# Wood Flour's Effect on the Properties of Geopolymerbased Composites at Different Curing Times

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Fly-ash (FA) /wood-flour (WF) geopolymer composites (FWGCs) were prepared to investigate the influence of WF on the properties of FA-based geopolymer composites at different curing times. The crystallization, surface morphology, geopolymerization, interface analysis, and mechanical properties were characterized. The results indicated that the curing time exerted positive effects on the mechanical strength of the FWGCs. Noticeably distinct microstructures and mechanical properties were observed with different WF contents. The FWGCs with low WF loading (1 wt% and 5 wt%) presented almost unchanged or even improved mechanical properties compared to the pure FA-based geopolymer due to the existence of bonds between the WF and geopolymer matrix in the interface. However, the addition of WF to a higher content (10 wt%, 15 wt%, and 20 wt%) posted a negative influence on mechanical properties with insufficient polymerization of geopolymer and degradation of WF detected by morphology and elemental microanalysis. This study will facilitate a better understanding of the interaction between geopolymers and wooden materials, and serve as a basis for further research and applications.

Keywords: Fly ash; Geopolymer; Wood flour; Composites; Interface

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### INTRODUCTION

The reduction of CO<sub>2</sub> emissions has recently become a global concern. The use of Portland cement has been responsible for the main global warming effects in the construction industry. Increasing environmental awareness has led to placing a focus on searching for an environmentally friendly alternative to Portland cement (Kabir *et al.* 2017; Lirer *et al.* 2017; Duan *et al.* 2017). Geopolymers have emerged as a new generation of alternatives. They are obtained as amorphous to semi-crystalline substances with a threedimensional framework structure and are mainly produced *via* activation by an aqueous solution of alkali silicate material, such as reactive aluminosilicates, metakaolin, or fly ash (FA) (Zhang *et al.* 2015; Ribeiro *et al.* 2016; Zawrah *et al.* 2016). Fly ash is an industrial byproduct that is the most common source for geopolymer production due to its abundance all over the world (Arulrajah *et al.* 2017; Narattha and Chaipanich 2018). Compared with Portland cement, the manufacturing of FA geopolymers is desirable as it contributes to the transformation of waste into useful construction material with a lower carbon footprint (Hoy *et al.* 2016; Ribeiro *et al.* 2016; Chindaprasirt and Rattanasak 2017). As a structural material, FA geopolymers possess a high compressive strength, low shrinkage ability, and excellent durability properties in terms of enhanced inflammability and acid resistance (Horpibulsuk *et al.* 2015; Chuah *et al.* 2016; Mehta and Siddique 2017; Okoye *et al.* 2017). Thus, geopolymers can be used to fabricate structural components, concrete columns, coatings for concrete, railway sleepers, toxic (or radioactive) immobilization, and contaminant encapsulation (Madheswaran *et al.* 2014; Chuah *et al.* 2016; Ozbakkaloglu and Xie 2016; Tahri *et al.* 2016; Aguirre-Guerrero *et al.* 2017; Nikolić *et al.* 2017).

However, similar to most ceramics, pure geopolymers suffer from brittleness issues, low fracture toughness, and low tensile strength (Alomayri *et al.* 2014; Alomayri *et al.* 2015; Ribeiro *et al.* 2016). The incorporation of short fibers into the matrix reduces the brittleness of the composites (Alomayri *et al.* 2014). Previous publications have shown that natural fibers (sisal fibers) can be incorporated into cement to produce composites (Wei *et al.* 2016). Metakaolin-based geopolymers reinforced by bamboo fibers showed excellent qualities in sustainable structural applications (Ribeiro *et al.* 2016). Woven cotton fabric-reinforced composites with fabrics placed in a horizontal orientation presented even better mechanical strength (Alomayri *et al.* 2014).

Wood flour (WF) is a low-cost industrial biowaste material with a low density that is obtained from the timber industry and can be used as natural fiber reinforcement (Petrillo *et al.* 2016). It is an important candidate for short fiber reinforcement that facilitates the decrease in the weight of geopolymer composites. Moreover, WFs utilization would reduce adverse environmental impacts and enhance the application of local materials (Ribeiro *et al.* 2016).

This study investigates the synthesis of FA-based geopolymer composites of mixed composition using a series of WF contents with different curing conditions. In addition, the influence of WF content and curing time was determined on the following properties of FA/WF geopolymer composites (FWGCs): crystallization, mechanical strength, surface morphology and elemental microanalysis, and the interface between the FA geopolymer matrix and WF. The results of this study will facilitate a better understanding of the interaction between geopolymers and wooden materials, and serve as the basis for further research and applications.

### EXPERIMENTAL

#### Materials

Poplar (*Populus* L.) flour (60- to 80-mesh) as a WF type with a moisture content of 2.16 wt% at 25 °C  $\pm$  2 °C was bought on the market (Xingdong Wood Powder Co. Ltd., Xuzhou, China). Low-calcium FA (Class F), passing through a 200-mesh sieve was supplied by Yulian Energy Group Co., Ltd. (Gongyi, Henan, China) and applied as an aluminosilicate source of the geopolymer.

#### Activator for geopolymerization

The activator for geopolymerization was a mixture of sodium hydroxide (NaOH) solution and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) solution with a Na<sub>2</sub>SiO<sub>3</sub>/NaOH (water glass) volume ratio of 2.5. The molar ratio (SiO<sub>2</sub>/Na<sub>2</sub>O) of the activator solution was maintained

at a value of 1.33 by use of 10 M NaOH. The Na<sub>2</sub>SiO<sub>3</sub> solution with Na<sub>2</sub>O  $\geq$  12.80 wt%, SiO<sub>2</sub>  $\geq$  29.20 wt%, H<sub>2</sub>O = 58 wt%, and molar ratio SiO<sub>2</sub>:Na<sub>2</sub>O = 2.31 (Wuxi Yateks Joint Chemical Industry Co., Ltd., Jiangsu, China). The NaOH pellets were of analytical grade (Xilong Scientific Co., Ltd., Shantou, China) and used as received.

## Composite preparation

Different contents of WF (1 wt%, 5 wt%, 10 wt%, 15 wt%, or 20 wt%) were first mixed with FA in a plastic sealed bag at room temperature. Activator solution (250 g) was then added to the prior mixture (500 g) to obtain the mortars. Water was added to maintain consistent viscosity in all mortars. The mortars were stirred and cast onto the open Teflon molds at an ambient temperature for 24 h and were then dried at 80 °C for 48 h. Before testing, the specimens were finally sealed into plastic bags for 0 d, 7 d, and 28 d at room temperature. The designated sample codes of the specimens are listed in Table 1. The different curing times was indicated by adding a suffix "-0 (7 or 28)" at the end of each of the samples, such as FWGC-1-0, FWGC-5-7, and FWGC-15-28, when mentioned in this paper.

Sample Code	FA (g)	WF (g)	Activator (g)	Water (mL)
FWGC-0	500	-	250	-
FWGC-1	495	5	250	10
FWGC-5	475	25	250	50
FWGC-10	450	50	250	150
FWGC-15	425	75	250	200
FWGC-20	400	100	250	350

Table 1. Nomenclature for the Obtained FWGCs

## Methods

### X-ray fluorescence (XRF)

The chemical compositions of the specimens were measured by an XRF spectrometer (XRF-1800, Shimadzu, Tokyo, Japan), and Rh K $\alpha$  and L $\alpha$  radiation were established with an excitation voltage of 40 kV and an electron current of 95 mA.

### Specific surface areas (SSA) and particle size distribution (PSD)

The SSAs of FA and WF were calculated by the multi-point Brunauer-Emmett-Teller (BET) method (Quantachrome Nova 2000 e, Quantachrome Instruments, Boyton Beach, FL, USA). The PSD of FA and WF were dispersed in water using a Mastersizer 2000 particle size laser analyzer (Malvern Instruments Ltd., Malvern, UK) with a Hydro 2000 MU as a dispersion unit.

## X-ray diffraction (XRD)

The XRD patterns of FA, WF, and FWGCs were determined with a Bruker D8 ADVANCE X-ray diffractometer (Bruker, Karlsruhe, Germany), using Cu K $\alpha$  radiation ( $\lambda$  = 1.5406 Å).

#### Scanning electron microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDS)

The images and element analysis of the cross-section of FWGCs were examined by SEM (Quanta FEG 250, FEI, Eindhoven, Netherlands) equipped with an EDS detector under high vacuum. Before testing, the samples were coated with gold using an auto-fine coater (MC1000, Hitachi Co., Ltd., Tokyo, Japan). At least three SEM images per specimen were analyzed. The most representative one of each specimen was presented in this paper.

### Density and mechanical tests

The specimens with the dimensions of 80 mm  $\times$  20 mm  $\times$  10 mm were prepared for the flexural strength tests. The densities of the specimens were calculated before the mechanical tests, and the results for each specimen were the average of at least five repeated measurements. Three-point bend tests were used to evaluate the flexural strength according to the ASTM D790-03 (2003). The testing data were recorded using an Instron 3365 Universal Testing Machine (Instron, Norwood, MA, USA) with a 1 kN load cell at a speed of 1 mm/min. The flexural strength ( $\sigma F$ ) was calculated using the following equation,

$$\sigma F = 3FL / 2bh^2, \tag{1}$$

where F is the maximum load of the specimen (N), L is the specimen span (64 mm), b is the width of the sample (mm), and h represents the sample thickness (mm).

The compressive strength determination, based on the ASTM C109/C109M-12 (2012), of the specimens (38 mm  $\times$  20 mm  $\times$  10 mm) was conducted on a MMW-50 mechanical testing machine (Jinan Resistance Test Machine Co., Ltd., Shandong, China) equipped with a 50 kN load cell. A speed of 1 mm/min was applied. The compressive strength ( $R_C$ ) was determined by the following equation,

$$R_{\rm C} = F_{\rm C} / A, \tag{2}$$

where  $F_{\rm C}$  is the maximum load on the specimen at failure (N) and A is the surface area of the specimen (mm<sup>2</sup>).

All mechanical tests per sample were repeated at least five times, and the average values were reported.

## **RESULTS AND DISCUSSION**

### **Characterization of Raw Resources**

The component composition of poplar wood is approximately 50% cellulose, 30% hemicelluloses, and  $\leq 20\%$  lignin (Balatinecz and Kretschmann 2001; Kim *et al.* 2011). The XRD patterns of poplar wood are shown in Fig. 1a. The noticeable peak of the cellulose crystal occurred near 22° and 35°, while the diffraction peak of the amorphous region in wood was observed around 15° (Yu *et al.* 2011; Johar *et al.* 2012). The specific SSAs of WF were 1.304 m<sup>2</sup>g<sup>-1</sup>. The chemical composition of FA was determined by XRF, and the values of the loss on ignition (LOI) of FA are presented in Table 2. According to this data, FA was composed of aluminosilicates, iron oxides, and minor oxides (Gharzouni *et al.* 2016). The value of the major active ingredients (silicon dioxide, SiO<sub>2</sub>, aluminum oxide, Al<sub>2</sub>O<sub>3</sub>, and iron (III) oxide, Fe<sub>2</sub>O<sub>3</sub>) for geopolymerization and CaO<sub>2</sub> were 88.89% and

4.27%, respectively. In addition, the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio of FA was recorded as 2.16. A crystalline composition of 60.5% quartz, 36.1% mullite, and 3.4% calcite was also established in the XRD patterns of FA (Fig. 1 b), which was consistent with the major components of FA, such as alumina and silica, confirmed by XRF. The specific SSAs of FA were calculated by the BET method as 2.467 m<sup>2</sup>g<sup>-1</sup>. The PSD values of the FA and WF, denoted as  $d_{10}$ ,  $d_{50}$ , and  $d_{90}$ , are presented in Table 3. The particle size of FA was much smaller than WF.

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	K <sub>2</sub> O	TiO <sub>2</sub>	Na <sub>2</sub> O
FA (%)	56.61	26.22	6.06	4.27	2.50	1.10	1.07
Oxide	MgO	SO₃	P <sub>2</sub> O <sub>5</sub>	SrO	MnO	ZrO <sub>2</sub>	LOI
FA (%)	1.05	0.50	0.37	0.09	0.09	0.08	5.92
LOI: Loss on ignition from 25 °C to 1000 °C							

Table 2. Chemical Composition and LOI of FA



Fig. 1. XRD patterns of (a) WF and (b) FA

Sample Code	<i>d</i> 10 (μm)	<i>d</i> ₅₀ (μm)	<i>d</i> 90 (μm)
FA	4.881	26.894	69.238
WF	53.665	79.794	468.978

Table 3.	The	<b>d</b> 10,	<b>d</b> 50,	and	<b>d</b> 90	Values	of FA a	and	WF
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#### **Effects on Crystallization**

The XRD patterns of FWGCs (day 28) are shown in Fig. 2. In general, a broad reflection in original FA ( $2\theta = 24^{\circ}$ ) shifted to  $2\theta = 26^{\circ}$  in the FWGCs, resulting from the transformation of the amorphous component in the geopolymer matrix. Similar results were also reported for FA geopolymer (Phoo-Ngernkham *et al.* 2014) and metakaolin geopolymer (Hajjaji *et al.* 2013). In general, the major activation products of FA geopolymers present were amorphous with a certain amount of unreacted mullite and quartz (Zhang *et al.* 2015). In this study, the composition (mainly composed by Si, Al, and Na) of an amorphous FA geopolymer was the dominant product, which corresponded to approximately 63% of all samples. By the increase in WF contents, the intensity at  $2\theta = 22^{\circ}$  increased due to cellulose in WF (Fig. 1a). The XRD patterns of FWGC-15 with curing times from 0 d to 28 d are illustrated in Fig. 2b. The intensities of the XRD peaks of CaCO<sub>3</sub> (around  $2\theta = 23^{\circ}$ ) clearly increased over time, which might have been a result from the chemical reactions between the Ca<sup>2+</sup> in the FA and CO<sub>2</sub> in the air during the curing stage. It seemed from the XRD patterns that the curing time had seldom influence on the crystallization of FWGCs.





**Fig. 2.** XRD patterns of samples: (a) the specimens with different WF contents (day 28) and (b) the specimens at different curing times (WF content is 15 wt%)

#### **Effects on Morphology**

The microstructure of FWGCs was analyzed by SEM in this study (Fig. 3). Typical features of FWGC-0 were detected in Figs. 3a through 3c, with some small dissolved FA particles co-existing with a part of the unreacted spheres distributed in the geopolymer gel (Temuujin *et al.* 2011). In addition, many of the crystalline phases of the FWGC-0 depicted in Figs. 3a through 3c were detected in the SEM micrographs and in the XRD analysis. The FA is known to be composed of a large proportion of particles containing hollow spheres. The larger proportion of the geopolymer gel indicated that a great amount of FA was dissolved into the matrix (Rickard *et al.* 2011).

The content of wood flour had a great influence on the morphology of geopolymerbased composites. Low-WF geopolymer composites (FWGC-1) were observed to be primarily composed of partially reacted FA particles bonded by the geopolymer gel on their surface. Shorter needle-shaped crystals (mullite crystals) with approximately homogeneous distribution in the geopolymer gel are shown in Fig. 3f. In addition, less pores appeared in the geopolymer matrix than in that of the pure FA geopolymers. Remarkably different structures were observed in the SEM images of the FWGCs with high percentages of WF (Figs. 3g through 3i). A great amount of unreacted FA spheres with a low level of geopolymerization aggregated together were partially attached on the WF surface. Meanwhile, the typical morphology of WF and the interface with the geopolymer matrix could not be observed. A rough and discontinuous surface of WF was detected, especially in the Fig. 3i, due to WF degradation caused by the alkaline condition, as evident in the SEM micrograph images.

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Fig. 3. SEM and Si/Al ratio of the FWGCs

The microstructures of FWGCs at different curing times (0 d, 7 d, and 28 d) are also depicted in Fig. 3. The content of mullite crystals observed in the FWGC-0 was augmented by prolonging the curing time, which revealed the presence of a denser structure. Nevertheless, the crystalline shape of mullite remained almost the same with different curing times, which was in agreement with the XRD results.

### Effects on Geopolymerization

The degree of geopolymerization can be noticeably affected by the Si/Al ratio, as established previously (Pimraksa *et al.* 2011). In this study, an EDS analysis was used to determine the Si/Al ratio of the geopolymer matrix produced. The values of the Si/Al ratio (element weight percentage) in typical FWGCs are shown in Fig. 3. As expected, the Si/Al ratio in FWGC-0-28 was higher than that in FWGC-1-28, which indicated better polymerization of the geopolymer in the pure FA geopolymer. However, the typical amorphous geopolymer area was almost impossible to observe as the WF content increased (Figs. 3g through 3e). The Si/Al ratio of the specimens obtained after curing for 0 d, 7 d, and 28 d are presented in Fig. 3. A growing degree of geopolymerization was evidenced by the increasing value of the Si/Al ratio.

### Effects on Wood-geopolymer Interface

The results of the SEM-EDS analysis of the interface between WF and the geopolymer matrix are shown in Fig. 4. In the FWGCs with low WF content (FWGC-1-

28), WF was bound by the geopolymer gel, and the wood vessels were filled with the geopolymer. The WF surface (Figs. 4a through 4b) and the pit border of WF (Fig. 5c) were attached by the geopolymer, indicating the existence of bonds between the WF and the geopolymer matrix. The occurrence of bonding was also implied by the wood pits being filled with geopolymer gels, as observed in Fig. 5b. To better understand the bonding mechanism between the WF and the geopolymer matrix, further research is needed to obtain more details. In the EDS results, the Si/Al ratio continuously increased from the WF surface (Spectrum 1) to the geopolymer matrix (Spectrum 3) with small differences, suggesting unnoticeable degradation of WF when the WF content was low in the FWGCs.



**Fig. 4.** SEM and EDS images of the interface of geopolymer composites: (a and b) FWGC-1-28 and (c and d) FWGC-15-28



Fig. 5. SEM of pits in the geopolymer composites: (a) FWGC-15-7 and (b-c) FWGC-5-7

The common figure of the FWGC-15-28 specimens (high WF content) is shown in Figs. 4c through 4d, where the differences from low-WF FWGCs can be seen. In the FWGC-15 specimens, the degradation of WF is obvious in Fig. 4c. Moreover, it is noteworthy that a large quantity of unreacted FA spheres can be observed in Figs. 3g

through 3e and Figs. 4c and 4d, as well as in the wood pits unfilled with geopolymer matrix (Fig. 5a), which were the evidence of the inadequate geopolymerization caused by the insufficient activation of the FA. Unreacted or partially reacted FA spheres were detected on the WF surface with a large space in the interface, indicating the weak bonding between WF and the geopolymer at a high WF content. In addition, a relatively high Si/Al ratio was detected in the WF (Spectrum 1), followed by the WF at the interface (Spectrum 2), and then the region of the geopolymer matrix (Spectrum 3), which can be explained mainly by the Si species absorption of sodium silicate from WF.

### **Effects on Mechanical Properties**

The WF contents exerted a negative effect on the density of FWGCs (Fig. 6). The relationships between the density and the mechanical strength (compressive strength and flexural strength) of the FWGCs are shown in Figs. 7a and 7b, respectively. The shapes of both obtained fit curves of the mechanical strength were similar to a parabola caused by the increasing density of the FWGCs. The augmented density of the obtained composites resulted in a greater mechanical strength.



Fig. 6. Relationships between the WF content and the density of the FWGCs



**Fig. 7.** Relationships between the density and the mechanical strength of the FWGCs with various WF contents: (a) compressive strength and (b) flexural strength

The compressive strength and flexural strength of the FWGCs with various WF contents at different curing times are shown in Figs. 8a and 8b, respectively. The FWGCs with low WF contents (1 wt% and 5 wt%) were almost unchanged or even improved the mechanical properties compared to those of the pure FA geopolymer. Similar results were reported as the enhanced compressive strength of FA based geopolymer composites with cotton fibers due to the absorption of stress transferred from the geopolymer matrix by fibers (Alomayri *et al.* 2014).

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**Fig. 8.** (a) Compressive strength and (b) flexural strength of the FWGCs with different WF contents and curing times

However, the addition of high WF contents (10 wt%, 15 wt%, and 20 wt%) started to adversely affect the strength of the geopolymer. The expected excellent flexural strength was not detected in the FWGCs with high WF contents. It was because that the increasing amount of WF, a kind of short fibers, caused a decrease in flexural strength. It was in agreement with previous reports of short natural fibre-reinforced biodegradable resin (Shibata *et al.* 2005). It was noticed that the compressive strength of the FWGCs was lower than that of the pure FA geopolymer, due to the low content of the geopolymer gel in the FWGCs (Rickard *et al.* 2011). Similar results were reported previously for a geopolymer reinforced by bamboo fiber bundles (Ribeiro *et al.* 2016). Moreover, the use of a high WF content resulted in relatively low compressive strength values due to the degradation of

WF and the insufficient activation of FA in the geopolymer illustrated by SEM and EDS (Figs. 3g through 3e and Figs. 4c and 4d, respectively).

The compressive strength of the samples rose with the prolongation of the curing time in the cases of FWGC-0 and low-WF FWGCs (1 wt% and 5 wt%), due to the dense geopolymer structure caused by improvement of the geopolymerization. The results agreed with the literature (Tho-In et al. 2018), who expressed their belief that the improvement of the geopolymerization process alongside curing time resulted in the increasing mechanical strength. The flexural strength of the specimens also increased in the cases of low-WF FWGCs as the extension of time in general. However, in the FWGC-5, flexural strength decreased slightly over time. This was because that the flexural strength of the FWGC was caused by the co-effect of geopolymer and WF. The geopolymer structure became denser; WF gradually degraded in the alkaline condition over time. This phenomenon was not obvious in the FWGC-1, because the strength of geopolymer played a main role in the flexural strength in the FWGC-1, as WF acted as a filling in the geopolymer. Compared to the low-WF FWGCs, a slightly different trend was established when the WF content was higher than 10 wt%. The reduction in the mechanical strength, especially in the compressive strength, was caused by the low level of geopolymerization and the degradation of WF with a longer curing time (Figs. 3g through 3i).

## CONCLUSIONS

- 1. The morphology, geopolymerization, interface structures, and mechanical properties of the fly-ash/wood-flour geopolymer composites (FWGCs) were greatly influenced by the wood-flour (WF) contents. In the cases of FWGCs with a low WF loading (1 wt% and 5 wt%), there was no change or even improved mechanical properties of the FWGCs detected due to less pores and better polymerization of the geopolymer, insignificant degradation of WF, and the existence of bonds between the WF and the geopolymer matrix.
- 2. In contrast, noticeable differences were found in the FWGCs with high WF contents. The addition of high WF content (10 wt%, 15 wt%, and 20 wt%) adversely affected the mechanical strength of the FWGCs. For the high-WF FWGCs, insufficient geopolymerization, degradation of WF, and the weak bonding between WF and the geopolymer were detected. A relatively high Si/Al ratio was also established in the WF, which was explained by the absorption of the activator by WF.
- 3. In general, the curing time had a positive effect on the mechanical properties of the FWGCs, which was consistent with denser microstructures and the growing degree of geopolymerization evidenced by SEM-EDS. Nevertheless, the crystallization of the FWGCs was seldom influenced by the longer curing times.

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