Research Article

Preparation and Performance Characterization of Warm-Mix Rejuvenated Bioasphalt

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Received 19 May 2020; Revised 21 July 2020; Accepted 30 July 2020; Published 13 August 2020

Academic Editor: Fan Gu

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Reclaimed asphalt pavement (RAP) material is generally under a large amount of aged asphalt coated on the surface of the aggregates, which often limits its (RAP) wide application. Bioasphalt is a renewable material with constituent components that are comparable to regular petroleum asphalt and has the potential to rejuvenate the performance of the aged asphalt in RAP materials. In this laboratory study, the optimum bioasphalt dosage for rejuvenating and optimizing warm-mix rejuvenated bioasphalt (WBA) performance, in the presence of styrene-butadiene-styrene (SBS) and naphthenic oil additives, was investigated. The laboratory test results of the single-factor and orthogonal experiment indicated that the optimal dosage of bioasphalt in WBA was 30% at 4% SBS and 10% naphthenic oil, respectively. The test results further indicated that the WBA’s ductility properties were highly responsive to the bioasphalt dosage. Naphthenic oil, on the other hand, had the greatest impact on the WBA fluidity. Likewise, the Brookfield viscosity experimentation showed that the fluidity of WBA and aged asphalt with different contents at 30%, 40%, 50%, 60%, and 70%, respectively, satisfactorily met the Chinese construction requirements. The adhesion test indicated excellent adhesion and good moisture stability properties for WBA. The resultant PG grades of WBA blended with 40% and 70% aged asphalt were PG 64-34 and PG 70-22, respectively. Microstructure and surface morphology analysis showed carbonyl absorption peaks with high rejuvenation and surface-crack repair potential for the WBA over the control asphalt that was evaluated in the study.

1. Introduction

Recycled asphalt pavement (RAP) is a kind of recycled material that is obtained by milling and reclaiming old existing asphalt pavements [1, 2]. Because the surface of RAP is generally covered with various aged asphalt [3], the RAP material may quite often reduce the performance of a freshly mixed asphalt pavement when it is used as an additive or aggregate replacement [4, 5]. Therefore, it becomes necessary to add an asphalt regeneration/rejuvenation agent [5]. However, the application of RAP in hot-mix asphalt (HMA) and warm-mix asphalt (WMA) technology may face the problem of secondary aging of asphalt [6, 7], which significantly weakens the performance and service life of the corresponding asphalt-mixture and pavement structure [8, 9]. Although modifiers such as styrene-butadiene-styrene (SBS), styrene-butadiene-rubber (SBR), and polyethylene (PE) can improve its performance, the asphalt mixture may still be prone to aging than the original asphalt [10–14].

Apart from being a by-product of industrial waste, bioasphalt is an environmentally friendly asphalt that is comparable to and compatible with regular petroleum asphalt [15–17]. Due to its cost-effectiveness, readily available, and recyclability [18, 19], it can be blended into petroleum asphalt as a modifier or be used to recycle/rejuvenate or completely replace regular asphalt [20–22]. However, the strong hydrophilicity and hydrolysis of bioasphalt often lead to poor moisture stability of the resultant mixture [23, 24].

In this laboratory study, bioasphalt was investigated for its potential to rejuvenate, enhance, and optimize the
rheological properties and performance of warm-mix rejuvenated bioasphalt (WBA). SBS and naphthenic oil were employed to improve the WBA fluidity. Single-factor and orthogonal experiments were designed to determine the optimal amount of each component. After mixing with different amounts of aged asphalt, the Brookfield viscosity was invoked as a control index to evaluate and quantify the WBA fluidity. The Dynamic Shear Rheometer (DSR), Bending Beam Rheometer (BBR), and adhesion tests were used to evaluate the rheological properties and moisture susceptibility of the recycled asphalt, respectively. Fourier transform infrared (FT-IR) and scanning electron microscopic (SEM) observations were used to analyze the repair and rejuvenation process of the aged asphalt including microevaluation of the repair mechanism and effects of WBA on the aged asphalt.

2. Materials, Experimental Design, and Test Methods

The materials used in the study are discussed below and also include the experimental design plan and test methods. The material description includes the index properties.

2.1. Materials

2.1.1. Bioasphalt. Bioasphalt was employed as a rejuvenated agent for the aged asphalt. The main technical indexes of bioasphalt are summarized in Table 1.

2.1.2. Original and Aged Petroleum Asphalt. The original, unaged, petroleum asphalt was graded as 70°F (i.e., Pen 70) in compliance with the Chinese standard specifications [25], whereas the aged asphalt comprised the original petroleum asphalt that was subjected to the standard rolling thin film oven test (RTFOT) and treated as RTFOT residue.

2.1.3. Naphthenic Oil (NO). Naphthenic oil was used as a softening agent for the original petroleum asphalt. NO contains many saturated cyclic carbon chains that provide compatibility with the original petroleum asphalt. The index properties of NO are shown in Table 2.

2.1.4. Styrene-Butadiene-Styrene (SBS). Aging of the petroleum asphalt undesirably reduces the low-temperature performance of the corresponding asphalt-mixture as well as the adhesion of the asphalt to the aggregates [26]. SBS was used in this study as a modifier for the preparation of WBA to mitigate the potential performance degradation resulting from aging.

2.2. Experimental Design and Test Methods. A single factor test was used to determine the influence of SBS, NO, and bioasphalt on the performance of the original asphalt. After blending with SBS, bioasphalt, and NO separately, the technical indexes of the asphalt blends were evaluated using the following test methods: penetration, softening point, ductility (at 5°C), and Brookfield viscosity (at 135°C) [27]. Based on the single-factor test results, the content range of each influencing factor was then determined for the orthogonal-factor experimentation to evaluate and quantify the interactive effects of the asphalt blends. Three dosages were tested in the orthogonal experiment to determine the optimal blend proportions of WBA. Three combinational dosage levels for SBS, bioasphalt, and NO were evaluated. Under the orthogonal experiment plan, the following standard laboratory tests were conducted to quantify the properties and performance characteristics of WBA: viscosity, ductility, BBR, DSR, FT-IR, and SEM.

3. Laboratory Test Results and Analysis

The laboratory test results are presented, analyzed, and discussed in this section. This includes the additive effects on the original asphalt properties, the WBA blend proportions, rheological properties, adhesion, and microstructure analysis.

3.1. Influence of Additives on the Asphalt Properties and Performance. The single factor test results for SBS, bioasphalt, and NO additives on the original asphalt are discussed below.

3.1.1. SBS Effects on the Original Asphalts. SBS and the original asphalt were preheated to 150°C for 30 min and thereafter, the temperature was elevated to 170°C. The blends were then mixed at 4000 rpm for 40 min, and finally, at 150°C, for 30 min. To study the effects of SBS dosage on the original asphalt performance, various contents of SBS were evaluated including 0%, 3%, 4%, 5%, and 6%, respectively, by weight of the total asphalt blend. The penetration, ductility, softening point, and apparent viscosity results are plotted in Figure 1. It has been reported in the literature that the apparent viscosity at 135°C [27] is an indicator of favorable workability for the asphalt. According to SHRP [26], the maximum apparent viscosity of 3 Pa·s is specified.
Figure 1(d) shows that when the SBS dosage increased, the apparent viscosity increased significantly. When 6% SBS was blended, the apparent viscosity was 1.71 Pa·s, at which point the penetration was 40 (0.1 mm) and the softening point was 83°C. As shown in Figure 1(c), peak ductility of 68 cm occurred at 4% SBS dosage. The ductility decreased to 64 cm when the SBS dosage was increased to 6%. Overall, the ductility results suggest that 4% was the optimum SBS dosage for optimizing laboratory performance (Figure 1(c)).

3.1.2. Bioasphalt Effects on the Original Asphalt. To ensure fluidity, bioasphalt and the original asphalt were preheated to 140°C in the oven for 45 minutes. Thereafter, the blends were mixed at 1500 rpm for 15 min to ensure a homogeneous blend. The mass fraction of the bioasphalt was 0, 25%, 30%, 35%, and 40%, respectively. The test results in terms of penetration, softening point, ductility, and viscosity are shown in Figure 2.

According to Figure 2(d), as the bioasphalt dosage increased, the apparent viscosity decreased, indicating that bioasphalt enhanced the fluidity of the asphalt blend. When the content of bioasphalt reached 30%, the downward trend of Brookfield viscosity became gentle. Figure 2(a) shows that the penetration of the asphalt blend increased. When the bioasphalt dosage was 30%, the penetration value increased slowly. Figures 2(b) and 2(c) suggest that the ductility and softening point of the asphalt blend decreased. The peak value, with a softening point of 41°C and ductility of 55 cm, appeared when the bioasphalt dosage was 0%. This is because the light component in bioasphalt softens the asphaltene. Overall, bioasphalt enhanced the viscosity characteristics of
the asphalt blend, but it reduced the low-temperature ductility performance of the asphalt blend.

3.1.3. Effects of NO on the Original Asphalt. Naphthenic oil and the original asphalt were preheated to 120°C in the oven for 45 min. Thereafter, the blend was mixed at 1500 rpm for 15 min. Figure 3(d) indicates that when 10% of NO was blended, the apparent viscosity was 0.18 Pa·s. However, the apparent viscosity exhibited a decreasing trend with increasing NO dosage—a similar response trend shared by the softening point and bioasphalt, as shown in Figures 3(b), 2(b), and 2(d), respectively. The results indicate that the NO could potentially enhance the asphalt’s fluidity, which is a critical aspect during construction in terms of workability and compactability. Figures 3(a) and 3(c) show an increasing trend in penetration (for all the dosage levels) and ductility with increasing NO dosage up to 8%. Overall, these graphical results suggest that while NO can improve the low-temperature properties and performance of the asphalt, it could adversely affect the asphalt’s high-temperature performance.

3.2. Optimal Blend Proportions for WBA. Based on the above single-factor test results, SBS with 3%, 4%, and 5% dosages were evaluated to determine the optimum WBA proportions along with bioasphalt (25%, 30%, and 35%) and NO (6%, 8%, and 10%). Owing to the relatively poor low-temperature performance of the aged asphalt [28] and the workability of WMA construction, the 5°C ductility and 135°C Brookfield viscosity was selected as the indicative parameters for quantifying performance and determining the optimum additive dosages. Three factors and three levels of orthogonal test experimentation, i.e., $L_9 (3^4)$, were selected to determine the optimal blending amount of each component of the WBA blend. The test results of the orthogonal experimentation and analysis are shown in Tables 3 and 4, respectively.
Figure 3: Single factor test results of naphthenic oil variation. (a) Penetration test results. (b) Softening point test results. (c) Ductility test results. (d) Brookfield viscosity test results.

Table 3: Orthogonal experimentation test results.

| No. | SBS (A) | Bioasphalt (B) | NO (C) | Ductility (cm) | Brookfield viscosity (Pa·s) |
|-----|---------|----------------|--------|---------------|---------------------------|
| 1   | 3% (1)  | 25% (1)        | 6% (1) | 50.3          | 0.486                     |
| 2   | 3% (1)  | 30% (2)        | 8% (2) | 50.8          | 0.431                     |
| 3   | 3% (1)  | 35% (3)        | 10% (3)| 50.6          | 0.395                     |
| 4   | 4% (2)  | 25% (1)        | 8% (2) | 57.1          | 0.657                     |
| 5   | 4% (2)  | 30% (2)        | 10% (3)| 59.6          | 0.433                     |
| 6   | 4% (2)  | 35% (3)        | 6% (1) | 55.2          | 0.643                     |
| 7   | 5% (3)  | 25% (1)        | 10% (3)| 51.4          | 0.825                     |
| 8   | 5% (3)  | 30% (2)        | 6% (1) | 50.6          | 0.842                     |
| 9   | 5% (3)  | 35% (3)        | 8% (2) | 45.8          | 0.886                     |
Table 3 shows that when the Brookfield viscosity was invoked as the reference parameter, the minimum viscosity value in the first group of three was 0.395 Pa·s, with ductility of 50.6 cm. With the addition of more aged asphalt, the low-temperature performance of the WBA blend worsened, so this group was abandoned.

The maximum ductility value in the second group was 59.6 cm (Table 3) with a Brookfield viscosity value of 0.43 Pa·s. The optimum dosage combination for optimizing performance was determined to be A2B2C3, i.e., 4% SBS, 30% bio-asphalt, and 10% NO. Table 4 suggests that bio-asphalt had the highest correlation with the ductility of WBA. This is because the ductility of WBA was more sensitive to bio-asphalt, registering the highest correlation magnitude of 6.07, consecutively followed by NO (4.60) and SBS (2.37), respectively. Hence, a higher bio-asphalt content in WBA is preferred. By contrary, the viscosity was slightly changed by these additives. Based on the relatively lower correlation magnitudes, Table 4 shows that viscosity was insignificantly affected by the SBS, bio-asphalt, and naphthenic oil additives.

3.3. Apparent Viscosity Test Results of WBA. To meet the WBA fluidity requirements, the apparent viscosity test was used to determine the optimum A2B2C3 dosage. Figure 4 shows that the Brookfield viscosity of the WBA at 120°C was 0.91 Pa·s. When the temperature increased, the slope of the curve became flat with the inflection point occurring at around 120°C, which was selected to determine the Brookfield viscosity of different amounts of the ordinary aged asphalt (i.e., RTFOT-aged). The results were shown in Figure 5.

Considering the volatility of naphthenic oil at 120°C as well as the potential mixing and compaction of high-volume RAP during the construction phase, 120°C was selected as the mixing temperature. As shown in Figure 5, with the addition of RTFOT aged asphalt, the Brookfield viscosity increased, inferring an increase in hardness and decay in fluidity. The loss of the light components in the aged asphalt has an adverse effect on the asphalt’s fluidity performance. NO can dissolve the asphaltenes in the aged asphalt, while light components in bioasphalt can be used as a rejuvenator for the aged asphalt. When the aged asphalt was 70%, the Brookfield viscosity reached 2.23 Pa·s, which meets the Chinese (JTG F40 2004) specification requirements for WMA construction, i.e., the viscosity should not exceed 3 Pa·s [28].

3.4. Characterization of the WBA Rheological Properties. The DSR experiment was used to characterize the high-temperature rheological properties of WBA, original asphalts, and RTFOT-aged asphalts, respectively [29]. The rutting parameter $G' / \sin \delta$ was utilized as an indicator of the high-temperature rheological and antirutting characteristics of asphalt under high-temperature conditions. In this test, the shear rate was 10 rad/s and the test temperature ranged from 40°C to 90°C [30]. The experimental test results are shown in Figures 6 and 7. In the figures, Z40, Z50, Z60, and Z70 means that 40% by weight of total asphalt, etc., of aged asphalt was blended with WBA.

Considering a similar temperature in both Figures 6 and 7, the $G' / \sin \delta$ results are as follows: Z70 > Z60 > Z50 > Z40 > Pen 70# > WBA. The higher the amount of the aged asphalt, the better its high-temperature performance characteristics. According to the performance-grading (PG)
specification requirement for original asphalt \((G^*/\sin \delta \geq t\) 1.0 \(\text{m.kPa})\) and RFTOT-aged asphalt \((G^*/\sin \delta \geq t\) 2.2 \(\text{m.kPa})\), the high-temperature PG classification of WBA from Figures 6 and 7 could be considered to be PG 58-XX [31, 32]. Pen 70\# original asphalt, Z50, and Z40 high-temperature PG classification would be PG 64-XX. And the high-temperature grade for Z70 and Z60 would be PG 70-XX. From the high-temperature rutting resistance perspective, the RFTOT-aged original asphalt Z40, Z50, Z60, and Z70 were found to be significantly better than the Pen 70\# and WBA asphalt as theoretically expected [33].

In general, aged asphalt is more prone to cracking at low temperatures due to the evaporation and degradation of the saturates and aromatic components [17, 22]. Therefore, it is critical to investigate the low-temperature performance of the aged asphalt with WBA.

The magnitude of \(S\) characterizes the strength of the asphalt against deformation under low-temperature conditions [18, 32]. The smaller the value, the smaller the internal stress under the low-temperature environment and the better its flexibility [33, 34]. The \(m\)-value is the slope of the stiffness modulus-time response curve, which is used to characterize the stress relaxation ability of asphalt. Comparing the \(S\) values of Pen 70\# original asphalt and reclaimed asphalt, the rank order of \(S\) superiority was as follows: Z70 > Pen 70\# > Z60 > Z50 > Z40 > WBA. When the amount of aging asphalt was greater than 70%, the \(S\) value of Z70 exceeded that of Pen 70\# original asphalt (Figure 8).

For the given temperature range, the absolute slope (i.e., \(m\)-value) of the stiffness-temperature (ST) curve was in the following rank order of superiority: Z70 > Pen 70\# > Z60 > Z50 > Z40 > WBA (Figure 9). The results indicated that if the proportion of the aged asphalt does not exceed 60%, the low-temperature rheological properties of WBA blended with aged asphalt were superior to the original asphalt. The PG classification, based on the DSR and BBR test results, are shown in Table 5.

### 3.5. Adhesion Characterization

The adhesion test was employed to characterize the adhesion and moisture sensitivity of the asphalts. According to literature reports [19, 35], bioasphalt has obvious shortcomings in terms of insufficient moisture stability. In this study, the rank order of adhesion superiority was found to be as follows: WBA, Z40, Z50, Z60, and Z70, respectively. The test results are shown in Table 6. Similar to the previous figures, Z40, Z50, Z60, and Z70 means that 30% by weight of total asphalt, etc., of aged asphalt, was blended with WBA.

The results of the adhesion experiments in the table show an adhesion grade of 4 for WBA, while the adhesion grades for Z40, Z50, Z60, and Z70 were all 5. The results suggest that the WBA blended with aged asphalt has good moisture-damage resistance performance. Therefore, WBA with the lowest adhesion grade 4 exhibited the best adhesion to aggregates, with moisture stability meeting the Chinese construction specification requirements [20].

### 3.6. Microstructure Characterization

For the orthogonal experimentation, WBA was prepared using the optimal content of SBS, bioasphalt, and NO, respectively. Z40 and Z70 were prepared utilizing WBA blended with 40%, and 70% aged asphalt at 135°C and 1500 rpm for 15 min, respectively.

#### 3.6.1. FT-IR Test Results and Analysis

To analyze the regeneration mechanism of the WBA, the FT-IR test was used to investigate the functional groups of WBA and the original asphalt after RFTOT and pressure aging vessel (PAV). As shown in Figure 10, the sulfide group was at a wavenumber of 1030 cm\(^{-1}\). The aged asphalt had an absorption peak of the sulfide group, whereas the WBA had no obvious sulfide group absorption peaks. The wavenumber
at 1700 cm\(^{-1}\) was attributed to the carbonyl group with an absorption peak of WBA. According to Tauste et al., the carbonyl group has the potential to dilute the sulfoxide group, which is why WBA can improve the performance of RAP. The absorption peak is shown at 2924 cm\(^{-1}\) which was produced by the asymmetric stretching vibration of -CH. The absorption peaks at 1456 cm\(^{-1}\) and 1376 cm\(^{-1}\) were due to the stretching vibration of the methyl groups and -COOH stretching vibrations, respectively.

3.6.2. SEM Test Results and Analysis. The SEM was used to study the microstructure of the WBA and the aged asphalt surfaces, to analyze the repair and regeneration effect of WBA in RAP. Depending on Figure 11, the surface of Pen 70\(^{#}\) original asphalt before RTFOT aging was relatively smooth and uniformly distributed without debris and cracking (Figures 11(a) and 11(b)). After RTFOT aging, the surface of the asphalt appeared to be rough. Owing to the greater pressure of the asphalt during the aging process, fragments with more fracture angles were generated (Figure 11(c)). The surface of the WBA exhibited an intricate network with an interweaving structure, whereas SBS and others were sheared into microscopic particles under high-speed shear [22]. In addition, NO contained many chain structures with saturated branched chains that were grafted on the carbon rings [36]. This made the appearance of the regenerated bioasphalt to exhibit a smooth network structure (Figure 11(d)) [37]. After mixing WBA with the aged asphalt, it was found that the surface of the Z40 and Z70 types became smooth. Also, the surface of Z40 was smoother than Z70. The main reason is that the proportion of WBA in Z40 was higher than that of Z70 so that the damaged parts of the aged asphalt could be repaired easily, as shown in Figures 11(e) and 11(f), respectively. This indicates that the bioasphalt and NO in WBA could well repair the surface damage in the aged asphalt and improve the overall performance of asphalt in RAP.

4. Summary of Conclusions and Recommendations

In this paper, bioasphalt, SBS, and NO recovered from waste industrial products and petroleum asphalt were used to produce an unconventional WBA. Different from traditional rejuvenated agents for aged asphalt, bioasphalt, a cost-effective (i.e., it costs half as much as standard asphalt) and environmentally friendly material was innovatively used as a rejuvenated agent in the study. The key findings, conclusions, and recommendations drawn from this paper are shown as follows:

1. The results of the single-factor and orthogonal tests indicated that the optimal blending amount of bioasphalt in WBA was 30% at 4% SBS and 10% NO, respectively. Additionally, bioasphalt was found to exhibit great sensitivity to the WBA ductility. Naphthenic oil, on the other hand, had the greatest impact on the WBA fluidity, a critical aspect of material constructability.

2. The Brookfield viscosity experiment showed that the fluidity of the WBA and aged asphalt with different
Figure 10: FT-IR test results.

Figure 11: SEM test results. (a, b) The original asphalt before aging. (c) The original asphalt after aging. (d) WBA. (e) Z40. (f) Z70.
contents satisfied the construction requirements. The adhesion test indicated excellent adhesion and good moisture stability properties for WBA, with an adhesion grade of 4. The rheological tests (DSR and BBR) indicated an improvement in both the high-temperature and low-temperature rheological properties of the aged asphalt relative to the unaged Pen 70° original asphalt.

(3) The IR test showed that the carbonyl group in the WBA was diluted and dissolved the sulfoxide group in the aged asphalt, thus revealing the rejuvenation mechanism of bioasphalt. The SEM test results reaffirmed the rejuvenation potential of bioasphalt in WBA through surface morphology characterization.

Overall, this study has provided a foundation’s platform towards using bioasphalt as a rejuvenated agent in WBA, which has great potential used in reclaimed asphalt pavement. However, more laboratory testing of this nature is recommended for future studies to diversely cover a wide spectrum of materials from different sources including field correlations and validations to further add on to these study findings.

Data Availability

The data used to support the findings of this study are included within the article.

Disclosure

The contents of this paper (which is not a standard) reflect the views of the authors who are solely responsible for the facts and accuracy of the data presented herein and do not necessarily reflect the official views or policies of any agency or institution. Trade names were used solely for information purposes and not for product endorsement, advertisement, promotions, or certification.

Conflicts of Interest

The authors declare no conflicts of interest in this study.

Authors’ Contributions

Zhu Zhang was responsible for conceptualization, investigation, methodology, formal analysis, validation, writing the original draft, and reviewing and editing the article. Jiusu Li was responsible for conceptualization, resources collection, investigation, methodology, formal analysis, validation, writing the original draft, reviewing and editing the article, and supervision. Zhengjun Duan carried out the investigation and experimental test. Shuaipeng Zhang was responsible for reviewing and editing the article and experimental test. Menglei Lou conducted the experimental test.

Acknowledgments

Special thanks and due gratitude also go to all those who helped during the study. The authors appreciate the financial support from the Changsha Industrial Science and Technology Commissioner Project (CSKJ2017).

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