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An Overview of Conventional and Surface Free Energy Methods Used to Determine Asphalt Aggregate Adhesion

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ABSTRACT

Asphalt aggregate adhesion strength is the fundamental property that determines the moisture damage of asphalt pavement. Moisture damage is related to loss of stiffness or strength as a result of cohesive and adhesive failure of the pavement material. The conventional test for asphalt aggregate adhesion is only comparative and does not provide a quantitative value. Currently research is being carried out to introduce surface free energy measurements as fundamental means to quantify asphalt aggregate adhesion and cohesion. Some of the methods developed to measure surface free energy are Sessile Drop Method, Wilhelmy Plate Method, Adsorption Method, Inverse Gas Chromatography, and Micro-calorimeter. This could eventually lead to the development of a cost effective, accurate and reliable surface free energy measurement instrument which can be used by field engineers.

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INTRODUCTION

The adhesion or bond between asphalt and aggregate is one of the most important criteria for a highly durable and water damage resistant asphalt pavement. However the asphalt aggregate adhesion is one of the least understood bonds partly due to the complexity of asphalt composition and partly due to the variety of aggregate used in asphalt pavement.

The conventional test method for asphalt aggregate adhesion is primarily aimed at comparative classification and does not measure fundamental adhesion strength. Typically, in the conventional test methods for asphalt aggregate adhesion, the aggregate is coated with asphalt and immersed in water under controlled condition and the effect of stripping determined after a period of time. The different method only differ on the type of specimen used, the conditions under which the sample is immersed in water and the method used to assess the degree of stripping (Kim Y. *et al*, 2012).

Promising results have been reported in the literature about the application of surface free energy (SFE) approach to evaluate moisture-induced damage potential of asphalt mixes. For example, Bhasin *et al*. (2007; 2006) suggested different combinations of SFE parameters including work of adhesion, work of debonding, work of cohesion, and specific surface area of aggregates to describe the moisture susceptibility of an asphalt binder-aggregate system as a single value. In another study, Cheng *et al*. (2002) utilized the SFE approach to calculate the work of adhesion and free energy of cohesion for different asphalt binders and aggregates with and without the presence of water. Their results were consistent with those obtained from the accelerated moisture-induced damage tests on mixes. In a recent study, Arabani *et al*. (2012) reported a significant correlation between moisture-induced damage potential of WMA mixes based on SFE and ratio of conditioned to unconditioned dynamic modulus of asphalt mixes.

The SFE can be used to compute both the *physical adhesion* of asphalt–aggregate systems and the loss of this physical adhesion due to the presence of water (i.e., debonding) at the asphalt–aggregate interface. As discussed by Bhasin A, 2006, physical adhesion is probably the adhesion component (over the chemical interactions and mechanical interlocking) that predominantly

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contributes to the overall adhesion of the asphalt–aggregate systems. Although large differences in SFE and physical adhesion have been previously reported, respectively, for aggregates of different mineralogy Kim RY, 2009 and for different aggregate–asphalt combinations Bhasin A, 2006.

Conventional Adhesion Test:

The categories of conventional adhesion test are:

- Static immersion test
- Dynamic immersion test
- Chemical immersion test
- Immersion mechanical test
- Absorption test
- Impact test
- Pull-off test

Static Immersion tests:

This is the simplest form of test which entails the aggregate being coated with asphalt and then immersed in water for a specified period after which the degree of stripping is estimated visually. This method is very subjective and hence has very poor reproducibility. Further positive laboratory performance of this test does not necessarily reflect positive results in the field.

Dynamic Immersion tests:

This test is similar to the Static Immersion test but for this test the immersed test sample is mechanically agitated. Again the degree is stripping is estimated visually and reproducibility is poor.

Chemical Immersion tests:

Aggregate coated with asphalt is boiled in various concentrations of sodium carbonate solution until first sign of stripping is observed. The concentration of sodium carbonate when first sign of stripping is observed is used as a measure the adhesion. This test does not reflect real life situation and is unlikely to accurately predict field performance.

Immersion Mechanical tests:

This test involves the measurement of degree of change of mechanical property of a compacted asphalt sample before and after immersion in water. Some of the mechanical properties used include shear strength, flexural strength and compressive strength. Some of the common tests under this category are the Retained Marshall Stability test, Retained Stiffness test and Retained Cantabro test.

Immersion trafficking tests:

This test consists of a specimen immersed in water bath and traversed by a loaded reciprocating solid rubber tire. Three samples prepared using the standard mould are tracked using a 20kg load at 25 cycles per minute at a water temperature of 40 degrees Celsius until failure occurs, for the standard method.

Adsorption tests:

This test was developed by The Strategic Highways Research Program (SHRP) in the United States, combined measurement of asphalt aggregate adhesion with a measure of moisture sensitivity (Curtis C.W. *et al* 1993).

Impact Test:

The two impact tests which can be used to determine the adhesion properties of asphalt are the Vialit pendulum test and the Vialit plate test.

The Vialit pendulum test consists of a metal block secured to a steel plate using the asphalt to be tested. The metal block is then placed in the path of a pendulum. When the pendulum is released it impacts the metal block and the rebound distance of the pendulum is taken as a measure of the cohesion of the asphalt tested. The greater the cohesion of the asphalt the lesser the distance of the pendulum travel after impact.

The Vialit plate test basically consist of a steel plate coated with asphalt and aggregated particles are pressed onto this plate. The plate is then turned upside down and a steel ball is dropped on the reverse side. The number of impact and number of aggregate particles detached is used as a measure of performance. Visual examination of the detached aggregate particles can at most instances determine the type of failure. A wide range of variables subjected to a wide range of conditioning procedure can be assessed using this test.

Pull-off tests:

Asphalt adhesion can be tested using different types of pull-off test. Two common type of pull-off test are Instron pull-off test and limpet pull-off test. In the Instron pull-off test, the Instron tensile test rig is used to extract aggregate sample from containers of asphalt under controlled laboratory conditions.

The limpet pull-off test is used to determine the bond strength between the aggregate of a surface dressing and the underlying surface. A 50mm diameter metal plate is attached to the pavement surface and the maximum force to achieve pull-off is determined.

The Pneumatic Adhesion Tensile Testing Instrument (PATTI) is a newly developed, simple and inexpensive pull out test utilizing pneumatic pressure to determine the adhesion properties of asphalt.

Surface Free Energy Method:

Molecules in the bulk of the material have higher bond energy compared to molecules at the surface because molecules in the bulk of the material are surrounded on all sides by other molecules. Work must be done in order to remove molecules form the bulk and create new surface molecules with excess energy. The definition of surface free energy is "the work required to create unit area of a new surface of the material in vacuum. Surface free energy is commonly indicated by γ.

One of the widely used theory to explain surface free energy is the Good-Van Oss-Chaudhury theory. This theory enables us to calculate the free energy of adhesion between two materials, provided that the SFE characteristics of the materials are known (Hefer *et al*. 2005). According to this theory the total surface free energy is divided into the following three components.

1) Lifshitz-van der Waals non-polar component, γ^{LW}
2) Lewis acid component,γ^{+}
3) Lewis base component, γ^{-}

The total surface free energy is given by equation (1).

$$\gamma^{total} = \gamma^{LW} + 2\sqrt{\gamma^{+}\gamma^{-}} \qquad (1)$$

There are two possible locations for the beginning of failure of asphalt mixture, through the bulk of the asphalt binder or along the asphalt binder and aggregate interface. The energy required for breaking the bond through the bulk of the asphalt binder is known as cohesive bond energy or work of cohesion. This cohesive bond energy (ΔG_{coh}) is twice the total surface free energy of the material and given by the equation (Eq.2).

$$\Delta G_{coh} = 2\gamma^{total} \qquad (2)$$

The energy required for breaking the bond along the asphalt binder and aggregate interface is known as the adhesive bond energy or work of adhesion. The adhesive bond energy (ΔG_{adh}) is a function incorporating the surface free energy of the asphalt binder and aggregate and is given by equation (Eq.3).

$$\Delta G_{adh} = 2\sqrt{\gamma_A^{LW}\gamma_S^{LW}} + 2\sqrt{\gamma_A^{+}\gamma_S^{-}} + 2\sqrt{\gamma_A^{-}\gamma_S^{+}} \qquad (3)$$

Where subscript (A) denotes asphalt binder and (S) the Substrate or aggregate. The higher the value of the cohesive or adhesive bond energy the larger the amount of energy required to break the cohesive and adhesive bond.

When water is located at the asphalt-aggregate interface, the energy required for the crack to form is a result of the combination of asphalt, aggregate and water surface free energy. The energy required for water to displace asphalt from aggregate is always less than zero and is a thermodynamically favorable process.

The energy required for water to displace asphalt (ΔG_{ASW}^{a}) is given by equation (EQ.4).

$$\Delta G_{ASW}^{a} = \gamma_{AW} + \gamma_{SW} - \gamma_{AS} \qquad (4)$$

The energy parameter ER, first reported by Bhasin *et al* is used to determine the susceptibility of asphalt-aggregate bond to water damage. A higher ER value indicates a higher resistance to water damage. ER is a function of the surface energies of water, asphalt and aggregate.

$$ER = \frac{\Delta G_{adh} - \Delta G_{coh}}{\Delta G_{ASW}^{a}} \qquad (5)$$

Based on the work by Cheng *et al* (2002), Hefer *et al*. (2005) and Bhasin and Little (2009), a detailed methodology has been developed to measure the surface free energy components of asphalt using the Wilhelmy Plate method and the surface free energy components of aggregate using the Universal Sorption Device (USD).

Methods Of Measurement Of Surface Free Energy:

Sessile Drop Method:

This test principle is based on measurement of contact angle of probe liquid with the surface of test sample. This method requires the capturing of the image of the probe liquid dispensed on the solid surface of the test sample. This test is suitable for low energy polymers with smooth flat surface. The expected surface free energy of the test sample must be less than the surface free energy of the probe liquid.

Wilhelmy Plate Method:

The Wilhelmy plate method is based on the principle of measuring the contact angles between sample coated slides and probe liquid with known surface free energy. This test is suitable for low energy surfaces such as polymers. The test sample must be prepared as a smooth surface on a suitable slide. The expected surface free energy of the test sample must be less than the surface free energy of the probe liquid.

Adsorption Method:

The adsorption isotherm of solid with vapor of probe liquid is used to calculate the surface area of the solid and the spreading pressure of the vapor with the solid surface. This test is suitable for high surface free energy solids having sufficient quantity to provide enough surface area for adsorption that can be precisely measured by the instruments. Samples must be prepared to ensure all physical molecules are removed from the sample surface. This test is very time consuming. The Universal Sorption Device (USD) is the test setup used to determine the spreading pressure of three probe vapors on the aggregate surface. The probe vapor spreading pressure is determined by measuring the full adsorption isotherm of the probe vapor on the aggregate surface. The surface energy of the aggregate is then calculated from the vapor spreading pressure.

Inverse Gas Chromatography:

This test measures the retention time and volume for vapors of probe liquid as it reacts with the solid sample. This test is suitable for high and low energy solids. The concentration of probe vapor is very low and therefore the vapor reacts with only high energy spots on the test solid surface and therefore yield higher values of surface free energy compared to other methods.

Micro-calorimeter:

This test measures the enthalpy of immersion of solid in probe liquid. The entropy component is taken into account by making suitable approximations or taking measurements at different temperatures. This test method is suitable for high energy solids. Sufficient specific surface area of the sample is required to generate heat of immersion that can be measured precisely using a micro-calorimeter. The test is much faster than the adsorption measurements but the effect of entropy must be accommodated before determining the surface free energy components from work of adhesion equations.

Discussions:

Current adhesion measurement practice for asphalt-aggregate consist of measuring the physical properties of the entire mix. However this physical property measurement is not an absolute measurement and is only a comparative measurement between samples. The three bonding mechanisms, physical adhesion, chemical interaction and mechanical interlocking, that can be used to explain the adhesion and debonding between asphalt and aggregate, however these three bonding mechanisms cannot be distinguished by the physical properties measured in the conventional adhesion measurement practice.

Surface free energy is a material property that can be used to quantify the dry work of adhesion and work of debonding, which can in turn be used to estimate the moisture sensitivity of asphalt mixes. However due to the small values of surface free energy measured, the sensitivity of the test method must be high in order to derive meaningful measurement. Further measurements must be made in a control environment in order to eliminate any errors caused by external factors. Test setup also differs based on the physical state of the test sample and the estimated magnitude of the surface free energy measured.

The surface free energy measures the physical adhesion between asphalt and aggregate similarly the chemical interactions should also be considered especially in situations where chemically active minerals such as limestone are used or when active fillers or liquid anti-strip agents are added to the asphalt. The micro-calorimeter can be used to measure the total interaction energy between asphalt and aggregate.

Conclusion:

Conventional adhesion or moisture sensitivity test based on physical properties measurements have the advantage that they quantify moisture sensitivity of the asphalt mix by taking into account the cumulative effect of material properties, mixture design parameters and environmental conditions. Despite these advantages and popularity these tests suffer from drawbacks due to poor correlation with field performance, requirement of extensive test time, lack of measurement of material properties related to the mechanism that cause moisture-induced damage, and inability to explain causes of good or poor performance of an asphalt mix. (Arabani.M,& Hamedi.G..H, 2011).

Surface free energy measurement is able to measure the fundamental physical property of asphalt and aggregate to form bond which is one of the factors contributing to the adhesion strength. Using surface free energy measurement, we are able to classify suitability of asphalt and aggregate to form strong bond and measure its moisture damage potential without resorting to trial and error method. This can eliminate premature failure of asphalt pavement due to compatibility issues between asphalt and aggregate. However due to the complexity and sensitivity of the surface energy measurement methods extreme care should be taken to ensure measurements are accurate and reliable. Further due to the difference in the assumptions made in most SFE calculation methods, values for various material may be compared only when the SFE measurement and calculation are performed in the same method (M.Zenkiewicz, 2007). Currently these surface energy measurements are not accessible to field engineers due to the complexity and the specialized knowledge required. Therefore the future research into the surface energy measurement technique would be to develop a cost effective, accurate and reliable measurement instrument which can be used by field engineers.

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