



Surface modification of basalt with silane coupling agent on asphalt mixture moisture damage



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ARTICLE INFO

Article history:

Received 16 December 2014

Received in revised form 16 March 2015

Accepted 1 April 2015

Available online 8 April 2015

Keywords:

Surface modification

Basalt rocks

Silane coupling agent

Interfacial performance

Moisture sensitivity

ABSTRACT

A new silane coupling agent was synthesized based on γ -(methacryloyloxy) propyltrimethoxysilane (KH570). The surface of basalt rocks was modified by KH570 and the new silane coupling agent (NSCA), and the interfacial interaction between silane coupling agent and basalt was also studied. Fourier transform infrared (FTIR) spectroscopy and X-ray photoelectron spectroscopy (XPS) analysis showed that the silane coupling agent molecule bound strongly with basalt rocks. Scanning electronic microscopy (SEM) observation showed that a thin layer of coupling agent was formed on the surface of modified basalt. The boiling test and immersion Marshall test confirmed that the moisture sensitivity of basalt modified with the new silane coupling agent increased more significantly than that untreated and treated with KH570. The Retained Marshall Strength of basalt modified with the new coupling agent increased from 71.74% to 87.79% compared with untreated basalt. The results indicated that the new silane coupling agent played an important role in improving the interfacial performance between basalt and asphalt.

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1. Introduction

Many highway agencies have been experiencing premature failures that diminish the performance and service life of the pavements. One of the major causes of premature pavement failure is the moisture damage of the asphalt concrete layer. Moisture damage happens when the presence or infiltration of moisture through air voids natively [1]. Separation and removal of asphalt from aggregate surface due primarily to the action of moisture and/or moisture vapor is generally termed “stripping” [2–4]. The stripping of asphalt from the aggregates results in the reduction of strength of asphalt concrete mixture. As moisture damage reduces the internal strength of the asphalt mixture, the stresses generated by traffic loading increase significantly and may contribute to the development of various forms of pavement deterioration such as rutting, raveling and fatigue cracking on the layer [5–8]. Enormous funds and human resources have been exhausting on the repairing and maintenances of asphalt pavement.

Studies have shown that the adhesion between asphalt and aggregate in asphalt concrete pavement largely affects the ability to resist moisture damage [9–11]. How to enhance the adhesion between asphalt and aggregate is the key to improve the moisture damage resist ability of asphalt pavement. Many methods have been taken to mitigate moisture induced damage in asphalt pavement [12–14]. A general way is to use anti-stripping additives, including amine anti-stripping agent, hydrated lime, and cement, etc. It is understood that amine anti-stripping agent generally used is based on amines, especially fatty polyamines, and amine derivatives such as amides, substituted imidazoline, etc. [15]. There are many advantages by using amine anti-stripping agent. However, amine anti-stripping agent, which is usually unstable, trends to decompose and release irritant gas at high temperature. As a result, it not only loses the efficiency of anti-stripping agent but also causes harm to human. Therefore, research and development of non amine anti-stripping agent is the crucial direction in the future.

Coupling agent, known as “molecular bridge”, can make two relatively inert materials cross-link by means of winding up covalent bond or molecule chains, thereby improve the performance of composite by adding it into organic material or inorganic fillers [16–19]. Liang et al. [20] stated that the adhesion of asphalt and

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Table 1
Physical properties of asphalt binder.

Test items	Units	Values	Standards
Original asphalt binder			
Penetration at 25 °C	0.1 mm	69	T0604-2011
Ductility at 15 °C	cm	152	T0605-2011
Ductility at 10 °C	cm	27	T0605-2011
Softening point	°C	48.8	T0606-2011
Flash point	°C	314	T0611-2011
Specific gravity	g cm ⁻³	1.034	T0603-2011
Viscosity at 60 °C	Pas	198	T0625-2011
RTFOT			
Mass loss	%	0.17	T0610-2011
Penetration ratio at 25 °C	%	65.4	T0604-2011
Ductility at 10 °C	cm	6	T0605-2011

aggregate enhanced by the addition of KH570. Zhang et al. [21] determined that modifying the asphalt concrete mixes with the titanate coupling agent and silane coupling agent improved the rutting resistance and water stability.

In this paper, a new silane coupling agent was synthesized based on KH570 and the surface of basalt were modified using KH570 and the new silane coupling agent. The interfacial interactions between basalt and the new silane coupling agent were studied. In order to study the moisture sensitivity of asphalt mixture, the boiling test and immersion Marshall test were also conducted.

2. Experimental

2.1. Materials

All commercial chemicals were used without further purification in this study. Silane coupling agent KH570 was obtained from Duocai chemical plant in Shenzhen in this research. M-xylylenediamine was purchased from Institute Chemical Industry in Changsha. Dehydrated alcohol and deionized water were used as solvents. Aggregates were used with local basalt rocks in Hunan Province. The aggregate was washed in distilled water to remove all fines dried at 105–110 °C to constant weight and stored in airtight containers until required for use. Experiments were carried out with commercial 50 # asphalt from Changsha. Standard methods were used to test the basic physical properties of asphalt binder according to the Chinese norm JTG E20-2011 [22]. The results were listed in Table 1.

2.2. Synthesis of a new silane coupling agent

A three necked 250 ml round bottomed flask equipped with an overhead stirrer and an electric-heated thermostatic water bath was charged with 6.8 g m-xylylenediamine and 15 g methanol. The mixture was stirred at 50 °C for 30 min. Then 75 g KH570 was added dropwise. The system was stirred for 12 h. The solvent was removed under vacuum to give the new silane coupling agent (NSCA). The reaction is shown in Fig. 1.

2.3. Surface modification of basalt with silane coupling agent

At the beginning, 10% KH570 and 10% NSCA were added to alcohol-water solution to hydrolyze it sufficiently, respectively. The volume proportion of alcohol and water is 8:1. Secondly, the prepared basalt rocks were added into the above solution under stirring. After stirring for 1 h, the basalt rocks were dried at 130 °C for 1 h and then at ambient temperature for 24 h in a vacuum oven. Thirdly, the reaction product was immersed in deionized water for 24 h and washed several times with alcohol and deionized water to eliminate the influence of physical absorption. Finally, the

surface modified basalt was obtained by drying at 110 °C for 12 h and subsequently at ambient temperature for 2 h in a vacuum oven.

2.4. Characterization

The infrared spectra of original and modified basalt were conducted using a FTIR spectrometer (Shimadzu IRAffinity-1) to observe the functional groups of the samples. The samples were ground and dispersed in KBr, followed by compression to consolidate the formation of a pellet. FTIR spectra were obtained by the KBr pellet method with the wavenumber range from 400 cm⁻¹ to 4000 cm⁻¹ at a resolution of 2 cm⁻¹.

X-ray photoelectron spectroscopy (XPS) spectra of basalt before and after modification were recorded to further study the element composition and interfacial interactions using Thermo Fisher Scientific K-Alpha 1063 X-ray photoelectron spectrometer. In the XPS analysis, a monochromatic Al K α X-ray source was operated at 12 KV.

Surface morphologies of original and modified basalt were characterized by scanning electron microscopy (Hitachi S4800 SEM). The sample for observation was prepared as follows: a small amount of basalt rock powder was dispersed in ethanol ultra-sound for 30 min, then dropped on copper foil to observe its morphology. Before observation, samples were vacuum sputter coated with a thin layer of gold to increase the electrical conductivity.

Marshall strength ratio was measured for estimating moisture sensitivity of asphalt mixture. A Marshall equipment was used for the test, which consists of fully automatic asphalt mixture mixer (BH-10), Marshall compaction instrument (MDJ-IIB), and Marshall stability test apparatus (LDW-5A). Marshall specimens were prepared with 75 blows on each side of cylindrical specimens which measuring 101.6 mm (diameter) by 63.5 mm (height) according to the specification [22]. These specimens were used for the laboratory tests of mechanical properties. The bulk specific gravity (G_{mb}) and air void (V_a) of asphalt mixture were measured according to the specification. Consequently, the void in mineral aggregate (VMA) was calculated as $VMA = (1 - G_{mb} \times P_s / G_{mm}) 100\%$, where G_{mm} is the maximum theoretical specific gravity of asphalt mixture, P_s is the aggregate percentage by mass of mixture. The volume percentage of asphalt filled in the voids of mineral aggregate (VFA) was determined as $VFA = (1 - V_a / VMA) 100\%$. In Marshall test, a compressive loading was applied on the specimen at a rate of 50 mm/min till it was broken. The maximum loading at material failure is called Marshall stability (MS), and the associated plastic flow (deformation) of specimen is called flow value (FV).

3. Results and discussion

3.1. Modification mechanism

The molecular formula of NSCA is shown in Fig. 1. During hydrolysis of the silane, the SiOCH₃ group will transform into Si-OH. We hypothesize that the surface modification mechanism of basalt follows the mode given in Fig. 2 as reported in references [16,23]. In the view of the molecular structure, there are two different chemical functional groups in the silane coupling agent. Therefore one of the functional group of silane coupling agents could react with the hydroxy groups of the inorganic material to form hydrogen bonds and then transfer to covalent bonds through hydrolysis condensation, dehydration and solidification under certain conditions. The other functional group combined with asphalt materials to form strong interfacial bonds within asphalt materials. Asphalt and basalt, in this way, the two kinds of materials with different properties are bound together finally [24,25].

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