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Development of metakaolin–fly ash based geopolymers for fire resistance applications

Hai Yan Zhang [a,b,c], Venkatesh Kodur [b,*], Shu Liang Qi [c], Liang Cao [c], Bo Wu [a,c]

[a] State Key Laboratory of Subtropical Architecture Science, South China University of Technology, Guangzhou 510640, PR China
[b] Department of Civil and Environmental Engineering, Michigan State University, East Lansing, MI 48824, USA
[c] Department of Civil Engineering, South China University of Technology, Guangzhou 510640, PR China

HIGHLIGHTS

- A metakaolin–fly ash based geopolymer binder with superior properties is developed.
- Effect of temperature on properties of metakaolin–fly ash based geopolymer is quantified.
- The feasibility of using geopolymers in fire resistance application is demonstrated.

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ABSTRACT

This paper presents results from experiments on geopolymer binders, composing of metakaolin (MK) and fly ash (FA) blend as a precursor, specially developed for fire resistance applications. Bending and compression tests were conducted at ambient temperature and after exposure to high temperatures on three batches of geopolymer specimens. Based on mechanical property tests and thermogravimetric analysis, an optimum MK–FA proportion required in geopolymers for achieving optimum performance both at ambient and high temperatures is developed. Data from strength tests is utilized to illustrate that MK–FA based geopolymer specimens exhibit comparable bending and compressive strength as that of ordinary Portland cement specimens both at ambient temperature and after exposure to high temperatures. Thus, MK–FA based geopolymers offer a feasible alternative to conventional Portland cement in practical building applications.

1. Introduction

Portland cement continues to be the primary material of choice in construction due to its superior thermal, mechanical and durability properties. However, in recent years there is a growing concern on the vast amounts of energy consumption and environment pollution generated during its production. With the mounting pressure to reduce energy demands and polluting gas emissions in all industrial processes in global manufacturing, the pursuit of alternative cementitious materials that can reduce energy consumption and pollution has become an important focus in a number of recent studies.

Geopolymer, a new environment friendly inorganic binder, produced by alkaline solution activating aluminosilicate source material, has attracted spotlight over the past decade with its comparable performance with Portland cement and is regarded as the most promising alternative to Portland cement [1].

Metakaolin (MK) and fly ash (FA) are two types of commonly used aluminosilicate source material for manufacturing geopolymers [2–5]. It is reported in the literature that MK-based geopolymers can exhibit good mechanical properties at ambient temperature, but is susceptible to cracking and strength degradation under high temperature exposure [6]. Compared to MK-based geopolymers, FA-based geopolymers exhibit better high temperature performance [7], but its strength at early-age is much lower than that of MK-based geopolymers and Portland cement [8,9].

Although several studies on geopolymers manufactured through activating MK and FA blends have been conducted, these studies mainly focused on compressive strength of MK–FA based geopolymers at ambient temperature [10,11]. There is very little data on the mechanical properties of MK–FA based geopolymers after high temperature exposure. Such data is critical for evaluating fire performance of structure members, which is a basic design requirement in buildings. Thus, the aim of the current study is to

* Corresponding author. Tel.: +1 517 353 9813; fax: +1 517 432 1827.
E-mail address: Kodur@egr.msu.edu (V. Kodur).

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Table 1
Details of specimens and temperatures for three stages of tests.

Test stage	Specimen type	Test temperature (°C)	Number of specimens
1	Geopolymer pastes with various MK and FA mix proportions	25 and 500	30
2	Geopolymer paste, mortar and concrete with optimum MK and FA mix proportion	25, 100, 300, 500, 700 and 800	18 × 3 = 54
3	OPC paste, mortar and concrete	25, 100, 300, 500, 700 and 800	18 × 3 = 54

develop an optimum MK–FA based geopolymer binder with good fire resistance performance and then to evaluate the effect of temperatures on mechanical properties of MK–FA based geopolymer paste, mortar and concrete.

2. Overview of experimental program

The experimental program was carried out in three stages and consisted of bending and compression tests on a large number of geopolymer paste, mortar and concrete specimens at ambient and after exposure to high temperatures. In Stage 1, a series of bending and compression tests were carried out on geopolymer pastes, with various FA and MK proportions to arrive at an optimum geopolymer mix proportion of metakaolin and fly ash. In Stage 2, a set of geopolymer paste, mortar and concrete specimens were prepared using the optimum geopolymer formulation developed in Stage 1, and were tested to evaluate mechanical properties of geopolymer mixtures at elevated temperatures. In Stage 3, a set of property tests were carried out on ordinary Portland cement (OPC) paste, mortar and concrete specimens and the properties were compared with that of geopolymer specimens to illustrate comparative fire performance of geopolymers. An overview of the test program, including test temperature, type and number of test specimens in each stage is presented in Table 1.

Thermogravimetric analysis (TGA) and thermal expansion measurements were also conducted to develop fundamental understanding on the response of geopolymers at elevated temperatures.

3. Experimental details

Three batches of specimens were prepared for undertaking the three stages of experiments respectively. Each stage of experiments consisted of a large number of bending and compression tests at ambient and after exposure to high temperatures.

3.1. Raw materials

The primary aluminosilicate source material used in preparing geopolymer specimens for property tests 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, is given in Table 2.

The alkaline-silicate activator of the desired composition were formulated by blending commercial potassium silicate solution with 15.8 wt% K_2O, 24.2 wt% SiO_2 and 60 wt% H_2O (SiO_2/K_2O molar ratio is 2.4), and potassium hydroxide flakes with 90% purity, and tap water, to obtain desired SiO_2/K_2O molar ratios of 1.0. Activator solutions were prepared 1 day prior to use, due to high heat released by dissolving potassium hydroxide flakes.

Previous studies have reported that MK-based geopolymers are relatively brittle and prone to cracking, especially under high temperature effect [7,12]. Thus, short carbon fibers (CF) were added to MK–FA blend precursor, as toughening agent, to control cracking and thus enhance bending strength in geopolymer specimens. The length, diameter and density of short carbon fibers are 6 mm, 7 μm and 1.76–1.80 g/cm^3 respectively. A constant mass ratio of 2% of CF to MK–FA precursor was adopted in this study for achieving adequate effect of reinforcement and also good workability.

The coarse aggregate consisted of graded gravel with a maximum size of 20 mm and fine aggregates consisted of locally available river sand. For undertaking comparative tests on Portland cement paste, mortar and concrete specimens, as part of Stage 3 experiments, ordinary Portland cement (OPC, Grade P.O.32.5) was used.

Table 2
Chemical composition of metakaolin and fly ash used in geopolymers.

Chemical	Component (%)	
	MK	FA
SiO_2	51.35	45.30
Al_2O_3	44.24	41.20
Fe_2O_3	0.98	3.18
TiO_2	0.90	1.62
MgO	0.48	0.44
P_2O_5	0.45	0.36
Na_2O	0.16	0.09
CaO	0.13	3.77
K_2O	0.08	0.38
MnO	0.01	0.05
SO_3	–	0.75
SrO	–	0.11
ZrO_2	–	0.08
Loss on ignition	0.72	2.4

3.2. Preparation of specimens

Three batches of specimens were prepared for undertaking bending and compression tests at three stages. The first batch (for Stage 1), consisting of 30 geopolymer paste specimens, was used to study the effect of fly ash content on bending and compressive strength and to arrive at an optimum mix proportion of MK and FA in the precursor of geopolymers. The second batch (for Stage 2) was composed of three types of 54 geopolymer mixture specimens, namely geopolymer paste, mortar and concrete specimens, and was prepared to evaluate mechanical properties at ambient and high temperatures. And the last batch (for Stage 3) included 54 ordinary Portland cement mixture specimens, for undertaking comparative property evaluation.

3.2.1. Geopolymer paste specimens with different content of fly ash

To develop an optimum mix proportion of MK and FA in the precursor, geopolymer paste specimens with varying FA content were prepared. Five combinations of MK–FA based geopolymers with mass ratios of FA/(MK + FA) of 0%, 20%, 50%, 75% and 100% were considered.

Geopolymer pastes with lower FA and higher MK content have higher liquid demand to achieve similar workability than those with higher FA and lower MK content, due to fly ash having spherical shape particles and larger particle size as compared to MK which has plate-like structure and finer particle size [7]. In this study, a solid-to-liquid ratio of 0.8 and 1.1, representing mass ratio of MK–FA blend precursor to alkaline activator solution, was adopted for the geopolymer pastes with lower FA content (0% and 20%) and higher FA content (50%, 75% and 100%) respectively. The solid-to-liquid ratio of 0.8 and 1.1 corresponds to water-to-solid ratio of 0.5 and 0.4 respectively, which denotes the mass ratio of solvent in the silicate solution to the sum of solute in the silicate solution and MK–FA precursor.

Geopolymer pastes were prepared by hand-mixing MK–FA precursor and short fibers for 2 min, then adding alkaline silicate solution and mixing all ingredients in a mixer for 12 min. Subsequently, geopoloymer pastes were cast into a triplet steel mould with each specimen of size of 160 × 40 × 40 mm and then vibrated on a shake table to remove any air bubbles. After covering with plastic films, specimens were cured in a constant temperature and humidity tank.

The detailed mix proportions of 5 groups of geopolymer paste specimens (GP00, GP20, GP50, GP75 and GP100) with different FA content were listed in Table 3. The designation GP denotes geopolymer specimen, and the numbers ("00") represent the content of FA. Three specimens are tested at each temperature (ambient temperature and after exposure to 500 °C) for each group.

3.2.2. Geopolymer paste, mortar and concrete specimens

Based on results of Stage 1 experiments on geopolymer paste specimens, a fly ash content of 50% was deemed to be the optimum mix proportion. Thus this formulation was adopted to prepare the second batch of specimens for Stage 2 experiments.