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# Properties of asphalt binder modified by bio-oil derived from waste cooking oil

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#### HIGHLIGHTS

- A kind of bio-oil derived from waste cooking oil is used as asphalt modifier.
- Chemical compositions of bio-oil and control asphalt are investigated.
- Bio-oil can reduce the deformation resistance of control asphalt.
- Bio-oil can improve the stress relaxation property of control asphalt.
- Bio-oil and control asphalt have good compatibility under static heated storage.

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#### ABSTRACT

The properties of asphalt binder modified by bio-oil derived from waste cooking oil were researched. Firstly, four components separation test and fourier transform infrared spectroscopy (FT-IR) test were carried out to investigate the chemical compositions of experimental materials. Then the physical and mechanical behaviors of bio-oil modified asphalt binders were studied by penetration grading tests, rotational viscosity (RV) test, separation tendency test, dynamic shear rheometer (DSR) test and bending beam rheometer (BBR) test. Four components separation test results indicate that bio-oil is mainly composed of aromatics, resins and saturates. Almost no chemical reactions between bio-oil and control asphalt are noticed through FT-IR test results. The addition of bio-oil decreases the softening point and viscosity, increases the penetration and ductility of control asphalt binder. Separation tendency test results demonstrate that bio-oil and control asphalt have good compatibility under static heated storage conditions. Furthermore, the addition of bio-oil decreases the complex modulus and creep stiffness, increases the phase angle and m-value of asphalt binder, which means that adding bio-oil could reduce the deformation resistance and elastic recovery performance of control asphalt, and improve the stress relaxation property and also helps to the improvement of thermal cracking resistance of control asphalt. © 2015 Elsevier Ltd. All rights reserved.

#### 1. Introduction

Petroleum asphalt, which is the by-product of petroleum refining, is commonly used in pavement construction. But petroleum is increasingly scarce as a non-renewable resource, resulting in the short supply of petroleum asphalt. Finding asphalt substitute is a new way to resolve the problem. Utilizing some waste materials from other industries to partially substitute asphalt, can not only reduce asphalt consumption but also improve the pavement performance of asphalt. This will also have great economic and

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http://dx.doi.org/10.1016/j.conbuildmat.2015.10.173 0950-0618/© 2015 Elsevier Ltd. All rights reserved. social significance to resources recycling and sustainable development.

In recent years, many researchers were looking for the substitute or modifier of traditional asphalt binder. The potential applications of alternative binders or modifiers, such as bio-binder from microalgae, fractionated bio-oil, cotton seed oil, crude glycerol, organo montmorillonite nanoclay and so on, have been investigated [1–8]. Yang et al. [9] also studied the performance of asphalt binder partially substituted by a kind of bio-oil, which was obtained by waste wood fast pyrolysis. Their works indicated that the addition of bio-oil increased the high temperature performance of asphalt binder, reduced the mixing temperature of the asphalt mixture, and it had adverse effects on the medium and low temperature performance of the asphalt binder. Fini et al.





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[10,11] investigated the performance of asphalt binder modified by bio-binder, which was produced from the thermochemical conversion of swine manure. The test results showed that the addition of bio-binder improved the low temperature properties, but it decreased the high temperature grade of asphalt binder. Peralta et al. [12,13] prepared bio-binder by fast pyrolysis of plant residue. It was found that this bio-binder could be used as modifier, additive or antioxidant of asphalt binder. The basic performance of bio-binder met the specification requirements for asphalt binder, and the incorporation of rubber could significantly improve the bio-binder properties. Although numerous researchers produced asphalt substitutes or modifiers from biomass materials, few of them focused on waste oil products [14–17]. In China, there is a great amount of waste cooking oil produced every year, which is believed more than 5 million tons. In recent years, biodiesel were obtained from waste cooking oil through alkaline catalysis procedure [18–20]. However, about 10% bio-oil by-product was coproduced during the biodiesel production [6]. This bio-oil is black viscous liquid mixing with glycerin and soap, which has been thought as a potential asphalt or asphalt modifier substitute.

In this paper, this bio-oil was used as asphalt modifier, and the properties of bio-oil modified asphalt had been researched to investigate the effect of bio-oil on the asphalt binder performance. Four components separation test, FT-IR test, viscosity grading tests, separation tendency test, DSR test and BBR test were conducted to primarily study the effect of bio-oil on the chemical, physical and mechanical behaviors of asphalt binder. Different from normal PG grading test conditions, the effect of aging was not included in this study during the DSR and BBR tests.

#### 2. Materials and experimental methods

#### 2.1. Asphalt binder

In this study, 40/60 penetration grade asphalt from Jilin province of China was used as control asphalt. Its basic properties were shown in Table 1, which all met the requirements of Chinese specification (JTG F40-2004). The heating speed in softening point test is 5 °C/min, and the stretching speed in ductility test is 5 cm/min.

#### 2.2. Bio-oil

The bio-oil used in this study is black oily liquid, which is the by-product of waste cooking oil refining for biodiesel. As shown in Fig. 1, in the process of producing bio-diesel from waste cooking oil, free fatty acids in waste cooking oil were firstly converted into fatty acid methyl ester by methanol with the action of sulfuric acid catalyst, which process is called pre-esterification. Then the fatty acid glycerides in waste cooking oil were converted into fatty acid methyl ester by methanol with the action of potassium hydroxide catalyst, which process is called transesterification. Fatty acid methyl ester is the main component of bio-diesel, and the residue during these procedures is the bio-oil used in this study. Before adding bio-oil into the control asphalt, the physical properties of bio-oil were investigated. Specific, the moisture content of bio-oil is 3.1% by weight, and the density at 15 °C is 0.95 g/cm<sup>3</sup>. In addition, the rotational viscosity of bio-oil at room temperature (25 °C) is determined to be 146.3 mPa s. As the pre-esterification and transesterification process may introduce acid and alkali into the original mixtures, the pH value of bio-oil was also measured, which was found to be 6.1. Therefore the biooil was almost neutral.

#### Table 1

Properties of 40/60 penetration grade asphalt.

#### 2.3. Materials preparation

At first, control asphalt was uniformly heated in a temperature-regulated heating mantle and continuously stirred using a high shear mixer. Then, 0%, 2%, 4%, 6%and 8% bio-oil were added into control asphalt by weight when test temperature reached 135 °C, and then blended for 40 min by high shear mixer with a speed of 5000 r/min to achieve a homogeneous mixing state. The IDs of asphalt binders used in this study were presented in Table 2.

#### 2.4. Experimental methods

Firstly, four components separation test and FT-IR test were carried out to analyze the chemical properties of BP and AMB. Then, the physical and mechanical behaviors of AMB were studied with penetration grading tests, RV test, separation tendency test, DSR test and BBR test. The experimental methods used in this study referred to Chinese specification (JTG E20-2011). The flowchart of this study was presented in Fig. 2.

#### 2.4.1. Four components separation test

According to T0618, four components separation tests were conducted to analyze the chemical components differences between bio-oil and control asphalt. In the test process, 0.5–1.0 g sample was dissolved in n-heptane. In this procedure, asphaltenes were firstly separated. The n-heptane soluble fractions were poured into vitreous adsorption column filled with active alumina, then the saturates, aromatics and resins were separated using n-heptane, toluene and toluene/ethanol (1:1 by volume) [21,22].

#### 2.4.2. Fourier transform infrared spectroscopy (FT-IR) test

FT-IR tests were performed to analyze the functional groups differences between BP and AMB. Test samples were dissolved in carbon disulfide with 5 wt. % concentration, dropped onto KBr table and controlled the thickness of the film to be appropriately 150  $\mu$ m, then scanned at 32 times with test spectrum range from 400 to 4000 cm<sup>-1</sup> [16].

#### 2.4.3. Penetration, softening point and ductility tests

According to T0604, the penetration tests were used to assess the hardness of asphalt materials. In the test process, a container filled with asphalt sample was stored in 25 °C water bath for 90 min, and then penetrated by a needle weighted 100 g, the penetration depth was measured as penetration in the unit of 0.1 mm.

According to T0606, the softening point tests were used to identify the temperatures at which phase change occurred in asphalt materials. In the test process, two steel balls were placed on the horizontal disks of asphalt sample contained in vertically supported metal rings. The assembly was heated in water bath at 5 °C/min. The softening point was recorded as the average temperature at which the two disks softened enough to allow each ball, enveloped in asphalt, to fall a distance of 25 mm (1.0 in). It was also known as the ring and ball softening temperature ( $T_{rsb}$ ) [23].

According to T0605, the ductility tests were used to measure the stretching length of standard asphalt sample before breaking under standard testing condition (5 cm/min stretching speed at 15 °C). It is usually considered that asphalt binder with low ductility has poor low temperature performance in service.

#### 2.4.4. Rotational viscosity (RV) test

According to T0625, Brookfield viscometer was used in rotational viscosity (RV) test to measure the flowing resistance of asphalt materials. In the test process, a cylindrical spindle with specific diameter and effective length rotates inside a container filled with asphalt binder at restricted speed. In this research, the test temperature was 60 °C.

Properties		Units	Requirements	Test results	Test methods
Penetration @25 °C		0.1 mm	40-60	43.0	T0604
Softening point		°C	≥49	52.3	T0606
Ductility @15 °C		cm	≥30	150	T0605
Flashing point		°C	≥260	310	T0611
Density @15 °C		g/cm <sup>3</sup>	-	1.13	T0603
Wax content		%	≤2.2	1.7	T0615
After short-term aged (RTFOT)	Mass loss	%	≤±0.8	0.75	T0610
	Retained penetration ratio @25 °C	%	≥63	70.2	
	Retained ductility @15 °C	cm	≥10	110	

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