



## Technical note

# A novel waterproof, fast setting and high early strength repair material derived from metakaolin geopolymer



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## HIGHLIGHTS

- A novel waterproof, fast setting geopolymer repair material was proposed.
- Contact angle reaches 122° and specimen floats on water level after modification.
- The newly repair material has short setting time of 24 min and high flow of 212 mm.
- Compressive strength and bond strength reach 10 MPa and 0.6 MPa at 3 h, respectively.

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## ABSTRACT

In this work, a novel waterproof, fast setting geopolymer repair material with hydrophobic surface and high compressive strength and bonding strength was proposed. The experimental results showed that surface hydrophobic modification was completely achieved due to the increasing contact angle on the surface of geopolymer from 21° to 122°, the floating of specimens on the water surface and low water adsorption of about 0.5% after 28 days of exposure. This newly developed geopolymer repair material has short setting time of 24 min, high flow of 212 mm and high early compressive strength and bonding strength of the interfacial bonding zone as indicated by the dense microstructure.

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## 1. Introduction

The common deterioration form of concrete materials starts from or near the surface, the quality of which determines the penetration rate of outer aggressive gases and ions [1] and cracks on the surface also damage the appearance and cause re-deterioration of repaired regions. Repair of deteriorated concrete structures are essential not only to utilize them for intended service-life but also to assure the safety and serviceability of the associated components. Selecting suitable repair materials for concrete requires an understanding of compatibility between the substrate and repair materials and behavior of concrete materials in the exposure conditions [2].

In recent years, research work on repair materials and technologies have been undertaken to enhance the durability of concrete.

Liu et al. [3] used highly flowable reactive powder mortar composed of cement, quartz sand, quartz powder, silica fume, superplasticizer and water as repair material for cracks and pores in concrete. Flowable system as promising repair materials was also proved by Dawood [4]. Qiao et al. [5] demonstrated that magnesium phosphate cement mortar had superior performance to ordinary Portland cement mortar and was promising to be utilized in repair and maintenance of concrete. Qian et al. [6] also assessed the bond performance of magnesium phosphate cement mortars as repair materials. Petcherdchoo et al. [7] concluded that the increase of the amount of fly ash in repair concrete could extend service life of chloride-exposed concrete structures. Kobayashi et al. [8] regarded the high performance fiber reinforced cement composites as a repair material and it effectively suppressed chloride penetration and prevented reinforcement corrosion. Wu et al. [9] proposed a polymer based crack repairing material and reported that the repairing material had great potential for repairing concrete cracks. Al-Zahrani et al. [10] also evaluated and

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compared the mechanical properties and durability characteristics of nine polymer- and cement-based repair mortars.

However, the previously reported findings mainly focused on cement based repair materials and the solved main problem was the bonding between substrate and repair material, which was enhanced from aspects of microstructure densification and pore refinement, adjusting of curing conditions and mix proportions or the addition of mineral admixtures. On reviewing the existing literatures, techniques and reports on achieving waterproof, fast setting and high strength geopolymer repair material by surface modification were scant and limited even though some primary work about geopolymer repair materials has been carried out [11–13]. Importantly, geopolymer has been used as protection coating for marine concrete by Zhang et al. [14–16] and it was proved that the geopolymer coating could extend the service life of concrete structures exposed to marine environments by inhibiting the intrusion of corrosive ions.

In order to enhance the durability and prolong the service life of concrete, new repair material with waterproof properties and high early strength, which could be used for the road concrete repair or as protection coatings for marine concrete, is urgently needed. In this work, the liquid surface modification agent was synthesized for the modification of geopolymer repair material. A new waterproof, fast setting geopolymer with hydrophobic surface and high early strength was developed and the microstructure of interfacial bonding zone was investigated.

## 2. Experimental procedure

Metakaolin was obtained by calcination of kaolinite at 850 °C. Alkali activator was a combination of sodium silicate and sodium hydroxide in analytical reagent degree. The liquid portions of alkali activator were 10 M sodium hydroxide and sodium silicate with 14.51% Na<sub>2</sub>O, 33.39% SiO<sub>2</sub>, and 48.53% H<sub>2</sub>O. Geopolymer specimens were synthesized by alkaline activation of metakaolin in the liquid alkali activator mentioned above (modulus of alkaline activator  $M_s = 1.5$ , the mass ratio of sodium silicate and sodium hydroxide = 8:1). The liquid/solid mass ratio equals to 0.5, in which the liquid material consists of alkali activator and the solid material is metakaolin. Fresh geopolymer were cast into triplet steel cubes molds with size of 40 × 40 × 40 mm<sup>3</sup> and vibrated to remove entrained air bubbles. After hardening, the samples were released from the molds and were subjected to further curing in a standard condition of 20 °C and 95% relative humidity up to acquired days for other tests.

The waterproof layer on the surface of geopolymer samples was built by the methods proposed in Ref. [13] published previously by the authors of this work. The waterproof layer on the surface of geopolymer samples was built by coating hydrophobic modification agent, which was liquid state and consisted of water, aluminum trichloride (12 mg L<sup>-1</sup>, as a catalyst) and fatty acid. Palmitic acid was chosen as the best modifier and it was dissolved by glacial acetic acid (4 g L<sup>-1</sup>). The fatty acid solution was 10% of the water, and the hydrophobic modification was carried out at normal temperature.

The workability of geopolymer repair material was measured by using a flow table test to check the flow diameter of fresh pastes before casting.

The process of setting was determined by the Vicat penetration test. The test consists of the measurement of the penetration depth of a metal needle which falls down under gravity in a fresh geopolymer mixture. The final setting time is defined as the period of time from the pouring of the fresh geopolymer mixture into the molds to the moment when the needle penetrates the solidified mass to the depth of 0.5 mm.

Compressive strength was performed on cubes with the dimensions of 40 × 40 × 40 mm<sup>3</sup> using a universal testing machine with a loading capacity of 3000 kN using a loading rate of 0.6 MPa/s at 3 h, 6 h, 1 day, 3 days and 28 days, respectively. For each testing age, six specimens were tested and the mean value of these measurements was reported.

The bonding strength was used to measure the bond strength between the cement mortar substrate and the repair material. It was defined as tensile bond strength carried out by pull-off method developed in Ref. [5]. The mix proportion of the substrate concrete was based on a 28-days compressive strength of 40 MPa, consisting of cement, water, sand and crushed aggregate by 1:0.5:1.5:2.5 (by weight). The cast substrate slabs were kept in laboratory condition for at least 28 days before the repair material was placed. Before casting geopolymer repair material over surface of substrate, the surfaces were roughened using a steel wire brush to remove the top cement paste layer. The estimated amplitude of roughness was 3–4 mm. For measurement of pull-off strength, disk specimens of 75 mm diameter and 15 mm height were cast on the roughed surface of OPC concrete and the test was performed with pull-off tester (Dyna Z16SE). The bond strength was calculated by dividing the maximum force by the bond area.

The contact angle was measured with the sessile drop method by using the contact angle goniometer, an OCA20 measuring system (Dataphysics, Filderstadt, Germany) with a maximum error was 0.05°. The liquid droplet was deposited by a syringe pointed vertically down onto the sample, and the angle could be captured by a high resolution camera and analyzed by analysis software.

Microstructure of specimens was performed using a Japanese Netherlands FESEM Quanta SU8010 electron microscope, operating at an accelerating voltage of 15 kV for photomicrographs.

Water absorption was measured by drying a specimen to a constant mass in an oven at 105 °C and allowed to cool in an air-tight container for 24 h. Each specimen was then weighed and immediately immersed in water, on removal from the water, the surface of the specimens was wiped and weighed again. The water absorption, measured at 1 h, 2 h, 6 h, 1 day, 3 days and 28 days, respectively, was calculated from the increase in mass of the specimen and expressed as a percentage of the dry specimens.

## 3. Results and discussion

Static water contact angles of geopolymer before and after surface hydrophobic modification are provided in Fig. 1a–b. It could be observed that the contact angle on the surface of geopolymer before modification is about 21° in Fig. 1a, while it significantly increases to 122° on the surface of modified geopolymer as indicated in Fig. 1b. It can be concluded that surface hydrophobic modification of geopolymer is completely achieved.

Waterproof performances of geopolymer before and after surface hydrophobic modification are illustrated in Fig. 1c. It can be observed that geopolymer floats on the water level after surface hydrophobic modification due to waterproof modification. The effects of surface hydrophobic modifier on water absorption of geopolymer at various water curing ages are shown in Fig. 1d. It is observed that the water absorption varies between 0.01% and 0.52%. It exhibits very low absorption characteristic when compared with geopolymer with water absorption as high as 4–22% by Alomayri et al. [17] and 10–13% by Nazari et al. [18]. Prolonged exposure to water for 28 days appears to have little significant influence on the water absorption properties. Surface hydrophobic modification improves waterproof properties and reduces water absorption of geopolymer repair material. Mechanism of surface hydrophobic modification of specimens was discussed and illustrated in Ref. [13] carried out by the authors of this work.

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