



## Usage of slurry oil for the preparation of crumb-rubber-modified asphalt emulsions



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### HIGHLIGHTS

- Progress to develop modified asphalt emulsions by using crumb rubber (CR).
- A method to prepare CR modified asphalt emulsions.
- Effect of FCC slurry oil on the emulsification characters and product properties.
- Characterization and discussion from routine tests and DSR measurements.

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### ABSTRACT

Due to the increased performance-related requirements on the products from aqueous asphalt emulsions, scrap tire crumb rubber (CR) has been successfully applied as a modifier. An approach to prepare CR-modified asphalt emulsions with satisfactory emulsifying characters and resultant product properties has been studied. In this paper, microwave pretreated CR was first reacted with asphalt materials under optimum conditions. Next, the CR/asphalt mixture (CRMA) was blended with slurry oil (decant slurry oil) from a fluid catalytic cracking process (FCC) in refineries to form an emulsifiable asphalt binder. In this work, 3% slurry oil content in this CRMA was demonstrated to be sufficient for the emulsification with the addition of water and an emulsifier. Under the optimum environmental factors to prepare the emulsions, as exemplified in this work, the resultant CRMA is capable of being well emulsified. The asphalt emulsions and related products produced in this work are able to meet the requirements. The effects of CR and slurry oil are studied by using the results from routine tests and dynamic shear rheometer (DSR) measurements. The results revealed that asphalt binders, obtained after these asphalt emulsions are broken, have improved road performance at high temperature, compared to that from the original asphalt. The consistency has also been increased. The CR powder asphalt modifier is an alternative to virgin polymer in the preparation of modified asphalt emulsions. The CR powder has significant environmental and economic advantages. A method to directly use slurry oil in a large amount was developed. The results in this work pave the way to prepare CR and slurry oil-modified asphalt emulsions.

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### 1. Introduction

The development of the automotive industry may produce approximately 1000 million tires worldwide every year [1]. This number is still rapidly increasing in this decade; hence, the disposal of scrap tires has become a serious issue in solid waste management. The only ideal market with a high potential for expansion to solve the scrap tire pollution problem is recognized to be the development of crumb-rubber-modified asphalt binders (CRMA) because asphalt pavements are also extensively produced every

year [2]. Compared to neat asphalt, CRMA has many benefits, including road resistance to rutting, fatigue cracking, and thermal cracking, which are required to meet the challenge of increasing traffic loads in varying climate environments. CRMA has also been proved in practice to be a very effective and economical alternative to polymer-modified asphalt (PMA).

In many countries, a basic transport network has already been constructed and the work for preventative maintenance of existing roadways to extend their service time has been of vital importance. To meet this challenge, many new technologies, including chip or slurry seal, cold regeneration and micro-surfacing, are rapidly developed. All of these technologies must use asphalt emulsions, which can be easily handled at low temperature. The use of asphalt

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emulsion also provides the advantages of fuel savings and environmental protection. In addition, binders from modified asphalt emulsions have higher consistency to provide the thickness and adhesion ability on the road, and their temperature susceptibility and rutting resistance may also be improved. At present, rubber latex is always added to asphalt emulsions as a modifier to produce modified asphalt emulsions. When solid CR is used as an alternative to rubber latex, CRMA should first be produced to prepare CRMA emulsions. It is known that CRMA is very resistant to being dispersed into tiny droplets, mainly because of its high viscosity [3]. Therefore, very few papers addressing this issue have been published in the literature [4,5].

In this decade, many researchers started making every effort to prepare aqueous CR-containing asphalt emulsions because of both environmental and economic requirements. Some patents have already been issued to declare their results for the production of CRMA emulsions (E-CRMA). These methods can be divided into two approaches. In the first approach, some additives are blended with the CR/asphalt mixture during the interaction. These additives include hydrocarbon oil, dodecyl-benzene, and *p*-toluene sulfonic acids or polyphosphoric acids. Hydrocarbon oil is known to accelerate both the absorption of the aromatic components into the CR polymeric chains and the chemical digestion of the rubber into the asphalt [3,6]. Dodecyl-benzene sulfonic acid (DDBSA) stimulates CR de-vulcanization and the reaction between the de-vulcanized CR and the double bonds of the asphalt [7]. In addition, polyphosphoric acid can be used to react with many functional groups of the asphalt and breaks the asphaltene agglomerates [8,9]. All of the above reactions are advantageous for decreasing the CRMA viscosity in the emulsification process. Of course, the negative effect of residual acids on the resultant de-emulsification, when the emulsion is in service, must be considered because cationic emulsifiers have now been widely used. In this option, CR is often in the range of 5–10% CRMA in these patents. These products have been proved to meet the current specification requirements for some of the slurry or chip seal asphalt pavements and crack fillers. Therefore, CRMA with a CR content of 6% as a representative example was prepared in this paper. The other approach is to prepare emulsions that have “incorporated” CR break-down products in hot asphalt to form “liquefied” rubber-modified asphalt binders with a very low solid particle content (only 2% or less). For this purpose, the interaction between asphalt and CR particles must be performed at very high temperatures of up to 260 °C under specialized conditions, which requires the use of sophisticated and expensive equipment. In some patents, this resultant CRMA is further treated with DDBSA to lower the viscosity [10,11]. Afterward, the CRMA is then combined with emulsifying solutions to prepare the CRMA emulsion (E-CRMA). In the second approach, the CR content is permitted to reach 25% or more in the CRMA binders, which are able to meet more specific requirements.

In the USA, 40% of gasoline is produced via the FCC process in petroleum refineries, and this ratio reaches up to 70% in China. Of course, a large amount of catalytic cracker bottom sludge (slurry oil) is also produced simultaneously [12]. It is difficult to subsequently use the slurry oil due to its poly-aromatic structure and entrained catalyst fines. The removal of such solid fines from slurry oil is a time-consuming effort because the incoming stream of the catalytic cracker slurry oil contains a catalyst fine level of 0.1–0.5% by weight, which must be reduced to 0.05% for subsequent product use. On the contrary, slurry oil can be directly used to prepare CRMA emulsions.

In this paper, CR is pretreated by microwave irradiation for devulcanization and cross-linking cleavage. Interactions between pretreated rubber and asphalt are then performed under higher temperatures with high shearing mixing. In addition, the CRMA is then treated with FCC slurry oil (slurry oil) to lower the viscosity

and modify the colloid structure, so that it meets the necessary requirement to prepare emulsions. Emulsification is then performed by adding aqueous emulsifying solutions containing emulsifiers and other ingredients. In this paper, the properties and visco-elastic behaviors of the related CRMA materials and the products, obtained after the emulsion is broken, are also studied via the results of DSR measurements and routine tests. Residues from evaporation in the laboratory are used as a representative for characterization of the latter products because a mild evaporation condition is selected to keep them unchanged. In addition, this paper also highlights the effect of the FCC slurry oil.

## 2. Experimental section

### 2.1. Starting materials

To prepare aqueous emulsions of CRMA that can be used for paving and sealing applications, CR from truck tires was used because it contains larger percentages of natural rubber in rubber hydrocarbons, which exhibit more decomposition into the asphalt matrix than synthetic rubber. The CR from truck tires was shredded and pulverized at ambient temperature. The particles that passed through the 80-mesh (175- $\mu\text{m}$ ) sieve were used in this work. All of the CR samples were taken from one batch to maintain the same properties of the CR starting material. The composition of the CR sample was measured using thermogravimetric analysis (TGA). The results are listed in Table 1. The CR content in each of the CRMA binders in this paper was kept at 6%.

One type of 90 penetration grade AH-90 heavy traffic asphalt in domestic use was selected as the base asphalt. As observed in Table 2, this asphalt binder contains a high concentration of aromatics and resins and is considered to be an ideal feed to produce CRMA emulsions (E-CRMA).

FCC slurry oil was taken from an FCC unit in a refinery. Some of the results of the routine tests and the SARA analysis are also listed in Table 2. The slurry oil was found to have a lower viscosity and to contain a high percentage of aromatics with very few asphaltene contents. Additionally, 60–80% of these aromatic components were reported to be poly-aromatic structures of primarily 3–4 cycles. It can hence be predicted that the addition of slurry oil to CRMA is able to modify the CRMA colloidal structure and decrease the viscosity.

### 2.2. Preparation of slurry oil-treated CRMA and their emulsions

Forty grams of CR powder was treated in a 480-watt microwave oven for 2–10 min and then added to the asphalt. The CR/asphalt mixture (CRMA) was preheated to 170–200 °C and mixed and sheared at that temperature for 2–4 h using a high shearing mixer. This product was designated as CRMA. Next, FCC slurry oil was added and stirred for 15 min at 190 °C. The resultant CRMA was designated as CRMA-*n*, where the number *n* indicates the slurry oil content. CRMA is the same material as CRMA-0. Afterward, CRMA-*n* was emulsified. The aqueous emulsions of CRMA-*n* are designated as E-CRMA-*n*.

### 2.3. CRMA sample or E-CRMA characterization

#### 2.3.1. Visco-elastic analysis

The complex shear modulus ( $G^*$ ) and the phase angle ( $\delta$ ) have been reported to allow prediction of the rheological behavior of an asphalt binder [13]. Therefore, asphalt dynamic shear rheometer (DSR) measurements are performed to provide the measurement of both  $G^*$  and  $\delta$  of the base asphalt and CRMA binders. In this test, a sinusoidal shear stress is applied to an asphalt sample that is sandwiched between two parallel plates. The gap between these two plates is controlled to be 2 mm for CRMA. The resulting sinusoidal shear stress is monitored as a function of frequency at 45 °C [14]. The ratio of the peak stress to the peak strain is defined as  $G'$ . The phase difference between the stress and strain in an oscillatory deformation is defined as  $\delta$ .

#### 2.3.2. Viscous flow behaviors

A conventional rotational viscometer, which has often been used for asphalt binders near the softening temperature, was selected for the measurement of viscosity. The viscometer contains a cylinder that rotates coaxially inside one other static cylinder containing the asphalt sample. The material between the inner and outer cylinders is therefore analogous to a thin asphalt film found in a sliding plate

**Table 1**

Composition of the CR sample.

	Volatile components	Rubber hydrocarbons	Carbon black	Inorganic fillers
Percentage (%)	2.3	54	40.3	3.4

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