

# Preparation and characterization of novel light induced self-healing materials for cracks in asphalt pavements



Rui Li <sup>a</sup>, Jiaqi Chen <sup>a</sup>, Tianshu Zhou <sup>b</sup>, Jianzhong Pei <sup>a,\*</sup>

<sup>a</sup> Highway School, Chang'an University, Xi'an 710064, China

<sup>b</sup> Bureau of Transportation in Jiaxing, Jiaxing 314599, China

## HIGHLIGHTS

- Light-induced self-healing materials were successfully prepared.
- The composition of the self-healing agent is the OXE-CHI-PUR networks.
- The self-healing agent can healing the asphalt pavement cracks upon the exposure to light.
- The self-healing agent can improve the performance of asphalt pavement.

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## ABSTRACT

This paper is about a kind of asphalt crack self-healing material upon exposure to UV light. In this work, the OXE-CHI-PUR networks self-healing agent are synthesized by the photocatalytic-based chemical method. It is a kind of asphalt crack self-healing material upon exposure to UV light. Fourier transform infrared spectroscopy is used for characterization of the self-healing agent. TG analysis shows self-healing agent has good thermal stability. Beside, this paper reveals the repair mechanism, validation and studies the influence of self-healing agent on the properties of asphalt. The result shows that this OXE-CHI-PUR network preferably synthesized self-healing agent for road materials. Self-healing agent can improve the low-temperature property, construction property and anti-aging property of asphalt. But high-temperature property of the asphalt has slight effect.

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## 1. Introduction

Due to the effect of natural environment, vehicle load and other factors, inevitably there would generate cracks in the asphalt pavement. Cracking is one of the main diseases of the asphalt pavement in every country [1]. The form of asphalt pavement cracking is not all the same. For the aspect of the asphalt pavement cracking reasons, the crack can be divided into load-type cracks and non-load-type cracks [2]. Load-type cracks are mainly due to traffic loads by force in the pavement structure; Non-load-type cracks mainly include temperature-shrinkage cracks and dry-shrinkage crack. Temperature-shrinkage cracks are the open type of crack formation mode, the cause of dry-shrinkage cracks are formed by the reflective cracks of dry-shrinkage of the semi-rigid base layer material.

After the pavement cracked, rain and snow would penetrate into surface layer and the base layer along the cracks. Coupled with the traffic loads, leading to the structure of the pavement gradually become weaken and even destroyed, resulting in varying degrees of damage on the road surface. That would seriously affect the usability of the road. The maintenance period of the road comes ahead, the costs of maintenance greatly increase and the service life of roads shortens [3–5]. Therefore, the study of preventive measures of the asphalt pavement cracking for improving road transport capacity and service life, improve investment efficiency, accelerate society development, and has important practical significance.

Nowadays the studies about the asphalt pavement crack-healing measures are various, but there is little healing agent that could make asphalt pavement crack healing upon the exposure to light [6–9]. Self-healing technology is an imitation of the process of the organism's damage automatically heal. When the polymer materials were cracked self-healing technology could heal it in time, thus partially restore the performances of the material [10].

\* Corresponding author.

E-mail address: [peijianzhong@126.com](mailto:peijianzhong@126.com) (J. Pei).

Thereinto microcapsules is a kind of common self-healing technology. However the technical requirements of microcapsules, such as mechanical properties and storage environment of the shell material, are very high [11–14]. It is difficult to control well. Light self-healing materials have several significant advantages: fast, efficiently, low-energy consumption and no pollution [15,16]. In this study, the self-healing agent should be mixed into the asphalt. When the asphalt were cracked the self-healing agent could enhance the self-healing ability of asphalt upon exposure to UV light, resulting the microcracks healing, and extending the life of the pavement.

## 2. Experimental

### 2.1. Materials

The Chitosan (CHI), isopropanol alcohol, 3-chloromethyl-3-methyl oxetane, tri-functional homopolymer of hexamethylene diisocyanate (HDI), polyethylene glycol. Sodium hydroxide (NaOH), dimethyl sulfoxide (DMSO).

Grade bitumen, Ssangyong-70, was from H.I.T.C Corp, the physical properties of grade bitumen were tested are shown in Table 1.

### 2.2. Fabrication of photocatalytic self-healing agent

Weighing a few chitosan added into a 1 mol/L NaOH solution and stirring for 48 h at 0 °C, followed by refrigeration at 0 °C for 48 h. Thawed that solution and mixed with isopropyl alcohol and then stirring for 1 h. A certain dosage of pre-heated 3-chloro-3-methyl oxetane added into the mixture and stirred 1 h at 80 °C. The product was filtered and washed several times with methanol until adjusting the pH to neutral, followed by drying it at 60 °C for 12 h. The product was dispersed in a certain pH of dimethyl sulfoxide, followed by irradiation with ultraviolet rays for 20 min. Cleaning the product with methanol, and then the product was placed in the drybox in a certain time. The resulting product was identified as oxetane-substituted chitosan (OXE-CHI).

OXE-CHI (OXE:CHI = 1:3) was dispersed in DMSO by sonicating at 25 °C for 12 h followed by continuous stirring at 80 °C for 48 h. OXE-CHI-PUR networks films were prepared by HDI reacting with dispersed OXE-CHI and polyethylene glycol (PEG) in a 500 ml three-neck reaction flasks at 25 °C for 10 min under N<sub>2</sub> atmosphere.

### 2.3. Fourier transform infrared spectroscopy (FT-IR)

Infrared spectral analysis was used to evaluate the synthetic degree of the self-healing agent. A drop of the colloidal solution was dripped between two pieces of KBr to form a liquid film for testing.

### 2.4. Thermogravimetric analysis (TG)

Thermal stability of self-healing agent were characterized at a scanning rate of 10 °C min<sup>-1</sup> with an N<sub>2</sub> flow of 20 ml min<sup>-1</sup>.

### 2.5. Optical morphology (OM)

An optical microscope was used to check the reparative process of the asphalt crack in emulsion. Approximately 1 ml of the colloidal solution was extracted and spread onto a clean glass slide (3 × 3 cm). Various images were taken at different stages of the crack-healing process.

### 2.6. Other instruments

The other major instruments include low temperature asphalt extension apparatus, asphalt softening point tester, asphalt movement viscometer, asphalt thin-film oven etc.

**Table 1**

The physical properties of bitumen.

Physical properties	Measured values
Penetration (25 °C, dmm)	72
Softening point (°C)	48.5
Ductility (15 °C/10 °C, cm)	140.0/15.9
Viscosity (60 °C, Pa s)	256
Viscosity (135 °C, Pa s)	0.52

## 3. Results and discussion

### 3.1. FT-IR analysis

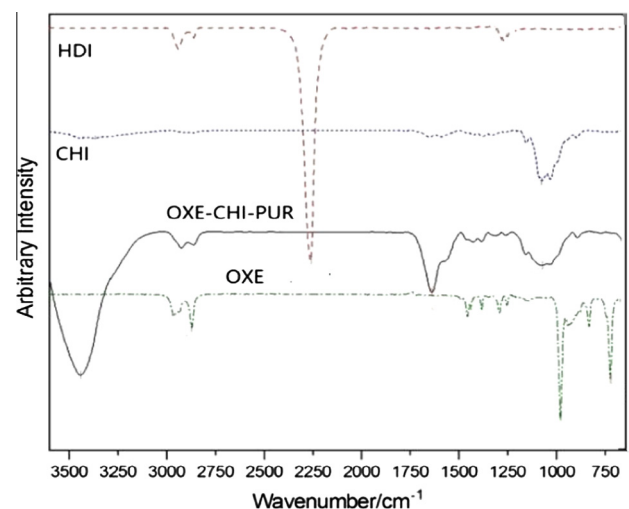
Fig. 1 is the IR spectra of OXE-CHI-PUR networks self-healing agent, we can see from it that the infrared spectra of OXE, vibration-shrink peak 725 cm<sup>-1</sup> shows the chlorine group of the oxetane. Vibration-shrink peak 2872 cm<sup>-1</sup> show -CH<sub>2</sub>- of oxetane. In the infrared spectra of HDI, vibration-shrink peak 2260 cm<sup>-1</sup> show the isocyanate group of the tri-functional homopolymer of hexamethylene diisocyanate. In the infrared spectra of CHI, vibration-shrink peak 3366 cm<sup>-1</sup> show the primary amide of the chitosan and vibration-shrink peak 1178 cm<sup>-1</sup> shows the ether bond of the chitosan. Vibration-shrink peak 3441 cm<sup>-1</sup> shows the deacetylation of chitosan and the amide of the tri-functional homopolymer of hexamethylene diisocyanate. Vibration-shrink peak 1639 cm<sup>-1</sup> shows the carbamido that formed in the reaction of the amidogen of chitosan with the ureido of isocyanate groups of HDI. Vibration-shrink peak 1074 cm<sup>-1</sup> shows the ether bond that formed in the reaction of the hydroxy of the chitosan and the halogen of 3-chloromethyl-3-methyl oxetane.

Compared the infrared spectra of raw materials with the infrared spectra of self-healing agent, the vibration-shrink peak 3366 cm<sup>-1</sup> of primary amide in chitosan was disappeared. First, it took deacetylation to generate amidogen. Then it reacted with the isocyanate groups of HDI, resulting ureido of self-healing agent could be found a vibration-shrink peak at around 1639 cm<sup>-1</sup>. In the infrared spectra of chitosan, the vibration-shrink peak 1078 cm<sup>-1</sup> was only changed at intensity, because the ether bonds of the chitosan did not react. In the infrared spectra of oxetane, the vibration-shrink peak 2872 cm<sup>-1</sup> was only changed at intensity, because the -CH<sub>2</sub>- of the oxetane did not react. In the infrared spectra of HDI, the vibration-shrink peak 2260 cm<sup>-1</sup> of carbamido was disappeared, because it reacted with the amidogen of chitosan and at last generated carbamido of the self-healing agent.

In the infrared spectra of oxetane, the vibration-shrink peak 725 cm<sup>-1</sup> of chlorine group was disappeared, because the chlorine group of the oxetane took hydrolysis reaction and at last took the dehydration-condensation reaction to generate ether bond.

### 3.2. TG analysis

Fig. 2 shows the TG analysis of the OXE-CHI-PUR networks self-healing agent. From Fig. 2 we can see that the weight loss ratio of



**Fig. 1.** IR spectra of self-healing agent.

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