[Construction and Building Materials 109 \(2016\) 17–24](http://dx.doi.org/10.1016/j.conbuildmat.2016.01.043)

Contents lists available at [ScienceDirect](http://www.sciencedirect.com/science/journal/09500618)

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Thermal behavior and mechanical properties of geopolymer mortar after exposure to elevated temperatures

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Mechanical properties of geopolymer mortar at elevated temperatures are quantified.

Thermal behavior of geopolymer mortar under fire conditions is evaluated.

Temperature induced mass loss and shrinkage in geopolymer mortar is evaluated.

article info

Article history: Received 8 July 2015 Received in revised form 3 January 2016 Accepted 26 January 2016 Available online 2 February 2016

Keywords: Geopolymer mortar Metakaolin Fly ash Mechanical property Thermal behavior High temperature

This paper presents results from experimental studies on mechanical properties and thermal behavior of geopolymer mortar, prepared by alkaline solution activating metakaolin and fly ash blend. Bending, compressive, tensile and bond strength tests were conducted on large sets of geopolymer mortar, Portland cement mortar, and commercially used repair mortar specimens at ambient temperature and after exposure to elevated temperatures. Thermogravimetry and differential scanning calorimetry analysis, and dilatometric tests were also carried out on geopolymer paste and mortar. Results from these tests show that geopolymer mortar exhibits higher temperature-induced degradation in bending and tensile strength, but lower degradation in compressive and bond strength than ordinary Portland cement mortar and commercially used repair mortar. Specifically, the bond strength of geopolymer mortar on cement mortar or concrete substrate is close to or even higher than that of commercially used repair mortar throughout $25-700$ °C range. The microstructural damage due to temperature-induced dehydration and dehydroxylation, and thermal incompatibility between geopolymer paste and aggregates is the main reason for the strength degradation of geopolymer mortar at high temperatures.

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1. Introduction

Geopolymer, a new environment friendly inorganic binder, derived by alkaline solution activating aluminosilicate source material (such as metakaolin, fly ash and slag), has attracted significant attention in recent years as a practical alternative to Portland cement [\[1–4\]](#page--1-0). Geopolymer exhibits comparable mechanical properties and durability characteristics as that of Portland cement, but has lower energy requirements and lower greenhouse gas emissions during its production [\[5,6\]](#page--1-0). Therefore, there is a growing research interest in developing viable processes for application of geopoly-mers and its resulting products in construction industry [\[2,7–10\]](#page--1-0).

Cement mortar is a commonly used binder and repair material. Feasibility of geopolymer mortar, as a promising replacement to

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<http://dx.doi.org/10.1016/j.conbuildmat.2016.01.043> 0950-0618/© 2016 Elsevier Ltd. All rights reserved.

cement mortar, has been extensively discussed in literature [\[11–15\]](#page--1-0). Temuujin [\[16\]](#page--1-0) reported that geopolymer binder exhibited strong bonding to sand aggregate. Chi and Huang [\[17\]](#page--1-0) investigated the compressive strength, flexural strength, drying shrinkage and water absorption of alkali-activated fly ash/slag (AAFS) mortars, and their test results showed that with the exception of drying shrinkage, better properties have been obtained in AAFS mortars than that in Portland cement mortar. Pacheco-Torgal et al. [\[18\]](#page--1-0) studied the effect of sodium hydroxide concentration, superplasticizer content and percentage substitution of metakaolin by calcium hydroxide on the workability, compressive and flexural strength of alkali-activated metakaolin based mortars. Ueng et al. [\[19\]](#page--1-0) conducted a series of laboratory tests to determine the deformational moduli and strength parameters of adhesion interface between geopolymer mortar and cement mortar. Vasconcelos et al. [\[7\]](#page--1-0) found that metakaolin geopolymer mortar exhibits a slighter lower adhesion to the concrete substrate than that of commercially available

pre-pack repair mortars, but geopolymer mortar is more costeffective.

The above review from experimental studies clearly show that geopolymer mortar exhibits great promise as a repair material, due to high compressive and flexural strength, high adhesion to ordinary Portland cement mortar and concrete substrate. However, these experimental results were obtained only at ambient temperature. The mechanical characteristics of geopolymer mortar at high temperature must be well understood if geopolymer mortar is to be used as a repair material in buildings where fire resistance is one of the primary requirements. Recently, several researchers discussed the mechanical properties of geopolymer mortar at high temperatures [\[20–23\]](#page--1-0). However, these studies mainly focused only on the degradation of compressive strength of geopolymer mortar after exposure to elevated temperatures. There is a lack of experimental data on other mechanical properties of geopolymer mortar at high temperatures.

A series of mechanical property tests were carried out in this study on geopolymer mortar specimens, to evaluate the residual compressive, bending, tensile and bond strength of geopolymer mortar after exposure to elevated temperatures. Comparative benchmark tests were also conducted on ordinary Portland cement (OPC) mortar and commercially used repair mortar specimens. The thermal behavior of geopolymer mortar, including temperatureinduced mass loss, thermal flow and expansion, were also investigated through thermogravimetry and differential scanning calorimetry (TG–DSC) analysis, and dilatometric tests.

2. Experimental details

Four types of specimens were prepared for undertaking bending, compressive, tensile and bond strength tests of geopolymer mortar on OPC mortar and concrete substrate, at ambient temperature and after exposure to elevated temperatures. Data from these tests is utilized to evaluate residual bending, compressive, tensile and bond strength of geopolymer mortar. Mass loss, thermal flow and thermal expansion were measured by TG–DSC analysis and dilatometric tests, to elucidate the strength degradation mechanism of geopolymer mortar at high temperatures.

2.1. Raw materials

The primary aluminosilicate source material used in preparing geopolymer mortar is metakaolin (MK) and fly ash (FA) blend. Commercially produced metakaolin with an average particle size of 0.017 mm, and low calcium fly ash with an average particle size of 0.032 mm, were sourced from suppliers in China. The chemical composition of MK and FA, as determined by X-ray fluorescence (XRF) analysis, can be referred to Zhang et al. [\[24\].](#page--1-0)

The alkaline-silicate activator with desired SiO_2/K_2O molar ratios of 1.0 were formulated by blending commercial potassium silicate solution with 15.8 wt% K₂O, 24.2 wt% SiO₂ and 60 wt% H₂O(SiO₂/K₂O molar ratio is 2.4), and potassium hydroxide flakes with 95% purity, and tap water. The alkaline-silicate activator was prepared one day prior to use.

For undertaking comparative benchmark tests on Portland cement mortar specimens, ordinary Portland cement (OPC, Grade P.O.32.5) was used. Portland cement of Grade P.O.32.5 was also used for preparing OPC mortar and concrete substrate for bond strength tests. To enhance the strength of OPC mortar and concrete substrate in bond strength tests, and to avoid the cohesion fracture in the substrate materials, polycarboxylate superplasticizer was added in the preparation of OPC mortar and concrete substrate.

The coarse aggregate for concrete substrate consisted of graded gravel with sizes of 5–20 mm and fine aggregates for mortar and concrete consisted of locally available river sand with a maximum size of 2 mm.

Based on the authors' previous test results on OPC mortar specimens, it was found that OPC mortar exhibits lower bond strength with concrete substrate. To further evaluate this trend, one type of commercially used repair mortar, TD-JS polymer-modified cement mortar (provided by a Chinese supplier), was also tested for establishing comparative benchmark data.

2.2. Preparation of geopolymer mortar and cement mortar

Geopolymer mortar (GM) was prepared by using alkaline silicate activator, MK– FA blend precursor (50% MK and 50% FA) and sand. Firstly, alkaline silicate activator was added into MK–FA blend precursor. Then the mixture was mixed in a mixer for 4–5 min. After that, sand was also added and agitation was carried out again for 6 min to get well-mixed GM.

In geopolymer mortar, the ratio of $m_{\text{water}}:m_{\text{solid}}:m_{\text{sand}}$ is 0.45:1:3, where m_{water} is the solvent mass in the silicate solution, m_{solid} is the mass sum of the solute in the silicate solution and MK–FA precursor, and m_{sand} is the mass of sand.

For comparative tests, ordinary Portland cement mortar (CM) and polymermodified cement mortar (PMCM) were prepared. The mass proportion of water:cement:sand is 0.45:1:3 for CM specimens. And the mass ratio of water to the solid mixture of polymer, cement and sand, is 0.13 for PMCM specimens.

2.3. Preparation of specimens

For undertaking bending and compression tests at ambient temperature and after exposure to elevated temperatures (100, 300, 500 and 700 °C), a total of 15 GM specimens with sizes of $160 \times 40 \times 40$ mm were prepared. In addition, three CM specimens and three PMCM specimens with the same sizes were prepared for comparative tests at ambient temperature.

For tensile strength tests, 15 GM specimens with a shape as number "8" were prepared. The dimensions of the steel mould for these ''8-shaped" specimens are shown in Fig. 1. To compare the tensile strength, 15 CM specimens and 15 PMCM specimens with the same sizes were also prepared.

Bond strength of GM on CM substrate was tested on 15 GM–CM composite specimens having a shape of number ''8". Half ''8"-shaped CM specimens were prepared and cured in advance, and then placed in the one halves of the ''8"-shaped steel moulds as the substrate. Fresh GM were cast into the other halves of the steel moulds and then bonded with the CM substrate, as shown in [Fig. 2.](#page--1-0) A small amount of red toner with main ingredients of $Fe₂O₃$ powder was added in fresh GM, to clearly distinguish the GM portion in the GM–CM composite specimens. The authors' exploratory trial tests showed that the addition of the red toner has no significant effect on the mechanical properties of GM. To enhance the strength of CM substrate and to avoid the cohesion fracture at CM substrate, polycarboxylate superplasticizer was added in the formulation of CM substrate. The mass proportion of water:cement:sand:superplasticizer is 0.31:1:3:0.018 for CM substrate. For comparative tests, fresh CM and PMCM was also cast into the one-half of the ''8"-shaped steel moulds respectively and bonded with the prepared CM substrate. These specimens of fresh CM and PMCM bonding to prepared CM substrate (namely CM–CM composite specimens and PMCM–CM composite specimens) were tested at ambient temperature and after exposure to 100, 300, 500 and 700 \degree C, to investigate the bond strength of CM and PMCM with older mortar.

In addition, fifteen composite specimens of Portland cement concrete (CC) with GM interlayer were prepared for testing the bond strength of GM on CC substrate at ambient temperature and after exposure to elevated temperatures. Freshly prepared cement concrete mix was cast into large ''8"-shaped plastic moulds, with wood partitions of 26 mm thickness in the middle. The mass proportion of water: cement:sand:coarse aggregates:superplasticizer in cement concrete 0.31:1:1.5:2.3:0.018. [Fig. 3](#page--1-0)(a) illustrates the sizes of the large ''8"-shaped plastic mould. The wood partition separated the cast concrete into two independent blocks. After 28-day curing at ambient temperature, these concrete blocks were placed back into the plastic moulds as CC substrate, and the wood partitions were taken away. Fresh GM was cast into the gaps left by the partitions and then bonded with the CC concrete, as shown in [Fig. 3](#page--1-0)(b). Fifteen CM–CC composite specimens and fifteen PMCM-CC composite specimens were also prepared, following similar procedure as that of GM–CC specimens, but using CM and PMCM as the interlayer material respectively.

All specimens cast above were cured for 6 days in a cabinet at a constant 22 $^{\circ}$ C temperature and 95% relative humidity, and then taken out to dry naturally in a room for 1 day prior to mechanical property tests. Bending, compressive, tensile and bond strength tests were carried out on these specimens at ambient temperature and after exposure to 100, 300, 500 and 700 °C. Three specimens were tested at each testing temperature from each group of specimens.

Geopolymer powders were prepared for thermogravimetry and differential scanning calorimetry (TG–DSC) analysis through grinding geopolymer paste specimens with the same formulation as that of GM, but without any sand. Mass loss and thermal flow of these powders was evaluated in $25-800$ °C temperature range.

Fig. 1. Dimensions of the steel mould.

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