



# Mechanisms of interfacial bond in steel and polypropylene fiber reinforced geopolymer composites



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## ABSTRACT

The fiber-matrix interface properties play an important role in determining the overall mechanical performance of the fiber reinforced brittle matrix composites. This study is to evaluate the effects of wettability, chemical characterization and nanometric roughness of two different fibers, polypropylene (PPF) and micro steel fibers (MSF), superposed by shrinkage of fly ash based geopolymer binder on fiber-matrix interaction and the consequent mechanical properties of the corresponding composites. The fiber incorporation in geopolymer matrix was performed by 0.5, 1, 2, 3 and 4% of the total volume of each type of fibers. The results show that MSF has strong contact with the geopolymer paste since it tends to behave as a hydrophilic material that resulted in significant improvement in energy absorption and flexural strength of the composite; while PPF led to fiber-matrix debonding due to hydrophobic characteristics of the fiber compounds by geopolymer shrinkage that weakened the mechanical performances of the composites.

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## 1. Introduction

Geopolymers are aluminosilicate inorganic polymers formed by polymerization of aluminosilicate precursors with alkaline solutions which have several desirable characteristics compare to conventional binders from the aspects of mechanical properties, thermal stability and durability [1–3]. Despite their outstanding properties, similar to other ceramic-like materials such as Portland cement based products, they show less resistant to tensile and flexural loading than compressive and suffer from sudden failure [4,5]. On the other hand, only a small portion of water known as “interstitial or structural water” has been incorporated into the geopolymerization while extra water demanded for producing a workable mixture, called “free water” [6–8]. The loss of such a free water leads to a considerable shrinkage deformation of the geopolymer matrix [9,10]. Therefore, quasi-brittle characteristics of geopolymers compound by the influence of shrinkage over time

and particularly at early ages.

Incorporation of fibers into the cementitious matrix is a well-known method to enhance the flexural properties and post peak characteristics of corresponding composites since fiber controls the crack propagation and widening under different type of mechanical loading or shrinkage [11]. In short fiber composite, fiber-matrix interface characteristics are significantly important in controlling the fiber debonding and pullout process that governs the stress-crack opening relationship and nonlinear behavior of such a material [12]. Generally, debonding, sliding and pulling-out of the fibers are the local mechanisms that enhance the bridging action during both micro and macro cracking of the matrix; this mechanism increases the demand of energy for the crack to propagate [13].

Fibers of different materials and geometric properties are used in constructional application which can be divided into two major categories of high modulus (metallic) and low modulus (non-metallic) [14]. Each category enhances particular properties of the matrix. Generally incorporation of metallic fiber result in flexural strength enhancement due to their higher stiffness while non-metallic fibers control the plastic shrinkage of the matrices since they have a higher aspect ratio and surface contact area [15]. Steel

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and polypropylene are used extensively as a metallic and non-metallic fibers in fiber reinforced concrete applications [16]. Incorporation of PPF is claimed to enhance concrete's performance due to the high impact resistance; increased strain to failure; a fine cracked free finish; more water permeable resistant and consequent improved durability [17]. The inclusion of steel fibers to conventional reinforced concrete improves the cracking strength and restricts the cracks' growth and consequently a smaller crack widths [18]. However, the incorporation of large scale fibers in cementitious matrices is limited due to extensive workability reduction; and it is supposed that increasing the interaction surface of fiber and matrix by reducing the scale of fiber is an effective way to enhance the composite mechanical properties beyond the improvement of conventional fibers [19,20].

The performance of fibers in geopolymer matrices is highly dependent on the inherent properties of the fibers, fiber content, geopolymer precursors, curing conditions and age of the composites [10,13]. However, fiber/matrix interface play the key role in the overall mechanical properties of composite structures which a strong contact interface has a potential to transfer load from the matrix to fibers with high load bearing capacity; while, fibers with inert surfaces result in a weak interfacial contact causes interface debonding based failure of composite [21]. It is reported that the rough surface of graphene nanoplatelet increases the contact area between the geopolymer matrix and the reinforcers, enhancing the mechanical properties by corresponding composite because of a higher pull out and debonding energy. The dispersion and contact of the fiber and geopolymer matrix were reported as the major factors affecting the mechanical mechanism of the graphene incorporated geopolymer composites [13]. Geopolymers showed strong bond with steel reinforcement and for an equivalent compressive strength, the geopolymer concrete bond strength was observed to be about 10% higher than that of the OPC concrete [22]. A clear boundary between the surface of the steel and hydrated phases in fly ash based geopolymer mortar was observed through the EDS and nanoindentation analysis [23]. Incorporation of wollastonite microfibers showed better reinforcing efficiency on geopolymers cement composites than Portland cement based as the nature of the bond between fiber and the geopolymer matrix as the result of the toughening mechanism of pull-out and debonding [24].

As geopolymer paste is water-based, a strong bond between fibers and matrix can be measured by wettability of the reinforcement by water. Good wettability which is defined by a low contact angle, helps the development of a strong bond and the degree of effectiveness of a reinforcer in a geopolymer composite [13,25].

This study compares the influence of variable dosages of two types of conventional fibers used in constructional material, PPF and MSF, on controlling of shrinkage and mechanical properties of the fly ash (FA) based geopolymers. The geopolymer matrix was reinforced by different MSF and PPF content in the range of 0.5%, 1%, 2%, 3% and 4% of the volume. Chemical characterization, atomic force microscopy, water contact angle measurement and FESEM analysis <http://www.aif.ncsu.edu/equipment/field-emission-scanning-electron-microscope-fei-verios-4601/> have been conducted to evaluate the fiber-matrix bond in a hardened state. Drying shrinkage over time, flexural and compressive strength and toughness of the corresponding material were measured to investigate the mechanical properties.

## 2. Material characteristics and testing methods

### 2.1. Material characterization

To prepare geopolymer paste, the low calcium FA was used as per the ASTM: C618-12a which collected from Lafarge Malayan Cement Bhd (Malaysia). The chemical composition was determined by X-ray fluorescence (XRF) using a PANalytical Axios mAX instrument and is shown in Table 1. The median particle size and specific gravity of the fly ash were 12.19  $\mu\text{m}$  and 2.18  $\text{g}/\text{cm}^3$ .

Combination of sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) and sodium hydroxide (NaOH) solutions was used as alkaline activator. NaOH was provided in the form of pellets with 99% purity from Merck (Germany). A 16M NaOH solution was prepared by using deionized water for the geopolymer mix design.  $\text{Na}_2\text{SiO}_3$  was obtained from (PC Laboratory Reagent) and used in liquid form with about 1.5 gr water per milliliter at 20 °C with a modulus ratio of 2.5 ( $\text{SiO}_2/\text{Na}_2\text{O}$ ,  $\text{SiO}_2 = 30\%$  and  $\text{Na}_2\text{O} = 12\%$ ). Physical and mechanical properties of the MSF and PPF have been provided in Table 2.

### 2.2. MSF and PPF geopolymer composite preparation

Due to the different properties of MSF and PPF, different methods were performed to produce MSF and PPF reinforced geopolymer composites.

NaOH,  $\text{Na}_2\text{SiO}_3$  and deionized water have been mixed in order to get a mass ratio of  $\text{Na}_2\text{SiO}_3:\text{NaOH}:\text{H}_2\text{O}$  of 2.5:1.0:0.7; the corresponding alkali activator solution was mixed with FA with the solution to solid ratio of 0.5 for 5 min to produce a workable geopolymer paste. MSF was gradually added to the mixture and kept mixing for 3 min more time to make a homogenous fibrous matrix. However, for PPF different method was used because multi filament fibers could not disperse homogeneously in the matrix. Therefore, PPF was mixed with the alkali activator solution and stirred until a uniform dispersion obtained. After that, the FA was added to the mixture gradually and mixed for 5 min.

Either MSF or PPF geopolymer matrices were poured immediately into stainless steel molds, vibrated and cured at 65 °C in a Memmert ULM600 oven for 24 h. Then the samples were dismantled and kept in ambient condition with an average temperature and humidity of 32 °C and 65%, respectively, until the day of testing day. The fiber content in geopolymer paste varied in the range of 0.5%, 1%, 2%, 3% and 4% of the volume.

### 2.3. Shrinkage measurement

Prisms with the size of 25 × 25 × 300 mm were prepared to measure the shrinkage of the composites over time. After the hot curing period in the oven and dismantling of the molds, demec points were fixed to the surfaces of the specimens by using Araldite 5-Minute AB Epoxy Adhesive. The variation of shrinkage over time was measured by Mitutoyo Absolute Digimatic Indicator ID-C112B apparatus with the range and resolution of 12.7 mm and 0.001 mm, respectively. The shrinkage measurement was recorded until 56 days for all samples.

### 2.4. Bulk density

Bulk density was measured by the Archimedes method in accordance with ASTM C-20 after 60 days. The measurement was first done for suspended and saturated conditions and then for dried condition. These sequences have been suggested by the standard to avoid the effect of crack formation during the drying. The saturated and suspended weight measured after 2 h boiling was followed by immersing specimens in deionized water in

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