Chapter 70
The Influence of Curing Conditions on the Compressive Strength of Lightweight Geopolymer Composite Containing Wood Aggregates

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Abstract In this study, lightweight geopolymer composite was produced using fly ash, metakaolin, and wood aggregate. The changes caused by the geopolymerization on the properties of the final product were investigated by applying curing on geopolymer composites using different types of wood aggregates at different curing temperatures and curing times. The purposes of this process were to determine the relationship between types of wood aggregates, curing temperature, and curing times toward the compressive strength of the 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 reinforced materials. 14 M 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 60 °C for 6 and 24 h (two different curing temperatures and

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five different curing times) and thereafter kept at room temperature (26–29 °C) until the day of the physical and mechanical test. As a result, this study determined that curing temperature and curing time had an effect on the compressive strength of the composite. It was observed that compressive strength values of the lightweight geopolymer composite cured at 20 and 60 °C increased depending on the curing time. Highest compressive strength values of 38.4 and 36.25 MPa were obtained from the mortar with C100 addition cured at 20 °C for 28 days and 60 °C for 24 h, respectively.

Keywords Curing periods · Compressive strength · Geopolymer Lightweight composite · Wood aggregates

1 Introduction

Cement is indispensable material in the development of concrete. The used of ordinary Portland cement (OPC) was not environmentally friendly and caused the adverse effect, resulting from the calcination of limestone and high energy consumption from OPC manufacture (Turner and Collins, 2013). In 1978, Davidovits proposed that binders could be produced by the reaction between the alkaline solution and source materials that are rich in silica (SiO_2) and alumina (Al_2O_3), commonly known as geopolymer. Geopolymers are amorphous three-dimensional aluminosilicate binder materials (Shaikh 2013) which have been proposed as an ecologically friendly alternative to OPC. Geopolymer materials contain aluminum (Al) and silicon (Si) species that are soluble in highly alkaline solutions (Davidovits 2008). Any material that is rich in Si and Al in amorphous form can be a possible source material for geopolymer binder (Shaikh 2013).

Lightweight concrete (LWC) is an ideal material construction in order to reduce building costs, eases construction, and has the advantage of being relatively "green" building material. LWC is a concrete with unit weight less than 2000 kg/m^3 (Dulsang et al. 2016) and is classified into three types depending on the method of production: LWC with lightweight aggregate; LWC with incorporation of voids by aeration, cellular, foamed, or gas concrete; and LWC with no-fine aggregate (Dulsang et al. 2016; Ahmaruzzaman 2010). LWC has several valuable characteristics such as good thermal and acoustic insulations, easy to fabricate, excellent freezing and thawing durability, internal curing, and reduced dead load.

Currently, plant-based aggregates are the most widely used in LWC production as a result of the increasing demand for environmentally friendly materials and the high cost of synthetic lightweight aggregates. These plant-based aggregates exhibit a grading comparable to that of conventional aggregates and in fact, an entirely renewable resource. They are low-density materials yielding relatively lightweight

composite. Utilization of plant-based aggregates is the subject of the several studies, for example, Torkaman et al. (2014) used wood fiber waste as a replacement material for lightweight concrete blocks; Faustino et al. (2015) found that corn cob is relevant to produce lightweight concrete masonry; Bouguerra et al. (1998) reported the microstructure properties of lightweight concrete that were prepared using wood aggregates; the study by Sales et al. (2010) concluded that the lightweight composite concrete produced using sawdust presented a 23% lower thermal conductivity than the conventional concrete and Kidalova et al. (2012) describes the effect of different binding agents in combinations with hemp fibers and wood cellulose in the preparation of lightweight composites.

There has been a rise in the research on the development of sustainable LWC-reinforced plant-based aggregates such as cotton fiber, oil palm shell, hemp fabric, wood fiber, and basalt fiber-based geopolymer concrete. However, the curing conditions varied from one case to another but the evaluation of temperature effects showed general trends. Thus, since there is no clear statement as to which curing conditions is the best, the main objective of the current research was to investigate the effect of different curing conditions of 20 °C for 7, 14, and 28 days, and at 60 °C for 6 and 24 h (two different curing temperatures and five different curing times) on the compressive strength of lightweight geopolymer composites synthesized from alkali activated fly ash and metakaolin. Different types of wood aggregates, varied in form and size, were introduced into the lightweight geopolymer concrete.

2 Materials and Method

2.1 Materials

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

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
Fly ash	56.8	23.8	6.79	2.9	1.28	0.43	1.99	0.94	3.5	98.43
Metakaolin	55.0	40.0	1.4	0.15	0.15	0.4	0.4	1.5	1.0	100.0

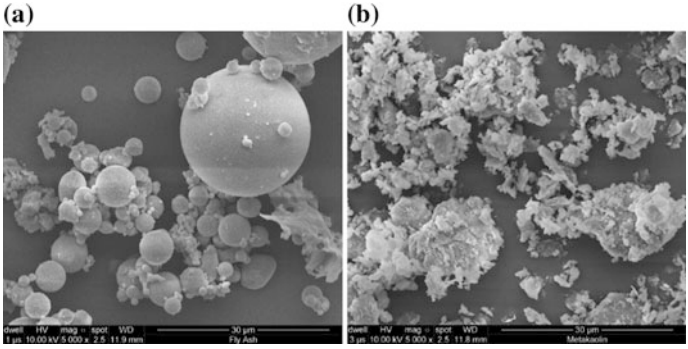

Fig. 1 FESEM image of **a** fly ash and **b** metakaolin with 5000× magnification

The microstructure of as-received fly ash and metakaolin was studied by the FESEM (Fig. 1). The fly ash particles were spherical in shape, having relatively smooth outer surfaces, with a size distribution between 930 and 25,000 nm. FESEM micrographs of metakaolin shown in Fig. 1b revealed vitreous unshaped fragments. It was clearly identified as non-crystallized, lamellar particles. The particle size distribution of the metakaolin ranged from 63 to 200 µm.

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions were used as alkali activators in the preparation of 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.

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	70–150 µm	–	–
Length	–	3–7 mm	3–5 mm
Width	–	43.6–44 µm	0.2–0.5 mm
Bulk density (g/cm^3)	0.20	0.17	0.15
Moisture content (%)	8.6	7.5	6.8
Species	Mix softwood	Mix softwood	Mix softwood

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 solid to alkaline solutions was 2.0:1.33. Figure 2 summarizes the synthesis protocol for the geopolymer composites. 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 its use. 70% fly ash and 30% metakaolin were first dry-mixed with 10% wood aggregate, WP, C100, and WF in order to ensure a uniform solid supply. 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 60 °C for 6 and 24 h, and during that period, after 24 h they were removed from the molds. The specimens were left to air-dried 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/C109M-12 (2012), using a Zwick universal testing machine. The

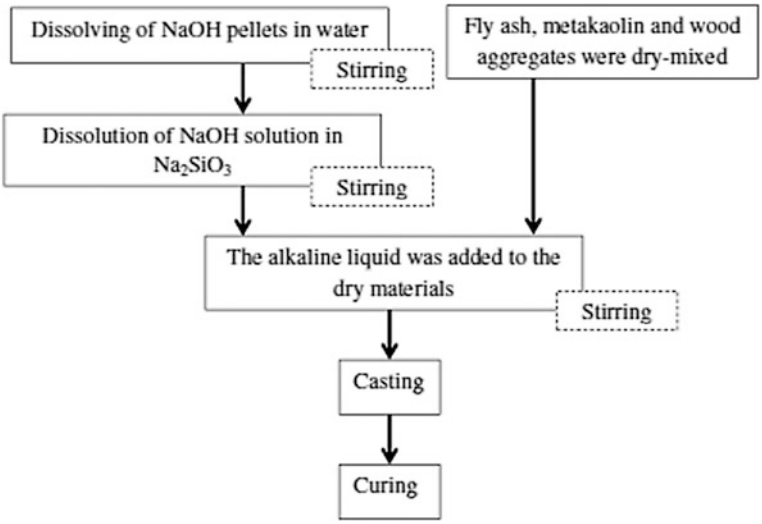

Fig. 2 Synthesis protocol of geopolymer composites

compressive strength values reported were averaged over the measurement of nine samples. The oven-dried density and water absorption were determined to ascertain the quality of the geopolymer composite specimens according to ASTM C140-01 (2012). The specimens were immersed in water at room temperature (22 °C) for 24 h. Weigh the specimens while suspended by a thin wire and completely submerged in water and record W_i (immersed weight). Remove the specimens from water and allow water to drain for 1 min by placing them on a wire mesh, removing visible surface water with a damp cloth, weigh and record as W_s (saturated weight). Then, dry all specimens in a ventilated oven at 105 °C for not less than 24 h, and two successive weightings at intervals of 2 h show an increment of loss not greater than 0.2% of the last previously determined weight of the specimen. Record the weight of the specimens as W_d (oven-dried weight). The calculation of oven-dried density (D_b) and water absorption were carried out using the following equation:

$$D_b = \frac{W_d}{W_s - W_i} \tag{1}$$

$$W_a = \left(\frac{W_s - W_d}{W_d}\right) \times 100 \tag{2}$$

The microstructures and fracture surfaces of the geopolymer composites were examined using a 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).

2.4 Results and Discussion

In regards to the lightweight requirement, density indicates the important parameter in this study. The result of density is shown in Fig. 3. Generally, the density values ranged from 1215 to 1478 kg/m^3 for all specimens. The highest density of the lightweight concrete composite in this study (1478 kg/m^3) was in the range of 1440–1850 kg/m^3 for structural lightweight concrete in accordance with ACI 213 (2003). The use of wood aggregates for making lightweight concrete composite produced lower density concrete. Two factors were considered regarding this phenomenon. The first assumption was the formation of voids in the interface areas between the wood aggregates and the geopolymer matrix. Poor dispersion and agglomerations of the wood aggregates may have an effect on the density value of specimens in this study. The agglomerations create more voids or large pores after geopolymerization and leave a large number of 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 irregular microstructure of the composites. The second assumption was the density and specific gravity of wood aggregates. As the specific gravity of wood aggregates used is recorded to be within

0.15 (WP) to 0.20 (C100), the replenishment of the wood aggregate to the geopolymer mixture supposed to lower the density value of hardened specimens. It was clearly seen from Fig. 3 that the lowest density value experienced by specimens with WP followed by WF and C100. From this result, it can be concluded that the specific gravity and the density of wood aggregates contributed to the reduction of the density of the geopolymer concrete.

Figure 4 shows the percentage of the water uptake of geopolymer composite specimens with wood aggregates after immersion in tap water for 7 days at room temperature. The 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 geopolymer composite with wood aggregates (Dhakal et al. 2007). Additionally, Alomayri et al. (2014) reported in their study on cotton fiber-reinforced geopolymer composites that the increase in water absorption is due to the greater interfacial area

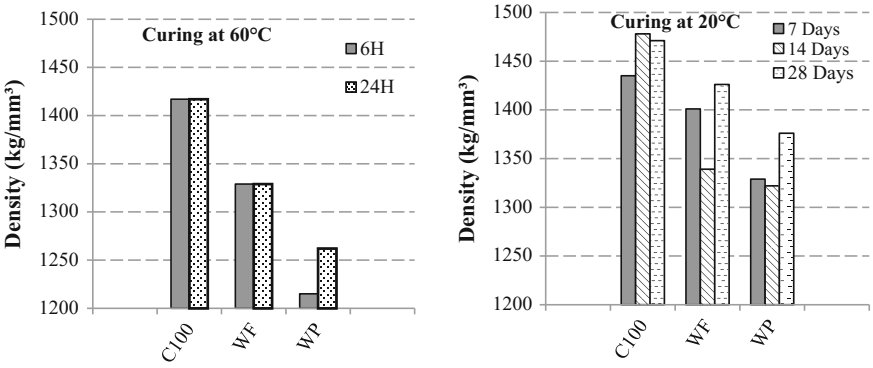

Fig. 3 Density values of the lightweight geopolymer composite as a function of different aggregates

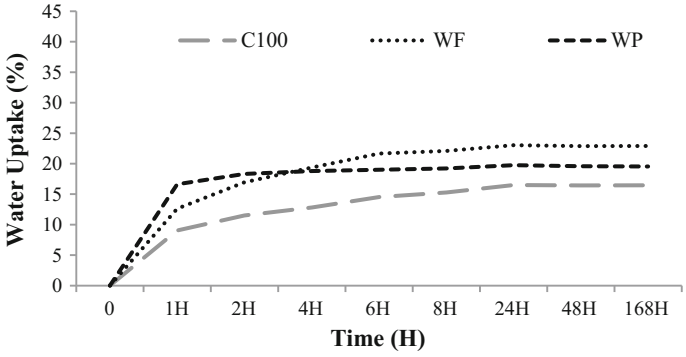

Fig. 4 Water absorption behavior of lightweight geopolymer composite

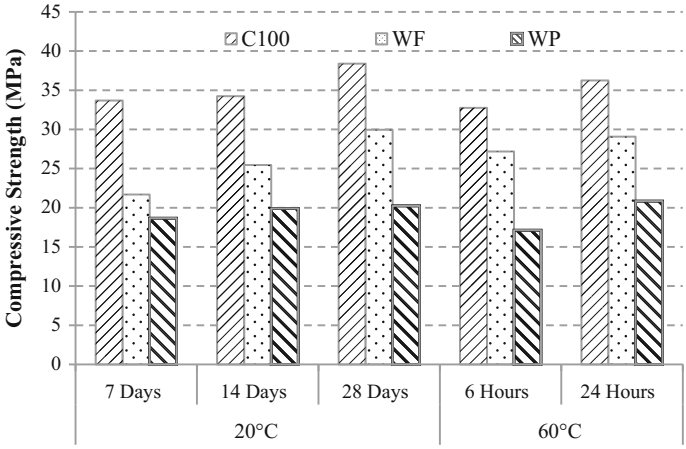

Fig. 5 Compressive strength of the lightweight geopolymer composites at different curing conditions with respect to the addition of wood aggregates

between the fiber and the matrix. In this study, the high cellulose content in wood aggregates absorbs water that penetrates the interface of the specimens. This phenomenon can be explained by considering the water uptake characteristics of WF compared to C100 and WP where WF experienced the highest percentage of water uptake.

Figure 5 shows the effect of curing temperature and curing time on the compressive strength of lightweight geopolymer composites after curing the test cubes for (i) 6 and 24 h at 60 °C and (ii) 7, 14, and 28 days at 20 °C. All other test variables were held constant. As expected, elevated temperature accelerated the early stage of geopolymerization reaction, resulting in better performance properties of the lightweight geopolymer composites. For all compositions, the compressive strength of the specimens cured at 20 °C for 7 days could be slightly reached by accelerated curing at 60 °C after only 6 h, whereas the specimens cured at 60 °C for 24 h showed the same strength if cured at 20 °C for 14–28 days. Therefore, it was concluded that, when the cost is taken into account, it is important to increase the curing temperature of the samples to gain strength quickly when higher strengths are intended to be achieved during a shorter period of time. This work is also in agreement with the research done by Hardjito et al. (2005). They found that fly ash-based geopolymer concrete cured at 60 °C up to 48 h shows an increase in its compressive strength. Al Bakria et al. (2011) reported that the maximum compressive strength of fly ash-based geopolymers was obtained at 60 °C. Rovnaník (2010) and Görhan et al. (2016) also reported the significant roles of temperature and the curing time based on their findings.

According to the data, the compressive strength development of lightweight geopolymer composites has been increased by increasing the curing time from 6 to 24 h and from 7 to 28 days. The increase in strength was affected by the increase of

the duration of the curing periods. The curing period of 24 h and 28 days produced the maximum compressive strength development. Longer curing time improved the polymerization process resulting in higher compressive strength. At longer curing duration, the geopolymer developed slowly and its quality is better in terms of lower porosity and higher strength. This suggestion is supported by comparison of the density of the specimens (Fig. 3). The density value increases with rising time of curing.

Figure 6 shows the representative FESEM images of the fracture surface of the lightweight concrete composite with and without the addition of wood aggregates after compression strength test. It is evidently noticeable that the dense and compact structure with heterogeneous, some un-reacted phases, pores, crack bridging, fiber pull-out and rupture, and matrix fracture are observed. In the geopolymer matrix

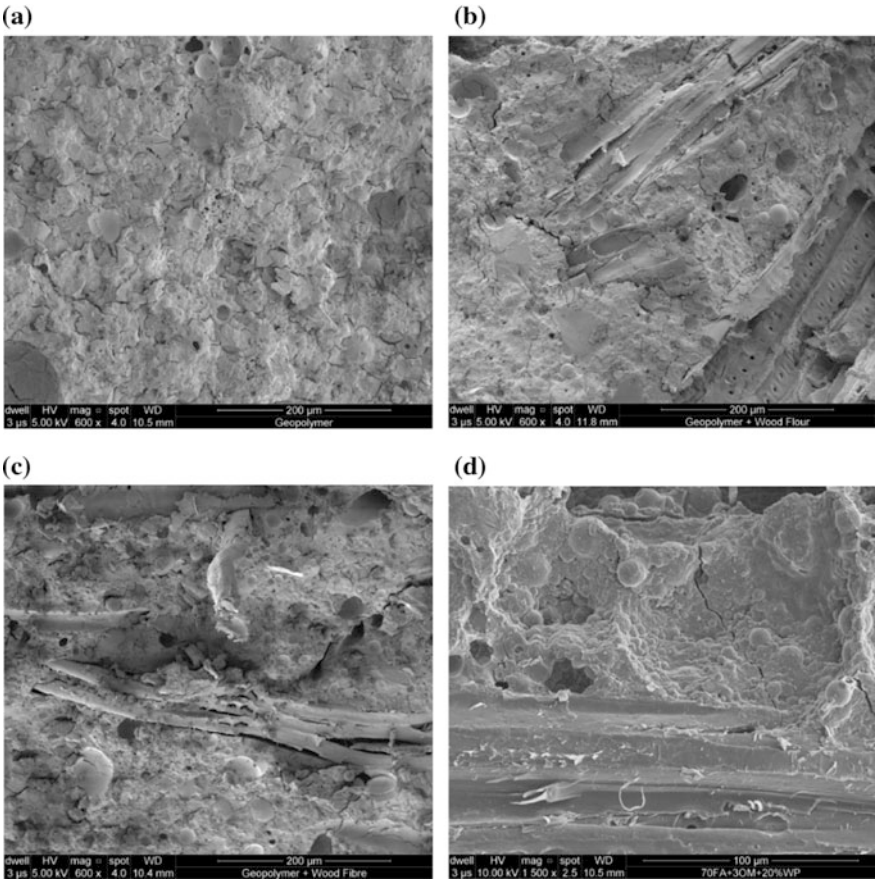

Fig. 6 FESEM image showing **a** plain geopolymer at 600× magnification, **b** geopolymer with C100 at 600× magnification, **c** geopolymer with WF at 600× magnification, and **d** geopolymer with WP at 1500× magnification

without wood aggregate addition, there was only dense aluminosilicate matrix (Fig. 6a), whereas the samples with wood aggregates addition contained aluminosilicate matrix and wood aggregates embedded in the matrix (Fig. 6b–d). The cracks observed in microstructure were believed to be caused by the compressive strength test. Closer inspection shows that some wood aggregates have good bonding with the matrix (Fig. 6b, d). It can also be observed extensive fiber pull-out (Fig. 6c) with traces of matrix adhere the WF which is an indication of good WF-matrix adhesion. Wood aggregates with good bonding with the surrounding matrix tend to fracture rather than pull-out at the failed surface. Wood aggregates poorly bonded to the geopolymer matrix contribute little to the improvement of the compressive strength, ductility, and toughness, and sometimes they may act as a flaw or crack initiation deteriorating the strong performances of the concrete composite.

3 Conclusion

The experimental results reported in this research indicate the significant effects of curing conditions on the compressive strength of lightweight geopolymer composite prepared using different types of wood aggregates. The results show that the compressive strength of the geopolymers increases with increase in the duration of heating periods. The samples with C100 were observed to have the optimum compressive strength values. It was determined that an increase in the curing temperature and curing time increased the compressive strength while it did not have a significant effect on the physical properties.

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