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## Effect of mineral fillers adsorption on rheological and chemical properties of asphalt binder

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Bitumen polar fractions preferentially adsorb on the surface of mineral fillers.

Bitumen is 0.3–0.85 and 5–8 times stiffer close to and away from mineral interface.

Magnitude of bitumen adsorption is dictated by specific surface area of fillers.

#### article info

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### **ABSTRACT**

An asphalt mastic imparts most of its characteristics to the asphalt concrete mixture and also dictates several forms of distresses in asphalt mixtures and pavements. Several studies have demonstrated that the interaction between asphalt binder and mineral fillers has a significant impact on the properties and performance of asphalt mastics and mixtures. The objective of this study was to investigate the nature of binder adsorption on mineral filler surface while simultaneously quantifying the influence of such adsorption on the properties of the binder in the immediate vicinity of the interface and bulk. An adsorption test using mineral fillers and binders was conducted to achieve this goal along with measurements of asphaltene content and rheology on the original binder and the residual binder from the adsorption tests. Results show that polar fractions preferentially adhered to the surface of the mineral filler. Such preferential adsorption resulted in a significant increase in the complex modulus of the adsorbed or fixed asphalt with a concomitant decrease in the complex modulus of the free or bulk asphalt binder. These changes in complex modulus varied only slightly as a function of the frequency. The magnitude of adsorption was dictated by the mineral nature of the surface and more importantly by the specific surface area of the particles. The findings from this study are useful to better understand and model the failure mechanisms in the micro structure of asphalt composites.

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#### 1. Introduction and Background

Asphalt mastic is typically regarded as a matrix that binds aggregate particles together in an asphalt mixture. As such, the properties of the mastic have a strong influence on the properties of the asphalt mixture. In the context of this paper, asphalt mastic is defined as the mixture of asphalt binder and filler particles (particles finer than  $75 \mu m$  or passing ASTM standard sieve number 200). In practice, the filler to binder ratio in a mastic is controlled based on weight or volumetric requirements. For example, the

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original Superpave mix design method recommends the filler to binder ratio (by weight) to be anywhere from 0.6 to 1.2. European standards (EN 1097-4) call for measurement and control of the Rigden voids in the filler while selecting fillers and designing mixes. Interestingly, in contrast to practice, research studies have shown that in addition to volumetrics, the physicochemical interactions between the filler particles and the binder are equally important in dictating the performance of the mastic and consequently that of the mix  $[1-4]$ . Some of these studies are briefly described below.

#### 1.1. Role of fillers in dictating the properties of mastic

Anderson et al. [\[5\]](#page--1-0) found that different fillers resulted in different amount of stiffening when added to asphalt. The extent







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of stiffening could not be explained solely on the basis of filler gradation. Other researchers also proposed that the effects of the physicochemical interactions must be studied carefully to better understand the influence of fillers on the performance of the mastic [\[6,7\]](#page--1-0). Petersen et al. [\[7\]](#page--1-0) used steady state and dynamic test methods to quantify the stiffening effects of active fillers such as hydrated lime and showed that there were significant interactive effects between the asphalt binders and hydrated lime that can affect aging as well as rheological properties.

More recently, Wang et al. [\[8\]](#page--1-0) investigated the effect of mineral filler characteristics on the permanent deformation characteristics of asphalt mastics and mixtures. They found that asphalt mastic performance was significantly affected by the fractional voids in the filler and possibly by the CaO content and fineness modulus. This effect, however, varied with the type of binder indicating the possibility of binder-filler interactions. Hesami et al. [\[9\]](#page--1-0) used a combination of numerical and experimental approaches to study the effects of interfacial interaction between asphalt binder and fillers on mastics. They arrived at a conclusion that was similar to the previous study, i.e. the shape and size of the filler particles, and the interfacial interaction between the filler and the binder had a significant influence on the properties of the composite.

Several studies have tried to better understand and quantify the influence of such interfacial interaction on the performance of the mastic. For example, Tan and Guo  $[10,11]$  studied the mechanism of interfacial interaction between asphalt binder and mineral fillers by using physical and chemical test methods (e.g. dynamic mechanics analysis - DMA, differential scanning calorimetry – DSC, and Fourier transform infrared spectroscopy – FTIR) and micro/nano scale imaging techniques (e.g. atomic force microscope – AFM). They found that the impact of physicochemical interactions was significant and that such interactions were dictated by the specific surface area of fillers.

In another similar study, Miljkovic and Radenberg demonstrated that the interfacial interaction between the binder and the sand in the localised contact regions influenced the fracture behavior of the bitumen emulsion mortar mixtures [\[12\].](#page--1-0)

#### 1.2. Nature of interactions at the binder-filler interface

While the aforementioned studies demonstrate the importance of binder-filler interaction on the overall or macroscopic properties of the mastic and consequently on the properties of the mixture, some studies have also investigated the nature of these interactions at the interface based on binder chemistry. For example, the adsorption of asphalt components on mineral fillers was investigated by many researchers. These studies showed that the polar components, such as asphaltenes, are preferentially adsorbed on the surface of the mineral filler  $[13-15]$ . Curtis et al.  $[13]$  studied the adsorption of asphalt model components (carboxylic acids, sulfoxides, phenol, pyridines, pyrrolics and ketones) representing the key asphaltic functional groups. They found that a competition between the functional groups existed towards mineral filler surface, and generally the polar functions adsorbed to a higher extent compared to nonpolar groups. Within the polar groups the sulfoxides, carboxylic acids, pyridines and phenols are the most adsorbed components.

The nature of interactions at the interface of mineral aggregates (or particles) and organic molecules has also been closely studied by researchers working on flow of crude oil. For example, Acevedo et al. [\[16\]](#page--1-0) reported that the asphaltenes dissolved in toluene were capable of creating a monolayer or a multilayer on the inorganic substrate. They also reported that the adsorption of resins dissolved in toluene on the silica was insignificant. Marczewski and Szymula [\[17\]](#page--1-0) studied the adsorption of asphaltene dissolved in toluene on rock minerals, including natural Brazilian quartz, dolomite, calcite and kaolin, as well as pure oxides:  $Fe<sub>2</sub>O<sub>3</sub>$  and  $TiO<sub>2</sub>$ . Their adsorption measurements showed that although very often adsorption of asphaltenes on minerals was described as ''Langmuir type", it was in fact quite distant from such a simple model. Saraji et al. <a>[\[18\]](#page--1-0)</a> measured the asphaltene adsorption in porous media under flow conditions using the ultraviolet visible spectrophotometry. They reported that the adsorption depended on the mineral type; Calcite showed higher adsorbance compared to quartz and dolomite.

Similar adsorption studies have also been conducted using asphalt binders. For example, [\[19\]](#page--1-0) found that different functional groups have different affinities towards mineral filler, and the Langmuir and Freundlich isotherm can be used to describe adsorption. The Langmuir isotherms are based on the assumption that a monolayer forms at the interface while Freundlich isotherms are not constrained by any such assumption. Also the Langmuir adsorption model explains adsorption by assuming an adsorbate behaves as an ideal gas at isothermal conditions. Freundlich isotherms are typically used for multisite adsorption on rough surfaces. [\[20\]](#page--1-0) studied the effect of asphaltenes/maltenes on adsorption. They found that the addition of the asphaltenes increased the amount of binder adsorbed, while the addition of maltenes reduced the amount of bitumen adsorbed. [\[10\]](#page--1-0) conducted a net adsorption test to study the adsorption type of asphalt binder on mineral fillers. They found that the type of adsorption isotherm that they observed corresponded to multimolecular layer adsorption. Fig. 1 illustrates the most common types of adsorption isotherms. Type I isotherm shown in Fig. 1 corresponds to monolayer adsorption typically seen in chemical adsorption. Adsorption Types II-V are multi-layer adsorption isotherms typically observed with physical adsorption of molecules on solid surfaces.

#### 1.3. Motivation and objective

In summary, there is significant amount of work done to understand the effect of fillers on the mechanical property of asphalt mastics as a composite. However, most of these studies have focused on the final outcome or overall behavior of the mastics without explicitly investigating the mechanisms of the fillerbinder interface that dictate the overall behavior. Other studies have investigated the detailed mechanisms of adsorption of organic molecules from crude oil or asphalt binder onto mineral aggregate surfaces. This study was motivated by the need to bridge the gap between these two streams of knowledge, i.e. not only to



Fig. 1. Types of adsorption isotherm representing the relationship between adsorbing capacity (a) and relative pressure  $(P/P<sub>0</sub>)$ . (Type I corresponds to monolayer adsorption, while types II-V correspond to multi-molecular layer adsorption; adapted from [\[10\]\)](#page--1-0).

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