



# Preparation, properties and modification mechanism of polyurethane modified emulsified asphalt

Xiaohui Sheng<sup>a</sup>, Mo Wang<sup>a</sup>, Tao Xu<sup>a,\*</sup>, Jun Chen<sup>b</sup>

<sup>a</sup> School of Civil Engineering, Nanjing Forestry University, 159, Longpan Road, Nanjing 210037, Jiangsu, China

<sup>b</sup> School of Civil and Transportation Engineering, Hohai University, 1, Xikang Road, Nanjing 210098, Jiangsu, China

## HIGHLIGHTS

- New PU modified emulsified asphalt is prepared using developed prepolymer method.
- PU modified emulsified asphalt process includes physical and chemical modifications.
- PU modified emulsified asphalt shows satisfactory mechanical and thermal properties.
- PU forms compact interpenetrating network of molecular chains in emulsified asphalt.
- A suitable PU content of 6% has been proposed to prepare modified emulsified asphalt.

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## ABSTRACT

To prepare new polyurethane (PU) modified emulsified asphalt (EA), and further understand modification mechanism of PU to EA, effects of PU on basic engineering performances, chemical composition compatibility, morphology and element compositions, thermal properties, phase microstructures of EA were discussed. The results indicate that basic properties of PU modified EA firstly meet relevant requirements of technical specifications. Improvement effects of PU with a suitable content on penetration and ductility is better than that of styrene butadiene styrene (SBS), but their softening points are similar. Secondly, PU modified EA process includes physical and chemical modifications. PU is dispersed uniformly in evaporation residues of EA when PU content is less than 6%, and meta-stable two-phase interface transition layers are formed between PU and asphalt binder, forming a uniform dispersion system and interpenetrating network structures in modified EA. The compatibility between PU and EA is satisfactory, forming a stable multiphase structure in modified EA. This is conducive to improve high-temperature stability and low-temperature ductility of modified EA. Thirdly, the contents of such main chemical elements as carbon (C), oxygen (O) and sulfur (S) in evaporation residues of EA show no obvious changes before and after modified by PU, indicating that physical modification is main method during the preparation of PU modified EA. Fourthly, PU is difficult to form a continuous crystalline phase in EA. However, the molecular chain spacing and microcrystalline size of PU modified EA are decreased at a suitable PU content, generating more compact interpenetrating network of molecular chains. Motions of asphalt molecules are limited, improving thermal stability and mechanical properties of EA. Finally, a suitable PU content of 6% is proposed to prepare modified EA.

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## 1. Introduction

Many distresses such as loose, crack and rutting were often found on asphalt pavement due to the continuous increase in asphalt aging, traffic volume, overloading, and canalized traffic in China [1]. It is necessary to maintain and restore the distressed

pavement surface performance. Currently, the commonly used pavement maintenance materials are emulsified asphalt (EA). However, since EA is a kind of water-emulsion materials, EA performance mainly depends on technical properties of evaporation residues after the moisture is evaporation [2]. Further, it is difficult for EA to meet traffic and environmental requirements of asphalt pavement before it is modified. Therefore, the modifier selection to modify first and then emulsify asphalt binder has been paid more attention, improving the durability of maintenance materials of asphalt pavement [3].

\* Corresponding author.

E-mail address: [seuxt@hotmail.com](mailto:seuxt@hotmail.com) (T. Xu).

In general, the modifiers of asphalt binder are classified into three categories according to their chemical structures and properties, including elastomers, plastomers and polymeric materials with reactive groups [4]. Among them, such thermoplastic elastomers as SBS and styrene-isoprene styrene (SIS) block copolymers are most commonly used. Their microstructures are generally made up of elastomers and plastic incompatible macromolecules or segments, forming a two-phase system to absorb light components of asphalt binder. Thus it is swelled to form a cross-linked network structure, which facilitates asphalt binder to show plasticity, elasticity and ductility [5]. Behnood et al. [6] found that SBS modifier could improve the high-temperature properties of asphalt binder, but SBS had not significantly effects on its low-temperature performance. Nciri et al. [7] reported effects of SBS modifier on physical and chemical properties of asphalt binder, and found SBS modification results strongly depended on asphalt component and SBS contents.

Additionally, other modifiers also utilized to modify asphalt binder. Liang et al. [8] optimized molecular structural parameters of ethylene vinyl acetate copolymer (EVA) to use as modifier of asphalt binder. EVA improved the high-temperature stability and low-temperature anti-cracking performance, and balanced the viscoelasticity and thermal stability of asphalt binder. Gama et al. [9] modified asphalt binder using elastomer polymers, high-density polyethylene, and polyphosphoric acid, increasing the elastic properties to lower the permanent deformation of asphalt pavement. Han et al. [10] used rubber powder and nanosilica to modify asphalt binder for improving the high-temperature stability and elastic recovery rate, but the low-temperature ductility was slightly decreased.

Compared with the elastomer modifier, reactive polymers provide a different modification method for asphalt binder. The reactive ethylene ternary copolymer was utilized as bulking agent between polyolefin and other polymers [11]. These copolymers could be theoretically used as asphalt modifiers or compatibilizers between asphalt binder and conventional polymers. However, this application in asphalt industry is actually very limited, and there are a few studies on these asphalt modifiers [12].

At present, in general, the commonly used polymer modifiers are physically blended with asphalt binder, and no obvious chemical changes occur [13]. In addition, there are some differences between asphalt binder and polymer modifiers in molecular weight, density, structure and other properties. It is difficult to form a stable thermodynamic system between polymer modifiers and asphalt binder so that their compatibility is limited, leading to delamination and segregation without continuous stirring at high temperature [13]. Therefore, several commonly used polymer modified EA has some deficiencies in production, storage, economical efficiency, and performance. For example, SBS modified asphalt binder is difficult to emulsify because its viscosity is so large. Also, the high-temperature stability of SBR modified asphalt is dissatisfactory and current epoxy resin is so expensive [14]. Therefore, it is necessary to find a new modifier and modified method to prepare modified EA.

More recently, PU, as a new type of modifier, has attracted more attention. PU is a kind of block carbamate polymer, which is usually synthesized by using long chain polyols (e.g. polyester and polyether) to react with isocyanate and chain extender [15]. PU properties greatly depend on microstructures of soft and hard segment phases, in which the soft segment phase provides the elasticity and low-temperature ductility of PU materials [16]. On the other hand, the hard segment phase provides the softening temperature, melting temperature, adhesivity, high-temperature stability, and mechanical properties [17]. Because PU has dual advantages of rubber and plastic, it has been applied in automobile

industry, construction, household, petroleum industry, intelligent packaging, textiles, biomedicine, etc [18].

Singh et al. [19] used PU prepolymer to modify asphalt binder, and found the glass transition temperature and moisture permeability of prepared modified asphalt binder were lowered. The waterproofing and sealing effects were limited due to the lack of flexibility at low temperature. Carrera et al. [20] studied the feasibility of preparing PU modified EA, and pointed out the modifier concentration was key factor to affect its storage stability and rheological properties. Xia et al. [21] reported that PU modifier improved the resistance to deformation and mechanical properties of asphalt binder due to the chemical modification process. Yu et al. [22] modified asphalt binder using nanoscale PU emulsion, and found the PU emulsion was uniformly dispersed in modified asphalt binder, enhancing the storage stability of the modified asphalt system.

It is notes that although PU has been applied in different fields, it is seldom used as EA modifier to prepare maintenance materials of asphalt pavement. The preparing method of PU modified EA was rarely discussed and it is still uncertain whether PU has satisfactory influences on properties of EA to better meet technical requirements of related engineering standards. Also, the modification mechanism of PU on EA is not very clear. The objectives of this study is to prepare a new PU modified EA using the developed prepolymer method, and to further understand modification mechanism of PU to EA, and thus propose a suitable PU content to modify EA, improving various properties of EA.

In this study, asphalt binder was first modified by PU using the developed pre-polymer method, and then chain extension and emulsification were performed to prepare PU modified EA. Synchronously, SBS modified EA was prepared to used as a control sample. Then basic performances of prepared modified EA with different PU contents were tested to confirm whether they can meet relevant technical requirements. After that, Fourier transform infrared spectroscopy (FTIR) was used to characterize effects of PU on chemical compositions and functional groups of EA, discussing the compatibility between EA and PU with different contents.

Also, influences of PU with different contents on morphology and element compositions of evaporation residues of EA were studied by environmental scanning electron microscope (ESEM) and energy dispersive spectrometer (EDS). Additionally, influences of PU contents on thermal stability, endothermic and exothermic reactions of EA samples were analyzed using a differential scanning calorimeter (DSC). Finally, effects of different PU contents on the phase microstructures and network structure of modified EA were investigated by X-ray diffraction (XRD), revealing the modification mechanism of PU to EA. As a result, a suitable PU content was proposed to modify EA and the modification mechanism of PU to EA was further understood.

## 2. Experiment and method

### 2.1. Materials

Polytetrahydrofuran ether glycol (PTMG, Aladdin Reagent Co., Ltd., Shanghai, China. Mn = 2000) was selected as soft segments in PU because its melting temperature is lower. Thus PTMG was used to synthesize PU with a lower glass transition temperature ( $T_g$ ).

The 2, 4-toluene diisocyanate (TDI, TCI Chemical Industry Development Co., Ltd., Shanghai, China.) was used as hard segments in PU due to its relatively regular microstructures so that the prepared PU has more obvious phase separation.

The 1, 4-butanediol (BDO, Sinopharm Group Chemical Reagent Co., Ltd., Shanghai, China.) was applied as chain extender to improve molecular chain regularity of hard segments, increase crystallinity and phase separation in PU, and elevate strength and modulus of PU.

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