



Development of a method to determine surface energy components of mineral fillers



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HIGHLIGHTS

- Developed a new model for mineral fillers wicking a probe liquid.
- Measured mass of probe liquids rising into filler samples.
- Determined spreading pressure of probe liquids on mineral fillers.
- Compared surface energies of mineral fillers with those of coarse aggregates.
- Demonstrated influences of geometric characteristics on aggregate surface energy.

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ABSTRACT

The surface energy components of the mineral fillers in asphalt mixtures are of significant importance to the fatigue and healing properties of the asphalt mastic. However, the Washburn equation did not apply to mineral fillers when using the column wicking method because mineral fillers can hardly form a non-zero contact angle with any probe liquid. This paper developed a new model for mineral fillers wicking a probe liquid; this model established a relationship among the mass of the wicked liquid, the wicking time and the spreading pressure of the probe liquid on the mineral fillers. Based on the developed model, an experimental protocol was designed to determine the spreading pressure of three probe liquids on five types of mineral fillers, respectively. The Gravimetric Sorption Analyzer was employed to perform the vapor adsorption tests to determine the spreading pressure of toluene on each type of mineral fillers; the Tensiometer System was utilized to measure the mass of probe liquids rising into the filler samples, respectively. Based on the test results, the spreading pressure of every probe liquid on each filler sample was determined, which was then used to solve for the surface energy components of the mineral fillers.

The surface energy components of the fillers were compared with those of the coarse aggregates from the same quarry. Significant variations were identified in each component between the fillers and the corresponding coarse aggregates, which demonstrated the importance of the geometric characteristics of the aggregates to their surface energy components. Furthermore, an asphalt binder was selected to evaluate its adhesive bond energies with the fillers and the corresponding coarse aggregates, respectively. With the same asphalt binder, the nonpolar components of the fillers were smaller than those of the corresponding coarse aggregates, while the polar components and the total adhesive bond energies of the fillers were larger than those of the corresponding coarse aggregates. The differences among the components of the cohesive bond energy and the adhesive bond energies suggested that cracking and healing per unit length would need different energies in the three media, asphalt film, asphalt mastic and asphalt-aggregate interface. These differences would significantly influence the cracking development in asphalt mixtures because cracks would always grow along the easiest path that would need the lowest energy to create crack surfaces.

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1. Introduction

The surface energy components of aggregates are proved to be critical to the fatigue properties and moisture susceptibility of asphalt mixtures [1–8]. When measuring the surface energy

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components of aggregates, the samples of the aggregates were usually taken from the aggregate fraction between the 2.36 mm and 4.75 mm sieve sizes [7–11]. The measured surface energy components of these coarse aggregate samples were then utilized to determine the adhesive bond energies between aggregates and asphalt binders and to further evaluate the fracture, healing and moisture damage of asphalt mixtures. However, the surface energy components of coarse aggregates may significantly differ from those of the corresponding mineral fillers whose surface crystal structure has been altered during the fabrication [10,11]. Therefore, it is critical to determine the surface energy components of mineral fillers separately, which pass No. 200 sieve with a sieve size of 0.075 mm, in order to investigate the performance of the asphalt mastic that is composed of the asphalt binder and the mineral fillers [12–14].

The column wicking method, which is also known as the capillary rise method, is a commonly used approach to measure the surface energy of powders [15–20]. When using the wicking method, the powders are uniformly packed in a column that dips into a known probe liquid, as illustrated in Fig. 1. The height of the probe liquid rising in the capillary channel is continuously measured at consecutive time points. The Washburn equation is then applied to determining the contact angle of the probe liquid with respect to the powder surface, as shown in Eq. (1) [21]:

$$\frac{h^2}{t} = \frac{R_e \gamma_L \cos \theta}{2\eta} \quad (1)$$

where h = height that the probe liquid reaches by capillary rise in time t ; t = test time; R_e = effective capillary radius; γ_L = surface energy of the probe liquid; θ = contact angle of the probe liquid with respect to the powder surface; and η = dynamic viscosity of the probe liquid.

Usually the parameter R_e is firstly determined by using a low-energy probe liquid that spreads over the powder surface so that $\theta = 0$ ($\cos \theta = 1$). With a known R_e , three other probe liquids are selected subsequently, each of which is able to form a non-zero contact angle with the powder surface, to perform the wicking experiments, respectively. Based on the measurements in the wicking experiments, the non-zero contact angle of every probe liquid with respect to the powder surface is then obtained using Eq. (1) [16–20]. The determined contact angles are then substituted into the Young-Dupre equation (see Eq. (2)) to establish a set of three linear equations [22,23]. Solving this set of linear equations, the surface energy components are therefore determined for the powders.

$$(1 + \cos \theta_i) \gamma_{Li} = 2 \left(\sqrt{\gamma_S^{LW} \gamma_{Li}^{LW}} + \sqrt{\gamma_S^+ \gamma_{Li}^+} + \sqrt{\gamma_S^- \gamma_{Li}^-} \right) \quad (2)$$

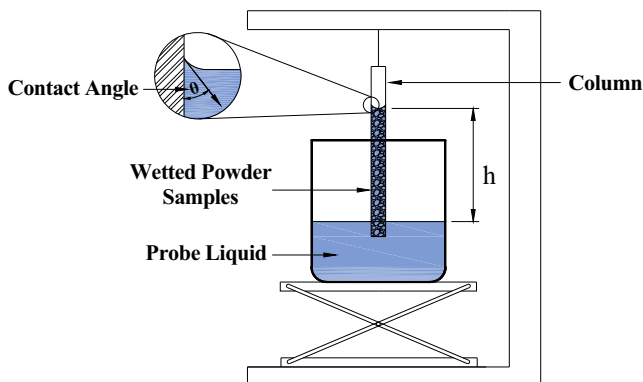


Fig. 1. Configuration of column wicking method.

where θ_i = contact angle between the i th probe liquid and the powder surface; γ_{Li} = surface energy of the i th probe liquid; γ_S^{LW} = Lifshitz-van der Waals component of the powders; γ_{Li}^{LW} = Lifshitz-van der Waals component of the i th probe liquid; γ_S^+ = Lewis acid component of the powders; γ_{Li}^+ = Lewis acid component of the i th probe liquid; γ_S^- = Lewis base component of the powders; and γ_{Li}^- = Lewis base component of the i th probe liquid.

The successful applications of the Washburn equation and the Young-Dupre equation require an essential prerequisite, which is that at least three probe liquids are able to form non-zero contact angles with the powder surface. If none of the selected probe liquid has a non-zero contact angle with respect to the powder surface, the above procedure can never successfully solve for the three surface energy components of the powders. Unfortunately, mineral fillers used for asphalt mixtures are exactly such powder particles that can hardly form a non-zero contact angle with any probe liquid. Even a high-energy probe liquid, such as distilled water, is not able to form a non-zero contact angle with the surface of the mineral fillers.

This fact was confirmed by the authors using the Drop Shape Analyzer (DSA) system. As shown in Fig. 2, a droplet of distilled water was released from a micro syringe onto the surface of a diabase filler sample. The sample was prepared by packing diabase fillers tightly in a metal dish with a diameter of 12 mm, which was then scraped by a metal spatula to make the sample surface smooth. Fig. 3 shows the samples of five types of mineral fillers selected in this study. A 2 μ L droplet was released through the micro syringe at a rate of 10 μ L/min to create a pendant drop, which was positioned approximately 20 mm above the surface of the sample. The sample was then moved up slowly until the bottom of the droplet reached the sample surface. The entire process was filmed using a digital camera equipped with the DSA system. Fig. 4 presents the images of the droplet and the sample recorded at six consecutive time points, which clearly demonstrate that the droplet of distilled water quickly spread over the tightly-packed diabase fillers without establishing a visible contact angle. The same procedure was repeated using distilled water, formamide, toluene, methyl propyl ketone (MPK) and n-hexane on the prepared samples of amphibolite, diabase, granite, gravel and limestone, respectively. No matter which probe liquid was selected, complete wetting was observed once the droplet reached the surface of the sample prepared using whichever type of mineral fillers.

Therefore, a new approach is urgently needed for the determination of the surface energy components of mineral fillers that are completely wetted by probe liquids. To address this research need, this study focused on the development of such a new approach with taking into account the spreading pressure of the probe liquid on the surface of the mineral fillers. The next section details the derivation of the theoretical formulation supporting the new test method. The succeeding section describes the test config-

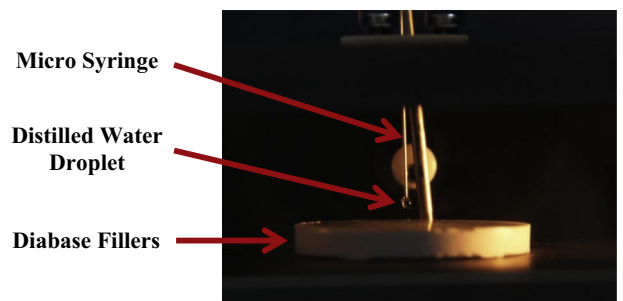


Fig. 2. Configuration of releasing a liquid droplet onto mineral fillers using Drop Shape Analyzer (DSA) system.

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