Feasibility analysis of bio-binder as non-petroleum alternative for bituminous materials

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Abstract
This study discussed the feasibility of bio binders derived from swine manure as the alternative of bitumen. The bio-oil was converted from swine manure via the fast pyrolysis technology. Subsequently, the bio-oil upgraded to bio binders through a preheating treatment. In terms of the lack of studies regarding the compatibility of bitumen and bio binders, this study respectively investigated the blends of bitumen and bio binders from chemical and mechanical perspectives. Mainly, the chemical characterization consisted of extensive measurements using the Thermo-Gravimetric Analysis (TGA), the Differential Scanning Calorimeter (DSC) and, the Fourier Transform Infrared Spectroscopy (FTIR). As for the mechanical performance, the blends of bitumen and bio binders with a content of 5% and 10% by mass were firstly produced and then subjected to the Dynamic Shear Rheometer (DSR) test. Results indicated that the addition of bio binders could enhance the low-temperature performance of the bitumen to some extent. At the low or medium temperature, the blends showed favorable thermal stability. However, the incompatibility issue is noticeable at high temperatures, especially during the mixing and compaction process in practice.

1. Introduction

As a by-product of the petroleum refining industry, bituminous materials are widely consumed in pavement engineering as a paving material due to its advantages such as easy construction, low tire-road noise, and even environmental potential. Bitumen, accounting for around 4% to 6% of the bituminous mixture by weight, plays a critical role in the service performance and service life of bituminous pavement as bitumen gluing the granular materials together. Nevertheless, due to the non-renewability and the environmental concerns of the petroleum bitumen, demands for the alternatives of bitumen is increasing in recent decades. For this purpose, many researchers made considerable efforts to develop sustainable paving materials that can partly or wholly replace bitumen. In related published studies, binders, or modifiers derived from waste plastic, waste polymers were extensively reported [1]. However, these materials reply on the petrochemicals. On the other side, bio binders attract more interests as it is a petroleum-independent material. In particular, Peralta et al [2] introduced bio-renewable substitutes and defined bio binders as the substance, which was made from non-petroleum-based resources and produced by upgrading bio-oils. A direct alternative binder indicates a 100% replacement of asphalt binders. As for the partial replacement, it refers to extenders or modifiers depending on the ratio of the blending.

In the past two decades, many studies attempted to produce bio binders. In general, the biomass source consists of three categories, which are the plant-based source, bio-oil based source, and human or animal waste. Raouf and Williams [3] claimed that biomasses oak flour, switchgrass, and corn stover were potential materials to produce bio-oils based on fast pyrolysis technology. Peralta et al [4] used red oak to produce bio-oils, and the combination of bio-oils and crumb rubber successfully realized using asphalt-rubber technology. Yang et al [5] verified that the incorporation of bio-oils derived from waste wood resources could improve the mechanical performance bitumen. With regards to bio-oil based sources, Zhang et al [6] modified the bitumen using
bio-based waster oil. Qu et al[7] also reported that waste cooking oil was capable of softening the bitumen. Another interesting study is to derive bio-oil from human or animal waste. Fini et al[8] converted the swine manure to a bio-oil through a hydrothermal process as a sustainable modifier for bituminous binders. You et al [9] applied a similar bio-oil from swine manure in the warm mixed asphalt mixtures and reclaimed asphalt mixtures.

It is not difficult to find that the diversity of biomass sources provides a broad prospect for the development of sustainable paving materials. Currently, extensive studies adopted bio binders to promote a specific service performance of bituminous materials. The investigation on temperature and shear susceptibility of bio-oil derived from plant-based biomass sources demonstrated that the products from fast pyrolysis have to undergo pre-treatment to comply with the Superior Performing Asphalt Pavements (Superpave) specification [3]. The bio-oils produced from switchgrass have similar rheological properties with bitumen, which indicates the potential of bio-oils as the direct alternatives of bitumen [10]. The incorporation of bio-oil derived from waste wood can considerably improve fatigue performance and slightly change the tensile strength of the modified bitumen. However, the effect of bio-oil on the rutting performance of asphalt mixtures was not significant [5]. The adding of waste cooking oil could lead to superior fatigue performance [7]. Moreover, a certain amount of bio-oil would also improve the low-temperature performance of bitumen [6, 8, 11].

Besides for the modifier, the possibility of using the bio binder as the rejuvenator for wet-processed asphalt shingles was examined [12]. The incorporation of bio binders can efficiently reduce the viscosity of aged bitumen and improve the blending workability between the recycled asphalt shingles and the virgin binder. Ji et al [13] evaluated the effectiveness of vegetable oils as rejuvenators for aged bitumen binders. Results showed that the vegetable oil rejuvenators are beneficial for both fatigue and low-temperature cracking resistance.

Although many case studies have proved the potential of bio binders as the alternative for bitumen, existing studies mainly focused on the mechanical properties of modified bitumen; rare studies investigated the role of bio binder inside the modified bitumen from the perspective of both chemical and mechanical perspectives. On the other hand, the absence of recognized and unified production devices and procedures for bio binders obstructs the large-scale promotion. Therefore, this study investigated the bio-oil derived from swine manure concerning the production of bio-oil, up-grading treatment of bio-oil, the incorporation of bio binders into bitumen, and subsequently, the rheological and chemical investigations. Notably, this study intentioned to figure out the role of bio-oils inside the bitumen and its potential application as an alternative for bitumen.

2. Objectives

The study aimed to investigate the bio-based substitute for bitumen concerning production, processing, and performance characterization. Notably, the bio-based alternative used for the research was the swine manure. The swine manure underwent a fast pyrolysis process, after which the product turned into bio binders through an upgrading treatment. Thermo-gravimetric Analysis (TGA), Differential Scanning Calorimeter (DSC), and Fourier Transform Infrared Spectroscopy (FTIR) were applied to characterize the chemical properties of bitumen and bio binder. Based on chemical analysis, the feasibility of bio binder as the alternative for bitumen could be fundamentally investigated. Then, rheological properties of bio binder, bitumen, as well as their blends, were also examined. Blends of bitumen with a content of 5% and 10% of bio binder by mass, are noted as B + 5% bio binder and B + 10% bio binder, respectively.

3. Materials and methodology

3.1. Bio binder production

The swine manure, collected from a local farm in Shanxi, China, was primarily heated for 6 h at 100 °C to evaporate the retained moisture. The dried swine manure was then used as feedstock for pyrolysis with a fluidized bed pyrolysis experimental system, as shown in figure 1. Intuitively, the fluidized bed pyrolysis experimental system consists of a fluidized bed reactor, two cyclones for char separation, a gas supply with the controlling unit, a thermocouple with temperature controlling unit, circulating water with condenser unit, and gas purifying with collecting unit. In practice, the dried swine manure steadily went through a screw feeder. Subsequently, the fast pyrolysis reaction occurred in the reactor at the temperature of 450 °C with a duration of the 2 s. As a result of this, the swine manure separately decomposed into products, including biochar, bio-oil, and gas. The bio-char condensed in char pots in the first place, followed by the bio-oil that retained inside a three-stage condenser. Eventually, the gas was combusted before its emission into the atmosphere.

Three condensers, as shown in figure 1, collected three kinds of bio-oils. However, this study only adopted the product from the first condenser based on preliminary attempts. Further treatment on the bio-oil was composed of continuous heating for 2 h at 110 °C[9].
3.2. Experimental program

Figure 2 illustrates the research framework of this study. As shown in figure 2, the bio binder was blended with the bitumen at 160 °C and the speed of 4000 rpm for 30 min with a mass percentage of 5% and 10%, respectively. After that, the speed slowed down to 1000 rpm to eliminate the inside bubbles. The whole mixing terminated depending on subjective judgment.

As mentioned, this study performed both chemical and rheological tests on the blends of bitumen and bio binders. Chemical tests intended to expound on the compatibility between the bio binder and bitumen. While the rheological analysis mainly figured out the variation of mechanical performance caused by the incorporation of bio binders into bitumen.

3.3. Test methods

3.3.1. Fourier Transform Infrared Spectroscopy (FTIR)

Infrared Spectroscopy is an analytical technique through which the functional groups of organic molecules can be identified [14]. This study used the ATR (Attenuated Total Reflectance) technique, a commonly used infrared spectroscopy procedure, to feature the functional group information. The wavenumber scan range was from 400 to 4000 cm⁻¹ with a resolution of 4 cm⁻¹.
3.3.2. Differential scanning calorimetry (DSC)
The endothermic and exothermic reactions of samples happened as the temperature changed. Differential Scanning Calorimetry can precisely measure the thermal variation by a temperature program during which the sample undergoes the glass transition, melting, and crystallization proceeded [15]. The endothermic or exothermic curves provided with the thermal compatibility information between bitumen and bio binders. Concerning the temperature program, it started with the conditioning temperature at 25 °C, followed by a total of five steps program with a heating rate of 20 K min⁻¹ to ensure a significant appearance of the glass transition. During the interval of steps, a duration time of 15 min for equilibrium was necessary to reach the predetermined temperature.

3.3.3. Thermogravimetric analysis (TGA)
The thermogravimetric analysis describes weight loss or gains as a function of temperature as a given thermal procedure [16]. It is the most common technique which is applied to characterize the thermal and oxidation stability of materials. In this study, a temperature program starting at 20 °C was conducted on the bitumen and bio binder with an increased rate of 10 °C/min until 750 °C.

3.3.4. Dynamic shear rheometer (DSR) test
Dynamic mechanical analysis (DMA) is an efficient method to define the rheological properties of binders within the linear viscoelastic (LVE) material behavior [17]. For this purpose, the DSR carried out a frequency sweep from 0.1 and 10 Hz at different temperatures from 12 to 84 °C (with 12 °C intervals). Parallel plates with a diameter of 25 mm and a gap size of 1 mm were adopted. The complex modulus master curves and phase angle master curves were then constructed based on the Time-Temperature Superposition Principle (TTSP) with the reference temperature of 24 °C (one of the measured temperatures).

3.4. Modelling the rheological results
The characterization of the viscoelastic property of bitumen is a four-dimensional issue regarding strain-stress-time-temperature. Studies have shown that there is an intrinsic relationship between temperature and time [18]. Specifically, the stiffness modulus measured under temperature $T_1$ at time $t_1$ is equivalent to the measured that under temperature $T_2$ at a specific time $t_2$. This phenomenon is called the Time-temperature superposition principle (TTSP), and the mathematical expression is as follows:

$$G(T_1, t_1) = G\left(T_1, \frac{t_1}{\alpha_T}\right)$$

With the aid of TTSP, multiple frequency sweep curves can be shifted into a continuous curve (master curve) through the shift factor $\alpha_T$. Many studies have developed varying approaches to determine the shift factor, including manual movement, WLF, Arrhenius, Log-linear and VTS [19]. In this study, the determination of shift factors relied on the Willian-Landel-Ferry (WLF) formula, as can be seen in equation (2) below [20].

$$\log \alpha_T = -\frac{C_1 \cdot (T - T_{ref})}{C_2 + T - T_{ref}}$$

Where the $T_{ref}$ is the reference temperature, the $C_1$ and $C_2$ are dimensionless parameters. $C_1$ and $C_2$ were flexibly determined depending on the materials to make a continuous and smooth master curve.

Mechanistic-Empirical Pavement Design Guide (MEPDG) recommended the Sigmoidal model to fit the master curve and it mainly describes the rate dependence of the modulus master curve. Mathematically, the Sigmoidal model is as follows:

$$\log|G^*| = \delta + \frac{\alpha}{1 + e^{\beta + \gamma (\log \omega)}}$$

where $\log \omega$ is the reduced log frequency, $\delta$ is the lower asymptote, $\alpha$ is the difference between the values of the upper and lower asymptote, the shape between the asymptotes and the location of the inflection point is dependent on $\beta$ and $\gamma$, respectively.

Kramers-König equation correlated the master curves for complex modulus and phase angle. [21]. Specifically, it reveals the relation between the real and imaginary parts of complex modulus as follows:

$$E'(\omega) = \frac{1}{\pi} P \int_{-\infty}^{+\infty} \frac{x_1(\omega^*)}{\omega^* - \omega} d\omega^*$$

$$E''(\omega) = \frac{1}{\pi} P \int_{-\infty}^{+\infty} \frac{x_2(\omega^*)}{\omega^* - \omega} d\omega^*$$
Where $P$ indicates the Cauchy principal value, $\omega^*$ represents the complex angular frequency. Kramers-Konig equation deduces the complex modulus and phase angle, respectively, as follows.

\[
\log|E^*(\omega)| - \log|E^*(\infty)| = -\frac{2}{\pi} \int_0^\infty \frac{u \cdot \delta(u) - \omega \cdot \delta(\omega)}{u^2 - \omega^2} \, du
\]

(6)

\[
\delta(\omega) = \frac{2}{\pi} \int_0^\infty \frac{\log|E^*(\omega)| - \log|E^*(\infty)|}{u^2 - \omega^2} \, du
\]

(7)

Based on the above two equations, the relationship between complex modulus and phase angle was defined as follows:

\[
\delta(\omega) \approx \frac{\pi}{2} \int_0^\infty \frac{d}{d\log(\omega)} \log|E^*(\omega)|
\]

(8)

The optimization process proceeded through the Solver function in Excel. The process minimized the sum of square error (SSE) between the measured and model data considering the complex modulus and phase angle at the same time [22]. The coefficient of determination $R^2$ evaluated the goodness-of-fit to guarantee the reliability of all these fitting works in this study. The definition of $R^2$ is as follows:

\[
SSE = \sum \frac{(\log|G_{exp}(f, T)| - \log|G_{model}(f, T)|)^2}{(\log|G_{exp}(f, T)|)^2} + \sum \frac{(\log|\delta_{exp}(f, T)| - \log|\delta_{model}(f, T)|)^2}{(\log|\delta_{exp}(f, T)|)^2}
\]

(9)

\[
R^2 = 1 - \frac{(n - q)}{(n - 1)} \times \left(\frac{S_e}{S_y}\right)^2
\]

(10)

Where $n$ indicates sample size, $q$ is the number of independent variables in the model. $S_e$ and $S_y$ represent the standard error of estimation and the standard error of deviation, respectively.

4. Results and discussion

4.1. Feasibility analysis from a functional group perspective

In general, the durability of bimanous materials is associated with aging exposing to the environment factors [23]. Airey [24] concluded that the mechanisms of bitumen aging ascribed three perspectives. Namely the volatilization of light components during the construction, oxidation of polar molecules such as resin, and the steric aging hardening. The oxidation of polar molecules, to a great extent, depends on the chemical compositions and specific functional groups. In this case, the difference between base bitumen and bio binders in terms of functional groups could deduce aging-related information.

Figure 3 shows the FTIR results of the base bitumen and the bio binder. Some typical functional group characteristic peaks were summarized, as seen in table 1 and in particular, four kinds of characteristic peaks related to the performance of bitumen. The bio binder had similar function groups with base bitumen. Therefore, the compatibility of these two matters is not a significant concern when mixing them. Bio binder possesses more alcohol groups, which could be the interpretation of the retained bound water even after dehydration treatment, as alcohol groups resulted in quite an amount of hydrogen bonds.

The dominate variation between two substances happened in the low-band region, and base bitumen contained more unsaturated alkane groups. During the short-term aging process, the unsaturated alkane groups change into saturated alkane groups by oxidation. Therefore, bio binder reduced aging resistance capacity due to
its light components and functional groups. Accordingly, the duration should be paid more attention to in the process of the replacement of bio binders.

### 4.2. Feasibility analysis from a thermal stability perspective

One of the most crucial concerns of bio binder application, similar to most composite materials research, is the compatibility of bitumen and bio binders and their performance variation with temperature. Standing on this point, it deserves to notice that the interaction contributes to the performance of blends in some situations, especially at a high replacement rate of bio binder. Hence, it is of necessity to figure out how the interactions, either between bio binder ‘particles’ or the bio binder and bitumen, proceeding with the variance in blending temperature and replacement rate of bio binder. Thermodynamic analysis and differential scanning calorimetry analysis are two of the most convincing approaches to characterize the thermal stability of a substance. Therefore, TGA and DSC measurements, in this study, were carried out on the bio binder and bitumen, aiming to seek the thermal compatibility of two materials.

Figures 4 and 5 depict the DSC and TGA results for base bitumen and bio binders, respectively. As seen from figure 4, the base bitumen had a phase transition at a temperature of \(-21.8^\circ C\), and it conformed to the normal range of the glassy modulus of unmodified bitumen materials. The bio binder presented a lower glass transition temperature than that of base bitumen with a value of \(-26.3^\circ C\). As the temperature increased, base bitumen achieved another transition temperature at 39.8 \(^\circ C\), and similarly, for the bio binder, the value of transition temperature was 35.7 \(^\circ C\). In the practical pavement engineering, bio binders inside the bitumen would melt before the bitumen when the surface temperature of the road reached over 35.7 \(^\circ C\). It is beneficial for the rutting resistance of pavement because the bio binder can absorb a part of heat, resembling the impact of the wax. However, the asynchronous melting point can induce some other issues, for instance, the weak interface between bitumen and bio binders from the micro perspective.

It is worth noting that when the temperature rises around 70 \(^\circ C\), the fluctuation for the bio binder ascribed to the retained bound water, although the bio binder was dehydrated and pre-treated during the production process. It demonstrated that the potential instability of the blend, and it should be noticeable in the practical construction.

The thermal stability of bio binders started to degrade at around 90 \(^\circ C\), as seen from figure 5. It indicated that the light component inside the bio binder volatilized around 90 \(^\circ C\). On the other hand, the thermal stability of the base bitumen is insignificant.

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![Figure 4. DSC results for base bitumen and bio binder.](image)

### Table 1. Functional group characteristic peaks for base bitumen and bio binder.

| Characteristic peaks | \(-\text{C}=\text{O}\) | \(-\text{C}-\text{OH}\) | \(-\text{CH}_2\) | \(-\text{CH}_3\) |
|---------------------|----------------|----------------|---------------|---------------|
| Wavenumber/cm\(^{-1}\) | 1700 | 1270 | 1460, 2930, 2850 | 1620 |

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4.3. Rheological properties of bio binder modified bitumen

Figure 6 presents the complex modulus and phase angle for base bitumen and their blends with bio binders. Three levels of measured temperature in terms of low (12 °C), medium (24 °C), and high (36 °C) were separately compared, as shown in Figure 6 in case of the arbitrary evaluation for a single temperature condition.

The incorporation of 5% bio binders dramatically softened the based bitumen while the viscoelastic property was not substantially changed (resemble phase angles). Interestingly, the complex modulus of the blend binder B + 10% rebounded slightly, and a considerable descent of phase angle occurred. In the case of low temperature (12 °C), the low phase angle raised a better performance of resistance to thermal-induced cracks, and accordingly, the demand for the bio binders should be over 5% by mass.

Complex moduli and phase angles of bio binders at temperatures of 36 °C and 60 °C complied with a similar tendency when the blend percentages of bio binders varied from 0% to 10%. The addition of bio binders efficiently changed the stiffness of blends, especially the percentage of bio binders increasing from 5% to 10%. Besides, a downward trend for phase angle was observed, nevertheless, with a tiny difference. In this case, the addition of bio binders can significantly soften the base bitumen and alter the viscoelastic property of bitumen delicately.

At a given level of temperature, the impact of bio binder depends on the adopted temperature or frequency. Thus, inappropriate conditioned temperatures might have an unexpected influence on bio binder. Black diagram, known as the origin presentation of rheological performance for thermal-simple-viscoelastic materials at a full temperature or frequency range, was adopted to make up for the weakness of a single indicator [25].

Figure 7 shows the complex modulus as a function of the percentage of added bio binders. As the increase of measured temperature, the correlation coefficient gradually increased. It illustrated that the change of complex modulus with the percentage of bio binder complied with the linear model at a high measured temperature. However, the complex modulus performed poor linear relation with the percentage of added bio binder at a low measured temperature.
Figure 8 shows the black diagrams for base bitumen with the blends of bitumen and bio binders. In general, the addition of bio binders is supposed to change the black diagram of base bitumen towards softer and more elastic trends. With the increase of temperature, complex modulus of the binder continuously decreased with phase angle increased, which complied with the behavior of conventional thermal-simple-viscoelastic materials. Whereas, the inflection of the black diagram appeared at the high-temperature region for B + 5% bio binder and B + 10% bio binder. Generally, the inflection phenomena indicated an asynchronous transition of two or more composite materials. Hence, the incompatibility of base bitumen and bio binder at a high temperature can contribute to the incipient fault of either the storage or construction concerns with great potential.

The black diagrams of B + 5% bio binder and B + 10% bio binder intersected with each other instead of monotone transformation. It is with a high possibility that, though only 5% variation compared to 100% percentage replacement, the roles of bio binders inside the base bitumen are substantially different in terms of the rheological perspective.

The provided black diagram presents a continuous and smooth curve. Moreover, the time-temperature superposition principle (TTSP) is allowed for the construction of the master curve. Therefore, the Sigmoidal and the Kramer-Konig relation were adopted to construct the master curves of complex modulus and phase angle simultaneously, as shown in figures 9 and 10. Table 2 lists the parameters of the sigmoidal model and the WLF formula. $C_1$ and $C_2$ for B + 5% bio binders showed abnormally high value. However, it was the calculated result for the better fitting result but not the critical point for this discussion.

Figure 9 compares the master curve of three samples with different addition. Apparent decrease of glassy modulus in the high-frequency area indicates that the softening effect was with higher identification capacity than that in the low-frequency area. Meanwhile, the base bitumen blended with a percentage of 5% bio binder had the lowest glassy modulus. Given the bio binder was dispersed in the base bitumen homogenously, a low replacement rate can lead to a ‘particle’ distribution form. In this case, volume displacement was the only interpretation for the softening effect. However, once the replacement rate reached a certain amount, the particle form disappeared due to the interaction between particles. Consequently, aggregation of bio binders or even its network will form inside the bitumen. Therefore, there exists a slight stiffness rebound when the replacement rate was 10%. Unfortunately, the so-called function of the interaction effect cannot be activated all the time. The weak interaction was negligible as the temperature increase, as can be seen in figure 9, which
Table 2. Rheological modeling parameters for bio binder modified bitumen.

| Samples               | Sigmoidal Model | WLF       |
|-----------------------|-----------------|-----------|
|                       | $\delta$  | $\alpha$ | $\beta$ | $\gamma$ | $C_1$ | $C_2$ |
| Base bitumen (B)      | -2.15    | 11.79    | -0.53   | -0.33    | -15.29 | 137.78 |
| B + 5% bio binder     | -1.98    | 10.78    | -0.76   | -0.37    | -1.35E 09 | 1.62E 10 |
| B + 10% bio binder    | -1.14    | 10.03    | -0.54   | -0.38    | -13.99 | 124.65 |

5. Conclusions

Although extensive studies have claimed the merits of using the bio binder as the bitumen alternative from varying perspectives, up to now, there is still a lack of discussion focusing on the material difference and its accompanying performance issues. This study investigated the feasibility of applying the bio binder into the bitumen from both chemical and rheological perspectives. Some vital findings are listed below:

1) The thermal aging of bitumen and bio binder blends should be paid much attention since the light components in the bio binder and alcohol groups can be easily oxidized.
(2) The addition of bio binder into the bitumen enhanced its low-temperature performance, as the bio binder presented a lower glass transition temperature. Meanwhile, although some variance existed, similar thermal susceptibility between bitumen and bio binder indicated that the compatibility issues are not significant within the service temperatures.

(3) The addition of bio binder was supposed to change the bitumen towards softer and more elastic as well as the temperature susceptibility. On the other hand, the incompatibility of base bitumen and bio binder at a high temperature can contribute to the incipient fault of either the storage or construction concerns from the rheological perspective.

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