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Elevated temperature properties of basalt microfibril filled geopolymer composites

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HIGHLIGHTS

- Basalt microfibrils (BMF) obtained by mechanical surface activation of basalt fibrous wastes.
- Effect of BMF loading on elevated temperature properties of geopolymers.
- Less pore area observed in higher BMF loaded geopolymer composites.
- Minimum compression strength loss in 10 wt% BMF loaded geopolymer composites.

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ABSTRACT

The present work deals with elevated temperature properties of basalt microfibril filled geo-polymer composites when exposed to 200, 400 and 800 °C. The basalt microfibrils were obtained after 30 min dry pulverization of basalt fibrous wastes. Further, the microfibrils were added into geopolymer synthesized from calcined kaolin and shale clay residues under 5, 10 and 15 wt% loading. The prepared composites were evaluated for physical properties, microstructural analysis and compression strength before and after exposure to elevated temperatures. The compression strength of basalt microfibril filled geopolymers was found higher than the neat geopolymer due to their increased thermal resistance and dense microstructures. The geopolymer composite of 10 wt% basalt microfibrils maintained the residual compressive strengths of 23.13 and 16.08 MPa at 400 °C and 800 °C, respectively and thus recording a minimum strength loss of 32 and 43%, respectively.

1. Introduction

Over the last few decades, the fiber reinforced concretes have been increasingly developed to get superior tensile, flexural and post-cracking ductility properties [1,2]. The reinforcement of asbestos, cellulose, steel, polypropylene and glass fibers showed drastic improvements in mechanical properties of cement products at ambient temperature [3,4]. However, when exposed to elevated temperatures in fire accidents, their strength was reported to decrease significantly due to micro structural damage through cracking. The different types of fibers have been studied in previous studies to overcome this deficiency, but many of them either lack structural strength or durability, or extremely costly for use in moderate loadings [1–4]. Therefore, further research is necessary to identify an alternative binder and fiber reinforcements which possesses good thermal resistance and maintain higher residual mechanical properties when exposed to elevated temperature [5].

Geopolymer has recently emerged as novel engineering binder material with environmentally sustainable properties [6]. The reduced energy consumption and CO_2 emission during manufacture of geopolymers make them attractive alternative over Portland cement [7–9]. Geopolymers are synthesized by reacting aluminosilicate source materials (i.e. fly ash, slag, metakaolin, etc) with highly alkaline activators. This results in dissolution and release of free $[SiO_4]^-$ and $[AlO_4]^-$ tetrahedral units in solution, which alternatively links to polymeric precursor by sharing oxygen atom. In this way, unique three-dimensional oxide network structures of Si—O—Al—O bonds can be formed in geopolymers [10,11]. It has advantages of high early strength, low in density, low permeability, excellent resistance to chemical attacks, good freezing-thawing cycles, light in weight, excellent in volume stability, and resistant to fire as well as to erosion [12,13]. Furthermore, the microstructures and mechanical properties of geopolymers depend on the chemical composition, mineralogical composition,

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morphology, fineness and glassy phase content present in aluminosilicate sources [14]. For instance, metakaolin based geopolymer offers better strength and permeability, however with some drawbacks of poor rheological properties, complex processing, higher water demand, accelerated hydration reactions [15]. The fly ash-based geopolymer is more durable and stronger than that of metakaolin-based geopolymer, but it has limitations of extended setting times, slow strength development, low early-age strength, construction delay, etc [16]. The slag-based geopolymer is advantageous for high early strength and greater acid resistance, but requires frequent maintenance of equipments and costly processing than metakaolin and fly ash [17,18].

Due to melting of polymeric fibers when exposed to elevated temperature, the use of inorganic fibers has developed significant interest in fiber reinforced concrete applications [19]. The several researchers discussed the mechanical properties of glass fiber filled geopolymer mortar at high temperatures, but the interest in using mineral basalt fibers has grown recently [20,21]. The basalt fibers are easy to process, non-toxic, natural, eco-friendly and inexpensive as compared to other inorganic fibers. They have extremely good modulus, high strength, improved strain to failure, high temperature resistance, excellent stability, good chemical resistance and reduced thermal and electrical conductivity [22]. The basalt fibers are prepared from volcanic rocks originated from frozen lava, with a melting temperature comprised between 1500 and 1700 °C. The fibers are chemically composed of pyroxene, clinopyroxene, olivine, and plagioclase minerals. Moreover, they are chemically rich with oxides of silica and alumina along with traces of magnesium, calcium, sodium, potassium, silicon and iron [23].

A lot of researches have been performed on continuous basalt fiber or basalt fabric as a strengthening material for cementitious concrete structures, however there are limited studies on the effect of short basalt fibers on the properties of geopolymers. The objective of this investigation was to study the microstructure and mechanical properties of basalt microfibril (BMF) reinforced geopolymers at elevated temperatures. The basalt microfibrils were obtained by mechanical activation of basalt fibrous wastes in ball milling process. The geopolymer composites were prepared by addition of 5, 10 and 15 wt% of basalt microfibrils and later exposed to the elevated temperatures of 200, 400, and 800 °C. Further, the basalt microfibril/geopolymer composites were evaluated for physical properties, microstructural analysis and compression strength.

2. Experimental methods

2.1. Materials

The short basalt fibrous waste was obtained from the VEBA Industries, Czech Republic. The basalt fibers had density of 2.65 g/cm^3, elastic modulus of 95 GPa, tensile strength of 4000 MPa, elongation at break of 3% and water absorption of less than 0.5%. The chemical composition of basalt fibers as measured from elemental analysis is shown in Table 1.

The Baucis L110 alumino-silicate geopolymer binder based on metakaolin was obtained from Ceske Lupkove Zavody, Czech Republic along with sodium alkali activator. The metakaolin geopolymer was synthesized from calcined kaolin and shale clay residues with Si/Al ratio of 2.0. The kaolin was mainly composed of kaolinite with small amounts of quartz, whereas shale clay was composed of kaolinite with low amount of quartz and anatase. At first, kaolin and shale clay were passed in rotary klin to result in 30–70% loss of kaolinitic structure due to dehydroxylation. Later, it was converted to metakaolin by additional calcinations at 750 °C for 10 h in bath oven. The chemical composition of the metakaolin geopolymer was as follows (wt%): SiO_2 47, Al_2O_3 24, LOI 0.5, Fe_2O_3 0.50, TiO_2 0.8, MgO 3.5, K_2O 0.40, CaO 17.50. The mean particle size (d50) was 5 µm. The sodium alkali activator was mixture of Na_2SiO_3 and NaOH.

2.2. Preparation of basalt microfibrils

The short basalt fibrous waste was dipped in acetone for 24 h to remove the surface finish and impurities. They were later transferred to high energy planetary ball milling machine Fritsch pulverisette 7, Germany for mechanical activation of surface and grinding to the scale of basalt microfibrils. The dry pulverization was carried out based on previous research experience [24–26]. The sintered corundum container of 80 ml capacity and zirconium balls of 10 mm diameter were chosen for 30 min of dry milling. The ball to material ratio was kept at 10:1 and the speed was kept at 850 rpm.

Particle size distribution of dry milled basalt particles was obtained from Malvern zetasizer nano series based on dynamic light scattering principle of Brownian motion of particles. Deionized water was used as dispersion medium and it was ultrasonicated for 5 min with bandelin ultrasonic probe before characterization. In addition, microstructure of basalt particles was observed on scanning electron microscope (SEM) of Hitachi-model TM-3000 at accelerated voltage of 15 kV.

2.3. Preparation of basalt microfibril/geopolymer composites

The four parts of alkali activator and five parts of metakaolin based geopolymer were manually mixed for 10 min to ensure homogeneous preparation of geopolymer binders keeping the ratio of Na_2O/Al_2O_3 equal to unity. For preparation of basalt microfibril filled geopolymer composites, the fibrils were initially pre-dried for 60 min at 70 °C in an oven. Next, the basalt microfibrils were added into the prepared geopolymer binder at 5 wt%, 10 wt% and 15 wt% loading. The mixing was homogeneously done in Hobart mixer for 5 min. Subsequently, the fresh prepared composite mortar was poured into 40 mm cubic-shaped moulds, vibrated for 2 min on the vibration table to remove air voids and wrapped using a thin plastic sheet to prevent water evaporation. The wrapped samples were demolded after 24 h of casting and then cured at room temperature (20 ± 2 °C) and a relative humidity of (70 ± 10%) for 28 days.

2.4. Exposure to elevated temperature

After curing of samples at age of 28 days, the dried geopolymer composites were exposed to elevated temperatures of 200, 400 and 800 °C. The specimens were placed into a furnace (Elektrické Pece Svoboda, Czech Republic) and heated at fixed heating rate of 5 °C/min. As soon as the target temperature was attained, it was

Table 1
Elemental analysis of basalt fibers.

Element	Carbon	Oxygen	Sodium	Magnesium	Aluminum	Silicon	Potassium	Calcium	Iron
Weight%	18.81	42.41	0.56	1.04	5.39	14.79	0.97	5.72	10.27