



Polyglycerol-based organic-inorganic hybrid adhesive with high early strength

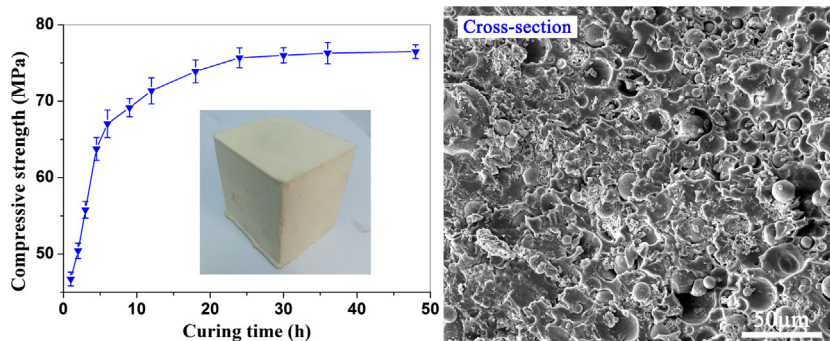
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HIGHLIGHTS

- Polyglycerol-based organic-inorganic hybrid adhesive was prepared via a simple and effective strategy.
- The adhesive showed a high early strength of 50 MPa after curing for 2 h.
- A probable formation mechanism of adhesive was proposed.
- The adhesive exhibited excellent thermostability and adhesion performance.

GRAPHICAL ABSTRACT



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ABSTRACT

The rapid achievement of high strength adhesive is of ever increasing importance for underground construction. Herein, a simple and effective strategy to make an adhesive in a “high early strength” way was based on excellent crosslinking and synergistic interactions among tripolyglycerol, waterglass and polyaryl polymethylene isocyanate (PAPI). The resultant organic-inorganic hybrid adhesive with three-dimensional interpenetrating network structure showed an excellent mechanical performance, the compressive strength could reach more than 50 and 60 MPa after curing for 2 and 4 h, respectively. The adhesive was mainly composed of amorphous polyurethane and crystalline polysilicic acid/ NaHCO_3 composite, and its probable formation mechanism was proposed. Furthermore, the adhesive was thermally stable below 260 °C and presented a favorable adhesion performance for dry/wet sands with various sizes. This work may open a simple and convenient avenue for the preparation of organic-inorganic hybrid adhesive with high early strength.

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1. Introduction

High early strength is gaining increasing attention in adhesives owing to the requirement of underground construction (etc. mine,

roadbed). Among various adhesives, polyurethanes, known as chemical adhesives with the advantages of both strong adhesion and easy manipulation, has attracted considerable interest as highly efficient consolidation material, and been regarded as one of the most promising candidates due to its light weight, low viscosity, good permeability, low thermal conductivity and high mechanical performance, and thus displayed impressive application to bonding and reinforcement of broken matrix [1–4]. However, most of these polyurethane adhesives

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show some drawbacks such as high cost, flammability, poor barrier property and thermal stability which limit its practical applications [5, 6]. Thus, finding a direct and effective way to achieve modified polyurethane adhesives with improved performance is highly desirable.

Inorganic modification is an important consideration in the performance improvement of polyurethane adhesives. These materials with particle morphology such as cement [7,8], limestone filler [9], montmorillonite [10,11], mortar [12], hydrotalcite [13], concrete [14], silica [15, 16] and sand-clay mixture [17] have been extensively explored for organic-inorganic hybrid polyurethane adhesives. However, the expected comprehensive properties (etc. flame resistance, thermal stability, high mechanical properties) cannot be still met due to the heterogeneous dispersion of particles in the polyurethane matrix. Waterglass, owing to its favorable characteristics such as being a true solution, low viscosity, high thermal stability, low cost and nontoxic nature, has been widely employed as an adhesive for various reinforcements [18–21]. In view of respective properties of waterglass and polyurethane, it is expected that the combination of polyurethane with waterglass into a composite will be a more effective and economic adhesive for reinforcement of underground construction. Recently, the waterglass-modified polyurethane adhesives with flame resistance, low cost, high thermal stability and mechanical properties have been developed, and exhibited a satisfactory performance towards grouting [22,23]. Our group also reported the protocol of the synthesis of silicate/polyurethaneurea composites based on dipropylene glycol dibenzoate. The obtained composites were thermally stable, and the compressive and flexural strength could reach 42.6 and 29.2 MPa after curing for 6 h, respectively [24]. Despite many advances in waterglass/polyurethane composite adhesives, it is still a challenge to search for some new materials and methods to meet the rapidly growing demand for high early strength in practical applications.

In this work, a high-early-strength waterglass/polyurethane adhesive based on polyglycerol was prepared successfully via a simple and effective strategy. The microstructure, composition and property of the resultant adhesive were explored in detail, and its possible formation mechanism is proposed.

2. Experimental

2.1. Materials

The PAPI (30.75 wt% NCO, 220 mPa·s) was purchased from Dow Chem. Co., America. Tripolyglycerol (99%, 1200 mg KOH/g, 82 cPa·s) was purchased from Jinan Dongrun Technology Co., Ltd. Waterglass ($\text{Na}_2\text{O} \cdot n\text{SiO}_2 \cdot m\text{H}_2\text{O}$, modulus 2.3) was supplied by Zhengzhou Jiankete Engineering Materials Co., Ltd. Alkylphenol polyoxyethylene (OP-9, 93 mg KOH/g) was obtained from Shanghai Sinopharm Chemical Reagent Co., Ltd. 2,2-dimorpholinodiethylether (DMDEE) was supplied by Shanghai Rongrong Chemical Co., Ltd. All other chemicals were of analytical grade and purchased from Shanghai Sinopharm Chemical Reagent Co., Ltd.

2.2. Preparation of adhesive

The adhesive was prepared through a facile room-temperature-cured process. Specifically, waterglass (90.8 g), tripolyglycerol (5.6 g), OP-9 (1.2 g), glucose (2 g) and DMDEE (0.4 g) were added into a plastic cup and homogenized at 400 rpm for 3–5 min, denoted as component A. The PAPI (100 g) was added into another plastic container, denoted as component B. Subsequently, the solutions in above two plastic containers were mixed for 30 s by vigorous stirring (500 rpm), and then transferred into a steel mold with dimensions $4.0 \times 4.0 \times 4.0 \text{ cm}^3$. Finally, a fully cured specimen for tests was rapidly achieved under the room temperature.

2.3. Characterization

Scanning electron micrograph (SEM) was performed on a Hitachi S-4800 scanning electron microscope (Hitachi Ltd., Tokyo, Japan) working at 15 kV, and an energy dispersive X-ray spectroscopy (EDS) was used for elemental analysis. IR spectra were recorded using Fourier transform infrared spectroscopy (FTIR: Thermo Nicolet, WI, USA). Raman spectra were conducted on a Renishaw RM3000 Raman spectrometer (InVia, Renishaw Co., UK). X-ray diffraction (XRD) was carried out on a Bruker-AXS D8 X-ray diffractometer using $\text{Cu K}\alpha$ radiation. The thermo gravimetric data were obtained between 28 and 800 °C with the DSC-TGA Q600 thermal analyzer system at a heating rate of 5 °C/min, under air atmosphere.

2.4. Mechanical measurements

All the compressive strength measurements were carried out on a compressive instrument (Model YAW-300/5, Shanghai Bairuo Instrument Equipment Co., Ltd., China) at room temperature. The compressive tests were performed on samples with dimensions $4.0 \times 4.0 \times 4.0 \text{ cm}^3$ with a constant loading rate of 0.3 MPa/s.

3. Results and discussion

Fig. 1a shows the digital image of adhesive after curing for 2 h at room temperature, the surface was uniform, dense and relatively flat, indicating the excellent homogeneous interactions among waterglass, PAPI and tripolyglycerol during curing of the adhesive, which was beneficial for its mechanical performance. The compressive strength of the adhesive as a function of curing time was examined, and the obtained results are presented in Fig. 1b. The compressive strength rapidly increased at early stage up to 6 h, and then increased slowly to reach a stable value (76 MPa). Remarkably, after curing for 2 and 4 h, the compressive strength could reach more than 50 and 60 MPa, respectively. The rapid achievement of favorable mechanical performance can be closely related to the excellent synergistic interactions among raw materials and microscopic structure of the adhesive.

The microscopic morphology and component of the curing adhesive were characterized using SEM and EDS. As seen in Fig. 2a–d, the fracture surface of adhesive was compact and free from appreciable cracks. The organic phase in the adhesive interconnected each other, and many spherical insertions with average size of around 12 μm in diameter were uniformly distributed in the organic polymer. Furthermore, note that some small, irregular and closed interspaces existed between spherical particles and polymer due to the resulting CO_2 produced in the curing process of the adhesive [22]. SEM observation confirms the formation of adhesive with an integrated and dense structure, indicating a homogenous reaction of the adhesive, which is favorable for its mechanical performance. Fig. 2e and f show the EDS analyses of spherical particles and organic phase, respectively. The elemental component of spherical particles indicates the presence of C, O, Na and a large amount of Si, and the organic phase displays abundant C but relatively small amounts of Na and O. According to the raw materials and related interactions of adhesive, EDS results suggest that spherical particles may be mainly composed of silicate, polysilicic acid, NaHCO_3 or Na_2CO_3 , and the possible composition of the organic phase is polyurethane.

FTIR, Raman and XRD analyses were conducted to further investigate the composition and structural information of the adhesive. Fig. 3 shows the FTIR spectra of waterglass, polyurethane and adhesive. In the spectrum of the adhesive, significant bands corresponding to polyurethane and waterglass were clearly observed, such as N–H stretching at 3414 cm^{-1} , asymmetric C–H stretching at $2847\text{--}2984 \text{ cm}^{-1}$, carboxylic C=O stretching at 1722 cm^{-1} , C–O stretching at 1076 cm^{-1} , bending vibration of aromatic and alkene moieties at $550\text{--}890 \text{ cm}^{-1}$, O–H stretching at $3100\text{--}3600 \text{ cm}^{-1}$, asymmetric Si–O stretching at $900\text{--}1100 \text{ cm}^{-1}$, Si–O–Si bond rocking vibration at 468 cm^{-1} .

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