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Original Article

Effects of different wood aggregates on the compressive strength of fly ash and metakaolin-based geopolymer lightweight composites

Siti Noorbaini Sarmin[1*], Nurjannah Salim[2], and Noorshashillawati Azura Mohammad[1]

[1] *Centre of Wood Industry, Faculty of Applied Science, Universiti Teknologi MARA Cawangan Pahang, Kampus Jengka, Jengka, Pahang, 26400 Malaysia*

[2] *Material Technology, Faculty of Industrial Sciences and Technology, Universiti Malaysia Pahang, Gambang, Pahang, 26300 Malaysia*

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Abstract

Class F fly ash with 30% substitution of metakaolin was used as an alumino-silicate source to produce lightweight geopolymer composites. The investigations focused on different types of wood aggregates that influence the compressive strength of lightweight geopolymer composites. Wood particles (WP), wood flour (C100), and wood fiber (WF) were added to fly ash and metakaolin-based geopolymers at 10% solid content as reinforcement materials. NaOH in combination with Na_2SiO_3 was used as the alkaline activator with a liquid-to-solid ratio of 1.33:2.00. The samples were cured at 20 °C for 7, 14, and 28 days, and at 80 °C for 6 and 24 h (two different curing temperatures and five different curing times). It was observed that the types of wood aggregates affected the properties and compressive strength of lightweight geopolymer composites. The highest compressive strength of about 38.4 MPa was achieved for the sample containing wood flour that was cured for 28 days.

Keywords: geopolymer, lightweight composite, fly ash, metakaolin, wood aggregate

1. Introduction

Lightweight concrete (LWC) is an ideal material construction in order to reduce building costs, ease construction, and it has the advantage of being a relatively 'green' building material. LWC is a concrete with unit weight less than 2000 kg/m^3 (Dulsang, Kasemsiri, Posi, Hiziroglu, & Chindraparasit, 2016). LWC is classified into three types depending on the method of production: LWC with lightweight aggregate; LWC with voids formed by aeration (i.e. cellular concrete, foamed or gas concrete); and LWC with coarse aggregate (Ahmaruzzaman, 2010; Dulsang, Kasemsiri, Posi, Hiziroglu, & Chindraparasit, 2016). In recent years, more attention has been paid to the use of plant-based natural aggregates which are believed to exhibit characteristics similar to those of lightweight aggregates used in the production of LWC.

The utilization of natural aggregates in cement matrix has many advantages, categorically in amending flexural/tensile strength (Simatupang & Geimer, 1990), enhancing fracture resistance properties (Alomayri, Shaikh, & Low, 2013; Hakami, Shaikh, & Low, 2014), and increasing impact toughness (Asasutjarit *et al.*, 2005). According to Onuaguluchi and Banthia (2016), natural aggregates that have been used successfully for concrete reinforcement can be categorized into three different categories: animal-based; mineral-derived; and plant-based. Of late, there has been a spurt in the studies and knowledge on plant-based, natural aggregate-reinforced cement composites. Compared to synthetic aggregates, natural aggregates are believed to be more environmentally friendly, cheap, and of low density. Besides, they are both renewable and recyclable (Alomayri, Shaikh, & Low, 2013; Pehanich, Blankenhorn, & Silsbee, 2004). Consequently, they are currently drawing a lot of attention for their use in replacing synthetic fibers.

*Corresponding author
Email address: baini@uitm.edu.my

Despite the advantages of plant-based natural aggregates, there are still obstacles that limit their application in the cement matrix. The main disadvantage is decomposition of certain chemical constituents of natural aggregates in the alkaline cement environment that leads to the degradation of natural aggregates, thereby weakening the interfacial bond between the natural aggregates and the cement matrix (Pehanich, Blankenhorn, & Silsbee, 2004; Hakamy, Shaikh, & Low, 2014; Simatupang & Geimer 1990;). According to Alomayri, Shaikh and Low (2013), the type of natural aggregates, its form, surface properties, and matrix properties need to be considered to overcome this disadvantage.

Fly ash has been widely studied as the main source of geopolymers because it is available in abundance throughout the world (Alomayri, Shaikh, & Low, 2013; Hardjito, Wallah, Sumajouw, & Rangan, 2005). It is a waste material generated from burning coal. The main constituents of fly ash are silicon (Si) and aluminum (Al) (Hardjito, Wallah, Sumajouw, & Rangan, 2005), which is considered a cement replacement material in concrete, widely known as geopolymer concrete. Geopolymer synthesis requires a highly alkaline solution to dissolve Si and Al ions and to release SiO_4 tetrahedral and AlO_4 tetrahedral units. These units are linked to the polymeric precursors by sharing oxygen atoms to form a rigid polymer. Geopolymers possess excellent properties including high compressive and flexural strength, low shrinkage, freeze-thaw resistance, sulphate, corrosion resistance, as well as heat and electrical resistance. All these make them ideal for various applications in geopolymer technology (Sarmin, Welling, Krause, & Shalbafan, 2014; Wu & Sun 2007,).

There has been a rise in research on the development of sustainable geopolymer-reinforced plant-based natural aggregates such as cotton fiber, oil palm shell, hemp fabric, wood fiber, and basalt fiber. However, the literature on the compressive strength of lightweight geopolymer composites using different types of wood aggregates has not been systematically studied. In the present study, different types of wood aggregates were introduced into the fly ash/metakaolin-based geopolymer lightweight composites. Wood aggregates were varied in form and size, and their effects on the compressive strength of fly ash/metakaolin-based geopolymer composites were studied. Field emission scanning electron microscopy (FESEM) was used to characterize the microstructure and wood aggregate dispersion of geopolymer composites. Also studied were the effects of varying the temperature and time for the curing of fly ash/metakaolin-based geopolymer composites.

2. Materials and Methods

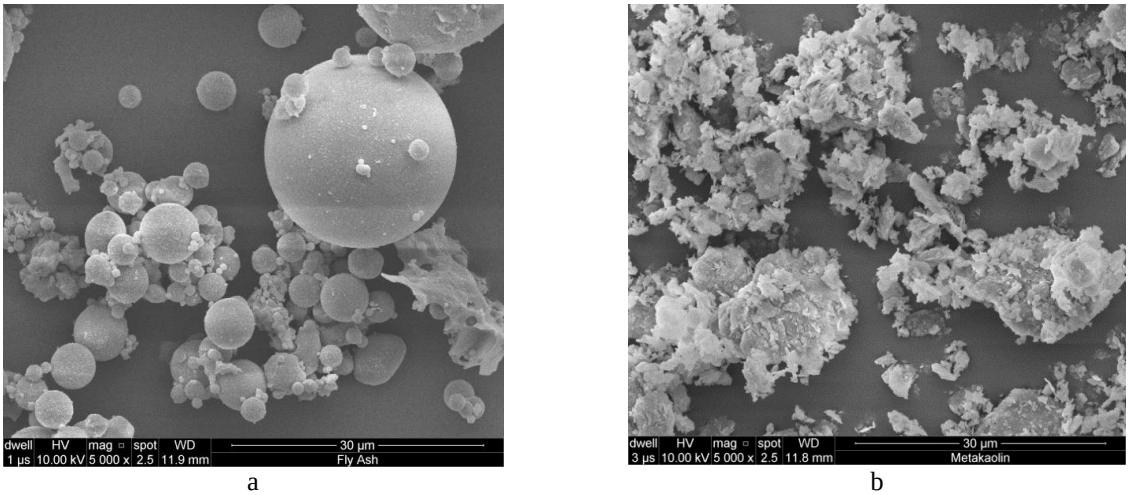
2.1 Materials

Low-calcium Class F fly ash and metakaolin were used as the basic materials for the preparation of the geopolymers. The fly ash was supplied by a power plant (GK Kiel GmbH, Kiel, Germany) while Metakaolin-Argical M1000 was obtained from IMERYS Refractory Minerals, Clérac, France. The chemical compositions of fly ash and metakaolin are shown in Table 1.

The microstructures of as-received fly ash and metakaolin were studied by FESEM (Figure 1). The fly ash particles were spherical in shape with a relatively smooth outer surface and the size distribution was 930-25000 nm. FESEM micrographs of metakaolin revealed vitreous unshaped fragments (Figure 1b). It was clearly identified as non-crystallized, lamellar particles. The particle size distribution of the metakaolin was 63-200 µm.

Table 1. Chemical composition (% Mass) of fly ash and metakaolin.

Oxide Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	L.O.I.	Total
Class F Fly ash	56.8	23.8	6.79	2.9	1.28	1.22	1.99	1.72	3.5	100.00
Metakaolin	55.0	40.0	1.4	0.15	0.15	0.4	0.4	1.5	1.0	100.0



a b

Figure 1. FESEM images of (a) fly ash and (b) metakaolin with 5000x magnification.

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions were used as alkali activators in the preparation of the geopolymer paste. Analytical-grade NaOH in pellet form with 98% purity and 2.13 g/cm^3 specific gravity was obtained from Fisher Scientific UK Ltd, Bishop Meadow Road, Loughborough. The Na_2SiO_3 solution (SiO_2 = 30.2%, Na_2O = 14.7%, water = 55.1%, SiO_2/Na_2O molar weight ratio = 2.0, with a density of 1.54 g/cm^3 at 20 °C) from Woellner GmbH & Co. KG, Ludwigshafen am Rhein, Germany, was used along with NaOH as the alkali activator.

Wood aggregates of different shapes and sizes were used to reinforce the geopolymer matrix. Three types of wood aggregates—wood particle (WP), wood flour (C100), and wood fiber (WF)—were obtained from local mills in Germany. The characteristics of each aggregate are shown in Table 2.

2.2 Preparation of geopolymer composites

In total, eight mixtures were prepared by varying the types of wood aggregates, curing time, and curing temperature. The fly ash, metakaolin, and activator contents were kept constant. The ratio of alkaline solutions to solid was 1.33:2.00. The mixture contents for specimens are provided in Table 3.

The alkali activator solution was prepared by dissolving NaOH pellets in water and then adding the $NaSiO_3$ solution in a weight ratio of 2.5:1. The activator solution was prepared at least one day prior to use. Fly ash (70%) and metakaolin (30%) were first dry-mixed with 10% wood aggregate (i.e. WP, C100 or WF) to ensure a uniform solid supply. Specimens without wood aggregates were also prepared as a reference for comparison with specimens containing wood aggregates. The alkaline liquid was then added to the dry materials and mixed for 5 min to ensure homogeneity of the mixture. The fresh pastes were cast into 5 cm cubic molds. Immediately after casting, the test specimens were wrapped with plastic film to minimize water evaporation during curing. The samples were cured at 20 °C for 7, 14, and 28 days and at 80 °C for 6 and 24 h. After 24 h after each time period, they were removed from the molds. The specimens were left to air-dry in the climate chamber at 20 °C and humidity of 60% until the day of the test.

2.3 Material characterizations

The cubes were tested in compression in conformity with the test procedures given in ASTM C109, using a Zwick universal testing machine. The compressive strength values reported were averaged over the measurement of nine samples.

The physical properties of the samples such as density, water absorption, and porosity were determined to ascertain the quality of the geopolymer composite specimens (Alomayri, Shaikh, & Low, 2013). The values reported were averaged over the measurement of nine samples.

The values of water absorption (W_a) and porosity (P_a) were determined based on the ASTM C642 standard. The values were calculated using the following equations:

$$W_a = \left(\frac{W_s - W_d}{W_d}\right) \times 100 \quad (1)$$

$$P_a = \left(\frac{W_w - W_d}{W_w - W_s}\right) \times 100 \quad (2)$$

where W_s, W_d, and W_w are the saturated weight of the sample, oven-dried weight of the sample, and the weight of the sample immersed in water respectively.

The microstructures and fracture surfaces of the geopolymer composites were examined using FESEM (Quanta FEG Type 250, FEI Electron Optics [SN: D9122], Netherlands). The specimens were crushed into small pieces before being coated with a thin layer of gold to avoid charging effects.

3. Results and Discussion

3.1 Density, porosity, and water absorption of lightweight geopolymer composites

The density and porosity values of the lightweight geopolymer composites are presented in Table 4, Figure 2, and Figure 3, respectively. Generally, the composites containing wood aggregates exhibited lower density compared to specimens without wood aggregates. The average (SD) densities of specimens without wood aggregate for curing times of 6 h and 24 h were 1540 (0.66) kg/m^3 and 1563 (0.25) kg/m^3, respectively (Table 4). For specimens that contained C100, the densities were 1417 (1.03) kg/m^3 and 1417 (0.96) kg/m^3 at the same curing conditions. For specimens that contained WF, the average densities were 1329 (1.07) kg/m^3 and 1329 (0.63) kg/m^3 and the average densities of specimens that contained WP were 1215 (1.06) kg/m^3 and 1262 (1.23) kg/m^3, respectively. The same trend was observed in all curing conditions. The specimens that contained wood aggregates had lower densities compared to specimens without wood aggregates. These results agreed with those obtained by other

Table 2. Properties and structure of wood aggregates.

Properties	Wood Flour (C100)	Wood Fiber (WF)	Wood Particle (WP)
Color	Beige	Brown	Light brown
Structure	Cubic	Longish fiber	Particle
Size	70 µm–150 µm	-	-
Length	-	3–7 mm	3–5 mm
Width	-	43.6–44 µm	0.2–0.5 mm
Bulk density	0.14–0.2 g/cm^3	0.1–0.23 g/cm^3	0.15–19 g/cm^3
Moisture content	8.6%	7.5%	6.8%
Species	Mix softwood	Mix softwood	Mix softwood

Table 3. Experimental mix design.

Solid:Alkaline	Fly ash (70%)	Metakaolin (30%)	Wood Aggregates (10%)	Alkaline Solution (1.0:2.5)		Water
				NaOH (14M)	Na_2SiO_3	
2.0:1.33	378 g	162 g	60 g	63 g	285 g	50 g

Table 4. Properties of specimens for different types of wood aggregates (standard deviation in grayish subscript).

Wood Aggregates	Curing Conditions		Density (kg/m³)	Compression Strength (MPa)	Porosity (%)
	Time	Temperature (°C)			
Without aggregates	6 h	80	1540 $_{0.66}$	35.73 $_{0.26}$	16.68 $_{0.66}$
	24 h	80	1563 $_{0.25}$	43.28 $_{0.33}$	15.42 $_{0.98}$
	7 days	20	1540 $_{0.69}$	69.43 $_{0.86}$	14.31 $_{0.36}$
	14 days	20	1568 $_{0.45}$	70.89 $_{0.69}$	14.14 $_{1.03}$
	28 days	20	1571 $_{0.57}$	76.49 $_{0.62}$	13.90 $_{1.45}$
Wood Flour (C100)	6 h	80	1417 $_{1.03}$	32.76 $_{0.96}$	16.46 $_{0.69}$
	24 h	80	1417 $_{0.96}$	36.25 $_{0.36}$	15.79 $_{0.77}$
	7 days	20	1435 $_{0.23}$	33.70 $_{0.66}$	16.10 $_{0.85}$
	14 days	20	1478 $_{1.05}$	34.24 $_{0.68}$	17.29 $_{0.68}$
	28 days	20	1471 $_{0.99}$	38.40 $_{0.78}$	18.39 $_{0.96}$
Wood Fiber (WF)	6 h	80	1329 $_{1.07}$	27.19 $_{0.45}$	22.90 $_{1.12}$
	24 h	80	1329 $_{0.63}$	29.09 $_{0.95}$	22.00 $_{0.69}$
	7 days	20	1401 $_{0.79}$	21.69 $_{0.22}$	19.89 $_{0.56}$
	14 days	20	1339 $_{0.62}$	25.47 $_{0.62}$	20.62 $_{0.89}$
	28 days	20	1426 $_{0.92}$	29.96 $_{0.47}$	20.38 $_{0.36}$
Wood Particle (WP)	6 h	80	1215 $_{1.06}$	17.15 $_{0.58}$	19.53 $_{0.25}$
	24 h	80	1262 $_{1.23}$	20.85 $_{0.41}$	19.94 $_{1.05}$
	7 days	20	1329 $_{1.52}$	18.67 $_{0.32}$	19.98 $_{0.66}$
	14 days	20	1322 $_{0.48}$	19.91 $_{0.29}$	18.68 $_{0.89}$
	28 days	20	1276 $_{0.93}$	20.24 $_{0.15}$	19.69 $_{0.75}$

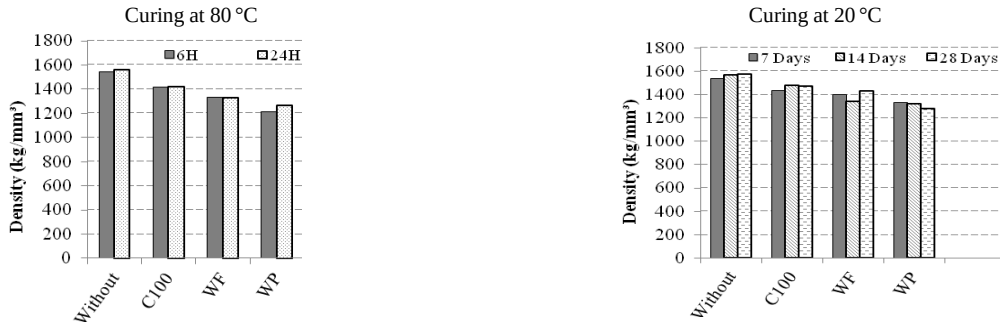


Figure 2. Density values of the geopolymer composites as a function of different wood aggregates.

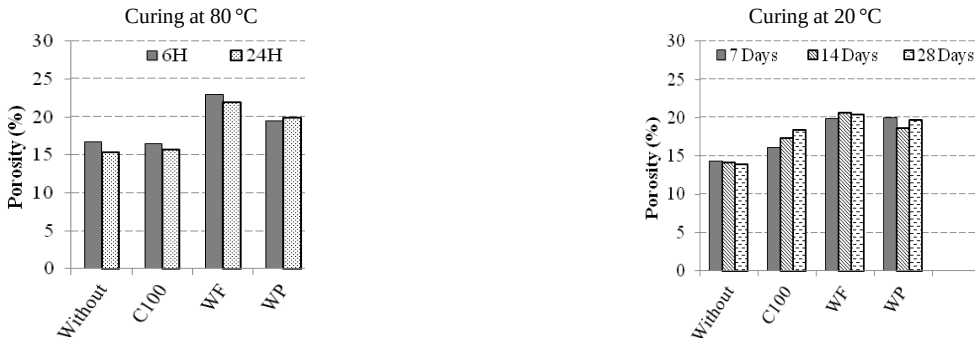
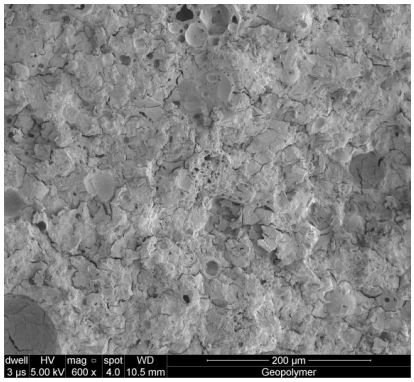


Figure 3. Porosity of the geopolymer composites.

researchers (Alomayri, Shaikh, & Low, 2013; Chen, Ahmari, & Zhang, 2014; Hakamy, Shaikh, & Low, 2014). For instance, the study on sweet sorghum fiber-reinforced fly ash-based geopolymer reported that the density values decreased as the fiber content increased (Chen, Ahmari, & Zhang, 2014). Similarly, Hakamy, Shaikh, and Low (2014) and Alomayri, Shaikh, and Low (2013) found in their studies that the density values of cement composites decreased as the fiber content increased. The addition of wood aggregates increased the porosity value of the lightweight geopolymer composites (Figure 3). Within the wood aggregates, the highest value of porosity was found in the specimen that contained WF, whereas the composites containing C100 showed the lowest porosity. The control sample that contained no wood aggregates exhibited the lowest porosity value.

The use of wood aggregates for making lightweight geopolymer composite produced lower density concrete. Two factors were considered regarding this phenomenon. The first assumption was the formation of voids at the interfacial areas between the wood aggregates and the geopolymer matrix. Poor dispersion and agglomerations of the wood aggregates may influence density and porosity in this study. The agglomerations create more voids or large pores after geopolymerization and leave many inter-granular pores in the microstructure after curing. In addition, wood aggregates are likely to tend to clump together during mixing, resulting in more voids and an irregular microstructure of the composites. These results were also supported by the FESEM analysis of the microstructure of the control specimens and specimens containing wood aggregates. Figure 4 shows the FESEM images of lightweight geopolymer composites with and without wood aggregates. Figure 4a shows that the structure of the pure geopolymer paste is denser and more compact with few pores. Besides, in Figures 4b and 4c, the geopolymer composites containing wood aggregates showed more pores and microcracks that weakened the structure. The second assumption was the density and specific gravity of wood aggregates. Since the specific gravity of the wood aggregates was recorded to be within 0.15 to 0.20, the replenishment of the wood aggregate to the geopolymer mixture was supposed to lower the density value of the hardened specimens. From this result, it could be concluded that the specific gravity and the density of wood aggregates contributed to the reduction of the density of the lightweight geopolymer concrete.

Figure 5 shows the percentage of the water uptake of lightweight geopolymer composite samples with and without wood aggregates after immersion in tap water for seven days at room temperature. Water absorption increased for the specimens with the addition of wood aggregates. Water absorption of all specimens was high in the early stages of exposure. After a long time, it slowed down and reached the saturation level. The hydrophilic nature of wood aggregates enhances the increase of water uptake in the lightweight geopolymer composite with wood aggregates (Alomayri, Assaedi, Shaikh, & Low, 2014; Dhakal, Chan, & Richardson, 2007). Additionally, Alomayri, Assaedi, Shaikh and Low (2014) reported in their study on cotton fiber-reinforced geopolymer composites that the increase in water absorption was due to the greater interfacial area between the fiber and the matrix. This resulted in microcracking of the geopolymer composite which created swelling stresses that led to composite failure.



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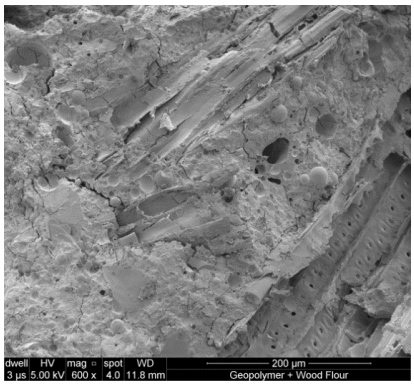

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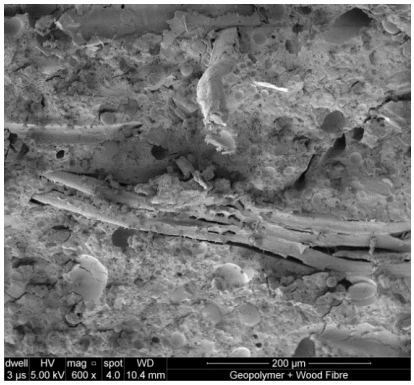

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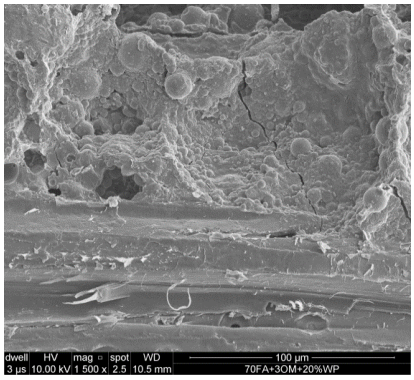

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Figure 4. FESEM images showing (a) geopolymer paste, (b) geopolymer with C100, (c) geopolymer with WF, and (d) geopolymer with WP at 600x magnification.

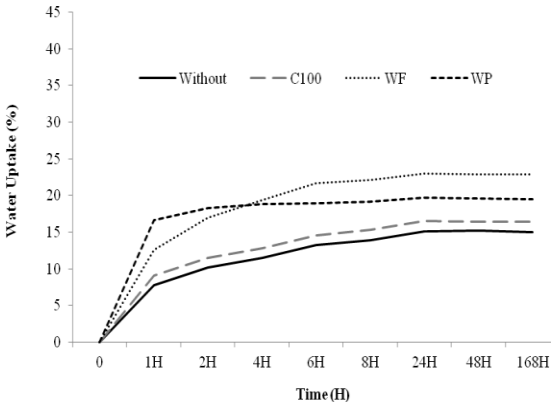

Figure 5. Water absorption behavior of geopolymer composite.

3.2 Effect of wood aggregates on compressive strength

The effects of wood aggregate content on the compressive strength of lightweight geopolymer composites are presented in Figure 6. It can be reported that the compressive strength decreased with the addition of wood aggregates. Specimens that contained wood aggregates had compression strengths between 17.15 (0.58) MPa to 38.40 (0.78) MPa at different curing conditions, while specimens without wood aggregates experienced higher compression strengths which were between 35.73 (0.26) MPa to 76.49 (0.62) MPa. This was consistent with the findings of other researchers. For example, a study by Chen, Ahmari, and Zhang (2014) showed a decrease in the compression strength value for fly ash-based geopolymer-reinforced sweet sorghum. Awang, Ahmad and Al-Mulali (2015) reported that the inclusion of fiber decreased the lightweight foamed concrete. Timakul, Rattanaprasit and Aungkavattana (2016) mentioned that compressive strength of fly ash-based geopolymer composites decreased with the addition of basalt fibers. Kriker, Debicki, Bali, Khenfer and Chabannet (2005) and Li, Wang and Wang (2006) also reported that the incorporation of natural fiber in cement caused a decrease in the compressive strength. This agreed with the general conception that the inclusion of fiber decreases the average compressive strength of concrete (Chen, Ahmari, & Zhang, 2014; Awang, Ahmad, & Mulali, 2015). The main function of aggregate inclusion is to control the cracking of the reinforced composite by bridging across the cracks and providing post-cracking ductility (Timakul, Rattanaprasit, & Aungkavattana, 2016). This was seen in the failure mode of the specimen without wood aggregates where multiple cracking was obviously detected. The specimens that contained C100, WF, and WP showed less cracking due to fracture resistance by the aggregates which resulted in increased energy dissipation from crack-deflection at the aggregate-matrix interface, aggregate bridging, and aggregate pullout (Banthia & Sheng, 1996; Rovnaník, 2010).

The compressive strength of the specimens made with C100 aggregate was higher than the compressive strength of those made with WF and WP (Figure 6). The difference in strength seems to arise from the different shape and size of the wood aggregates. C100 is around 70-150 μm in size with a

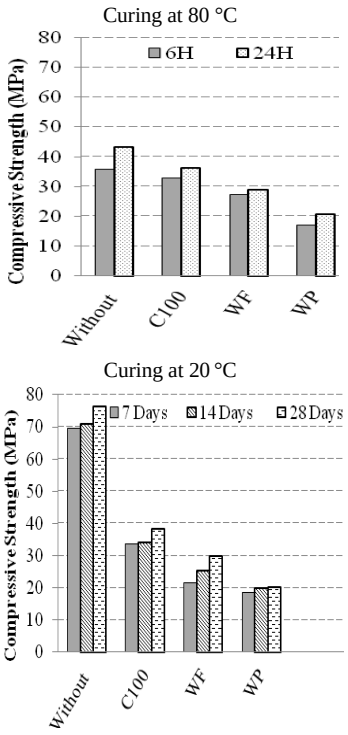

Figure 6. Compressive strength of the geopolymer composites at different curing conditions with respect to the addition of wood aggregates.

cubic shape. Compared to WF and WP, C100 has a filling effect whereby the small size of C100 fills the voids or pores in the geopolymer paste in which the C100 particles are uniformly dispersed in the matrix, thus making the microstructure of the lightweight geopolymer composites denser. Therefore, good interfacial bonding between the matrix and C100 was achieved. The specimens that contained C100 exhibited higher density and lower porosity compared to specimens with WF and WP.

3.3 Curing time and temperature effect

The investigation also focused on the influence of curing temperature and curing time. The influence of curing temperature and time on the compressive strength of lightweight geopolymer composites is tabulated in Figure 6. The specimens with C100 cured at 20 °C reached compressive strengths of 38.4 (0.78) MPa, 34.24 (0.68) MPa, and 33.70 (0.66) MPa at the ages of 28, 14, and 7 days respectively. As expected, the elevated temperature accelerated the early stage of geopolymerization reaction which resulted in better performance properties of the composites. For all three compositions—C100, WF, and WP—the compressive strength of the specimens cured at 20 °C for 7 days could be reached by accelerated curing at 80 °C after only 6 h, whereas the specimens cured at 80 °C for 24 h showed the same strength if cured at 20 °C for 14-28 days. Therefore, it was concluded that, when cost is taken into account, the optimum curing condition at 80 °C is 6 h or even less (Hardjito, Wallah, Sumajouw, & Rangan, 2005; Rovnaník, 2010; Wu & Sun, 2007). This work was also in agreement with the research

done by Hardjito, Wallah, Sumajouw, and Rangan, (2005). They found that fly ash-based geopolymer concrete cured at 80 °C up to 48 h showed an increase in its compressive strength. Al Bakria, Kamarudin, BinHussain, Nizar, Zarina and Rafiza (2011) reported that the maximum compressive strength of fly ash-based geopolymers was obtained at 80 °C. Rovanik (2010) and Görhan, Aslaner and Sinik (2016) also reported the significant roles of temperature and the curing time based on their findings.

4. Conclusions

In this study, the effects of different wood aggregates on the properties of fly ash and metakaolin-based geopolymer lightweight composites were investigated by measuring density, porosity, water uptake, and compressive strength. From the experimental and analytical observations, it can be concluded that wood aggregates can be used to produce lightweight geopolymer composites. The shape and size of the wood aggregates affect the properties of the lightweight geopolymer composites. The optimum wood aggregate type is C100, followed by WF and WP. The densities of the lightweight geopolymer composites decreased while the porosity increased with the addition of wood aggregates. According to the FESEM results, lightweight geopolymer composites that contained C100 showed better aggregate matrix interfacial bonding than those with WF or WP. The curing temperature and curing time play significant roles in lightweight geopolymer composites. The strength of the geopolymer composites developed rapidly when cured at a higher temperature than at room temperature. Curing at 80 °C for 24 h had almost the same compressive strength as curing at 20 °C for 28 days. Wood aggregate-reinforced geopolymer lightweight composites could be an interesting alternative to synthetic aggregate-reinforced lightweight geopolymer composites. However, extensive research is needed to overcome problems arising from agglomeration and poor dispersion during the preparation of the geopolymer paste. In addition, the interfacial bonding between geopolymers and wood needs to be investigated further.

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