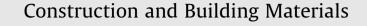
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### Viscous properties, storage stability and their relationships with microstructure of tire scrap rubber modified asphalt



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Ming Liang<sup>a</sup>, Xue Xin<sup>a</sup>, Weiyu Fan<sup>a,\*</sup>, Huadong Sun<sup>a</sup>, Yan Yao<sup>a</sup>, Baodong Xing<sup>b</sup>

<sup>a</sup> State Key Laboratory of Heavy Oil Processing, China University of Petroleum, Qingdao, Shandong Province 266580, PR China <sup>b</sup> China National Offshore Oil Corp. (Qingdao) Heavy Oil Processing Engineering Technology Research Center, Qingdao 266580, PR China

#### HIGHLIGHTS

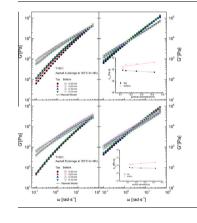
- The relationship of microstructure with viscous behavior and storage stability is uncovered.
- The flow behavior tends to turn into non-Newtonian fluid with increasing particle size.
- The features of dispersed rubber particles in asphalt are elongated or strip-type.
- Viscosity increase with particle size owing to ratio of length and diameter increasing.

#### A R T I C L E I N F O

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#### ABSTRACT

Recycled-polymer modified asphalt has been extensively used in road construction, especially the recycled tire scrap rubber-modified asphalt (TSRMA). However, the main problem during the application of TSRMA is poor storage stability which finally affects the service performance of the pavement. The objective of this work is to evaluate viscous properties, storage stability and morphology of TSRMA and to reveal the relationship of microstructure with rheological behavior and storage stability. With this aim, two different penetration grade asphalts were modified by rubber particles with various mean diameter in a four-paddle mixer at 170 °C. Steady state flow measurements, frequency sweep tests in linear viscoelastic region, storage stability tests as well as fluorescence microscopy were carried out on mixes. Rheological evaluation reveals that the addition of tire scrap rubber to asphalt lead to a significant increase in viscosity at 60 °C, improving rutting or permanent deformation resistance. Furthermore, the increase in particle size causes an enhancement in viscosity and the steady flow behavior tend to turn into non-Newtonian fluid with increasing particle size and temperature. Storage tests presented that the viscosity of samples obtained from the bottom section of a tube are always higher and the difference also becomes significant as the increase of rubber particles size, tending to poor stability. Fluorescence microscopy shows that dispersed particles in asphalt are the elongated features or strip-type and the aspect ratio (length/diameter) increases with particle size. In addition, smaller particle tend to be unobvious in matrix under microscopy view, indicating improved compatibility between rubber and asphalt. © 2014 Published by Elsevier Ltd.

\* Corresponding author. Tel.: +86 15853256892.

*E-mail addresses:* liangmingupc@126.com (M. Liang), xinxue19910103@163. com (X. Xin), fanwyu@upc.edu.cn (W. Fan), sunhuadongupc@163.com (H. Sun), yaoyanupc@163.com (Y. Yao), xingbaodongupc@163.com (B. Xing).

#### 1. Introduction

Asphalt, almost exclusively derived from vacuum distillation of petroleum, has been broadly applied in road construction and roofing field as a binder, thanks to its adhesion to other material and viscoelastic properties [1,2]. It can be primarily classified as straight-run asphalt and oxidized asphalt according to manufacturing. Additional processes obtaining asphalt include solvent deasphalting, blending and visbreaking [1,3,4]. In terms of composition, asphalt is generally separated into saturates, aromatics, resins and asphaltenes, known as SARA fractions, according to their solubility in non-polar, aromatic or polar solvents [5–10]. The proportion and chemical feature of SARA fractions remarkably affect the viscoelastic behavior of asphalt and then impact the service performance on road pavement.

Nowadays, with the increased traffic loads and adverse atmospheric effects, several types of pavement distresses (permanent deformation in high temperature region and crack prone in the low temperature region) exist in road pavement during their operative time, which is related to the rheological properties of asphalt [11–14]. Thus, adding polymers are a beneficial way to enhance the quality and performance of asphalt reducing above-mentioned distresses and maintenance costs. Virgin polymers used as the modifier for asphalt can be categories as thermoplastics, thermosets, thermoplastic elastomers and rubber [15–19]. Concerning on the application of recycled polymers as replacement for virgin polymers has grown in recent years which are an environmentally friendly way to dispose of wastes.

On the other hand, literatures pointed out that about 14 million vehicle tires are discarded per year in China and only about 7 million are reused [20,21]. The remaining waste tires are added to stockpiles and landfills, giving rise to environmental problems. Therefore, the use of tire scrap rubber as an additive to asphalt can not only endow asphalt with improved elasticity but also be an effective way to solve a waste disposal problem. In general, tire scrap rubber is applied to manufacture tire scrap rubber modified asphalt (TSRMA) by two distinct approaches (dry method and wet method) [22–25]. The dry method mixes tire scrap rubber with mineral aggregate before incorporating asphalt. On the contrary, in a wet method, tire scrap tire rubber and asphalt mixed together at elevated temperatures for several hours to produce asphaltrubber which in turn is introduced to the mixture. The second method generates an enhancement in viscosity because of the chemical and physical reaction between asphalt and rubber particles.

Tire scrap rubber modified asphalt has several advantages such as the increased viscosity and softening point, improved penetration index and preferable low-temperature ductility, which means superior rutting resistance at the high temperatures, intensive crack resistance at the low temperatures and fatigue resistance of road pavement [26,27]. Numerous literatures reported that preparation conditions and characters of rubber particles as well as asphalt are crucial factors in order to obtain the desired properties of TSRMA, including tire scrap rubber particles diameters, processing temperatures, blending time, chemical or physical properties of matrix asphalt and so on [28-31]. Navarro et al. [32] studied the effects of rubber particles on linear viscoelastic behavior and thermo-rheological properties in the medium temperature region. They claimed that viscoelastic functions increase with larger particle size due to non-spherical particles and also emphasized that particle sizes lower than 0.35 mm to modify asphalt are recommended for the manufacturing operations [33].

Although the large number of papers [19–27] focused on conventional properties, manufacturing process, aging properties of tire scrap rubber modified asphalt as well as microstructure, a few studies have been reported on how the characters of rubber particles affect the properties of tire scrap rubber modified asphalt. Furthermore, these reports for the most part focused on conventional properties testing with limited scopes and did not reveal the relationship of microstructure with conventional properties, aging properties, rheological properties, etc. Therefore, the objective of this work is to evaluate viscous properties, storage stability and morphology of TSRMA and to reveal the relationship of microstructure with rheological behavior and storage stability. In order to achieve this aim, TSRMA was prepared by two different grade matrix asphalts and rubber with various particle sizes, which in turn was subjected to viscous measurements, frequency sweep tests in the linear viscoelastic region, storage stability tests and fluorescence microscopy. The neat asphalt as well as aged asphalt went through process techniques was also investigated.

#### 2. Experiment and methods

#### 2.1. Materials

Two different grade asphalts (A and B) obtained from CNOOC (China National Offshore Oil Corp.) were used as matrix for tire scrap rubber modification. The conventional tests were carried out so as to evaluate the properties and composition of base asphalt, including penetration (ASTM D5 [34]), softening point (ASTM D13 [35]), ductility (ASTM D113 [36]) and SARA fractions (ASTM D4124 [37]). The conventional characterization results of base asphalt were displayed in Table 1. Furthermore, the colloidal index was also calculated to identify a discrepancy of chemical composition of base asphalt. Tire scrap rubber was made from waste tires by ambient grinding method and the moisture content was 0.1% and the ash content was 8%. Four different tire scrap rubber with a mean diameter of 0.15, 0.18, 0.25, 0.42 mm were obtained via screening.

#### 2.2. Preparation of TSRMA

TRSMA, whose rubber content was 9% by weight, was prepared in a four-blade impeller (IKA-20) made in Germany. Quantified base asphalt was primary heated in a cylindrical vessel to 170 °C and then tire scrap rubber was added into asphalt matrix at a rotating speed of 1200 rpm. Samples were stirred for 3 h at 170 °C and in turn analyzed. Being compared with the TSRMA, a blank sample also underwent the process conditions (called aged asphalt).

#### 2.3. Steady state flow tests

Steady state flow tests were conducted in a controlled-stress rheometer AR2000ex from USA with parallel plate geometry (25 mm diameters, 1 and 2 mm gap). Steady flow behavior of mixes were evaluated in a wide shear rate and temperature range ( $10^{-3}$ – $10^{2}$  s<sup>-1</sup>, 10–100 °C). Two replicates of specimen needed to be done to obtain the accordant results.

#### 2.4. Fluorescence microscopy

The state of tire rubber particles within asphalt matrix are observed by fluorescence microscopy. A small amount of molten samples were first loaded between two glass slides, which were then squashed carefully. Then, thin glass slide with samples were observed at room temperature under optical microscopy of Olympus BX51 (Japan) equipped with DP72 digital camera. Fluorescence images were analyzed by Cellsens Standard and magnified 100 times.

Properties and compositions of base asphalt.

Properties and composition	Asphalt A	Asphalt B
Penetration (0.1 mm)	63	93
Softening point (°C)	48.5	43.4
Ductility (25 °C) (cm)	>100	>100
Saturates (%)	19.07	15.29
Aromatics (%)	39.85	39.79
Resins (%)	27.19	32.91
Asphaltenes (%)	14.17	12.01
Colloidal index	0.50	0.37

Colloidal index (CI) = (saturates + asphaltenes)/(aromatics + resins).

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