantly increased compared to that of the asphalt without the rejuvenator. Simultaneously, the splitting strength of the WEOB-S-rejuvenated asphalt mixture modified using a liquid SBR was further increased than that of the WEOB-rejuvenated asphalt mixture. This indicates that liquid SBR has a good modification effect on WEOB and can enhance the water stability of a WEOB-rejuvenated asphalt mixture.

| Table 5: Grade composition of rejuvenated asphalt mixture. |
|-----------------------------------------------------------|
| Sieve hole size (mm) | Design grading | Pass rate (%) | Grading range |
|----------------------|----------------|---------------|---------------|
| 26.50                | 100            | 100           |               |
| 19.00                | 92.3           | 90–100        |               |
| 16.00                | 88.5           | 78–92         |               |
| 13.20                | 75.9           | 62–80         |               |
| 9.50                 | 60.4           | 50–72         |               |
| 4.75                 | 34.4           | 26–45         |               |
| 2.36                 | 23.5           | 16–44         |               |
| 1.18                 | 17.7           | 12–33         |               |
| 0.60                 | 13.2           | 8–24          |               |
| 0.30                 | 9.0            | 5–17          |               |
| 0.15                 | 7.3            | 4–13          |               |
| 0.08                 | 5.6            | 3–7           |               |

| Table 6: Marshall specimen test results of different rejuvenated asphalt mixes. |
|-------------------------------------------------------------------------------|
| Mix type | Asphalt-aggregate ratio (%) | Gross volume relative density | Maximum theoretical density | Synthetic gross bulk density of minerals | Porosity VV (%) | Mineral gap rate VMA (%) | Asphalt saturation VFA (%) | Stability (KN) | Flow value (mm) |
|----------|-----------------------------|--------------------------------|----------------------------|------------------------------------------|----------------|------------------------|------------------|---------------|----------------|
| A-mix    | 4.5                         | 2.440                          | 2.56                       | 2.732                                    | 4.7            | 14.5                   | 67.7             | 12.6          | 3.2            |
| B-mix    | 4.5                         | 2.447                          | 2.558                      | 2.732                                    | 4.3            | 14.3                   | 69.6             | 11.6          | 3.5            |
| C-mix    | 4.5                         | 2.444                          | 2.556                      | 2.732                                    | 4.5            | 14.5                   | 68.8             | 13.2          | 2.8            |
| Technical specifications |                         |                                |                             |                                          | 3–5            | 12                     | 55–70            | 8             | 2–4            |

| Table 7: Results of 60°C rutting test on rejuvenated asphalt mixes. |
|---------------------------------------------------------------------|
| Rejuvenator type | A-mix | B-mix | C-mix |
| Dynamic stability DS (times/mm) | 3684  | 3333  | 3387  |

| Table 8: Rejuvenated asphalt mixture –10°C bending test results. |
|---------------------------------------------------------------|
| Rejuvenator type | Maximum load $P_b$ (N) | Span center disturbance $d$ (mm) | Maximum bending tensile strain $\varepsilon_B$ (µε) | Bending and tensile strength $R_{B}$ (MPa) | Bending stiffness modulus $S_{B}$ (MPa) |
|-------------------|----------------------|-------------------------------|--------------------------------|------------------------|-------------------------------|
| A-mix             | 605                  | 0.38                          | 1983                         | 4.90                   | 2471                          |
| B-mix             | 637                  | 0.40                          | 2132                         | 5.07                   | 2383                          |
| C-mix             | 713                  | 0.46                          | 2445                         | 5.71                   | 2341                          |

| Table 9: Freeze-thaw splitting test results of rejuvenated asphalt mixes. |
|---------------------------------------------------------------------|
| Rejuvenator type | Unfrozen-thawed splitting strength (MPa) | Splitting strength after freeze-thaw (MPa) | Splitting strength ratio TSR (%) |
|-------------------|------------------------------------------|-------------------------------------------|-------------------------------|
| O-mix             | 0.875                                    | 0.681                                     | 77.8                          |
| WEOB-mix          | 0.991                                    | 0.834                                     | 84.3                          |
| WEOB–S-mix        | 1.035                                    | 0.916                                     | 88.6                          |
4. Conclusion

The WEOB has a good recovery effect on the penetration and softening point of aging asphalt, can reduce its low-temperature performance grade and glass transition temperature, and improves its low-temperature deformation capacity; however, the improvement effect on the low-temperature ductility of aged asphalt is not ideal.

A compound rejuvenator (WEOB-S) composed of liquid SBR and WEOB restores the penetration, softening point, and low-temperature ductility of aged asphalt. Compared with WEOB, WEOB-S further reduces the continuous grading temperature and glass transition temperature of aged asphalt, thereby improving its low-temperature performance.

An AFM test showed that the number of bee-like structures formed by wax crystals or macromolecular asphaltene decreases after asphalt aging, the crests become significantly higher, the structures become longer, and the uniformity of the asphalt becomes poor. After adding WEOB or WEOB-S to the aged asphalt, the bee-like structures are effectively dispersed, their number increases, the crests become lower, the structure becomes shorter, and the microscopic uniformity of the aged asphalt is improved. Compared with WEOB, the WEOB-S-rejuvenated asphalt has a higher number of bee-like structures, with lower peaks, a more uniform distribution, and is closer to the bee-like structures on the surface of the original asphalt, indicating that adding liquid SBR is beneficial for restoring the microstructure uniformity on the surface of the rejuvenated asphalt, which may lead to better macroscopic physical properties.

WEOB as a rejuvenator can improve the low-temperature performance and water stability of rejuvenated asphalt mixtures. Adding liquid SBR has a greater improvement on the low-temperature performance and water stability of rejuvenated asphalt mixtures.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this article.

Authors’ Contributions

Gang Zhou and Yi Tian conceived the study; helped with methodology; validated the study; and formally analyzed the study. Yi Tian and Liang Gong investigated the study. Gang Zhou helped with resources. Yi Tian, Liang Gong, and Zhansheng Pang curated the study; Yi Tian and Tianquan Chen wrote the original draft of the manuscript; Gang Zhou reviewed and edited the manuscript; Yi Tian helped with the study; Gang Zhou supervised and helped with project administration and funding acquisition.

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