Lightweight Geopolymer Wood Composite Synthesized from Alkali-Activated Fly Ash and Metakaolin

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Graphical abstract
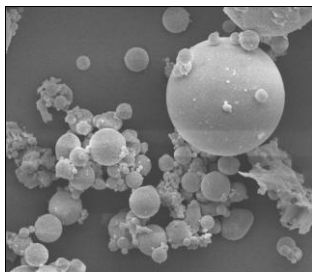
Abstract

This research paper presents the report on the use of wood particle as reinforcement to produce lightweight geopolymer wood composite. In this study, Class F fly ash and metakaolin used as alumino-silicate source were activated by alkaline activator and samples were cured at two different temperatures, 80°C and 20°C. Wood particle, 3 to 5 mm in size was added to fly ash/metakaolin based geopolymers for 10 – 60% solid content, and its influence on the compressive strength has been investigated. The microstructure investigation using field emission scanning electron microscopy (FESEM) illustrated fly ash; metakaolin and wood particle were embedded in a dense alumino-silicate matrix, though there was some unreacted phase occurred. The compressive strength of lightweight geopolymer matrix without wood particle added samples cured at 80°C temperature was 5.4 MPa which significantly increased 45% when the 10% wood particle were added. However, the addition of wood particle from 20 to 60% has not shown a major improvement in compressive strength. It was found that the density of the lightweight geopolymer composite was decrease relevant to the amount of wood particles addition. It is suggested that wood particle are one of the potential candidates as reinforcement for lightweight geopolymer composite development.

Keywords: Fly ash, metakaolin, lightweight concrete, alkaline activator, wood particles

Abstrak

Penyelidikan ini adalah mengenai penggunaan serbuk kayu sebagai bahan penguat untuk menghasilkan komposit kayu-geopolymer berketumpatan rendah. Dalam kajian ini, *class F fly ash* dan *metakaolin* digunakan sebagai sumber alumino-silikat diaktifkan dengan pengaktif alkali dan sampel dikeringkan pada dua suhu yang berbeza, 80 ° C dan 20 ° C. Serbuk kayu bersaiz 3-5 mm ditambah sebanyak 10 - 60% berasaskan kandungan pepejal dan pengaruhnya ke atas kekuatan mampatan komposit kayu-geopolimer berketumpatan rendah dilaporkan. Penelitian mikrostruktur menggunakan FESEM mendapati *fly ash*, *metakaolin* dan serbuk kayu telah bersatu dalam matriks alumino-silikat, walaupun terdapat beberapa fasa tidak bertindakbalas. Kekuatan mampatan matriks komposit kayu-geopolimer ringan tanpa serbuk kayu yang dikeringkan pada suhu 80 ° C adalah 5.4 MPa yang ketara meningkat 45% apabila 10% kayu zarah telah ditambah. Walau bagaimanapun, penambahan zarah kayu 20-60% tidak menunjukkan peningkatan yang besar dalam kekuatan mampatan. Kesimpulannya, serbuk kayu berpotensi sebagai penguat bagi menghasilkan komposit kayu-geopolimer berketumpatan rendah.

Kata kunci: *Fly ash, metakaolin*, konkrit ringan, pengaktif alkali, serbuk kayu

1.0 INTRODUCTION

Lightweight concrete (LWC) is an innovative product in the construction sector. It offers advantages such as reduction of deadweight in structures, better thermal and sound insulation for buildings, and lower transportation costs [19, 26]. Lightweight concrete has light densities ranging from 300 to 1800 kg/m³ [10]. From a materials perspective, the difference between normal concrete and LWC is that there are no coarse aggregates, which makes LWC a lighter product. According to Ahmaruzzaman [5], LWC is classified into three types depending on the method of production: LWC with lightweight aggregate, LWC with artificial air bubbles, and LWC with no fine aggregate.

Wood and other lignocellulose materials in the composite act as aggregates or reinforcing materials. According to Shaikh [22], the addition of synthetic or natural aggregates resources to concrete changes its brittle behavior to ductile or quasi-ductile with significant improvements in tensile strength, tensile strain, toughness, and energy absorption capacities. Besides its availability and low cost, the advantages of using natural resources in concrete composites are low density, biodegradability, acceptable specific properties, recyclability, and environmentally friendliness [6, 10]

Due to their performance properties, geopolymers have been evaluated as potential replacements for inorganic binders in lightweight concrete [2, 10, 15, 21, 26]. Geopolymer formation involves a chemical reaction with alumino-silicate reactive materials and strong alkali solutions, which forms a rigid polymer. Many materials that are rich in Al and Si in amorphous form are sources for geopolymers. Some materials, such as fly ash, furnace slag, silica fume, metakaolin, pozzolan, building residues, and natural minerals are also potential sources for geopolymer [15]. The development of geopolymer concrete has produced more environmentally friendly construction materials that reduce greenhouse gas emissions during their production compared with ordinary Portland cement.

There is very limited information on the use of wood particles as aggregates in lightweight geopolymer concrete for structural and non-load bearing applications. Therefore, for this research, geopolymer and wood particles were combined together to study the effect of several mixes of wood particles to the properties of lightweight geopolymer wood composite and the feasibility of using wood particle as reinforcement aggregates in geopolymer concrete. The result is very important for the understanding and future improvement for this composite. Lightweight concrete wood composites will be prepared using fly ash, metakaolin, and wood particles. In LWC containing different percentages of wood particles, important properties such as density, compressive strength, and porosity were determined; LWC was also examined by infrared spectroscopy

(FT-IR) and field emission scanning electron microscopy (FESEM).

2.0 METHODOLOGY

Class F fly ash, provided by GK Kiel GmbH power plant in Kiel, Germany, and Metakaolin-Argical M1000, obtained from IMERYS Refractory Minerals in Clérac, France, and were used as the starting materials to prepare geopolymers. The chemical composition of the fly ash and metakaolin are tabulated in Table 1.

Table 1 Chemical Composition (% Mass) of Fly Ash and Metakaolin

Oxides Materials	Class F Fly ash	Metakaolin
SiO_2	56.8	55.0
Al_2O_3	23.8	40.0
Fe_2O_3	6.79	1.4
CaO	2.9	0.15
MgO	1.28	0.15
Na_2O	0.43	0.4
K_2O	1.99	0.4
TiO_2	0.94	1.5
LOI	3.5	1.0

*LOI = Loss on ignition at 1050 °C for 1 h

The microstructure of fly ash and metakaolin were studied by field emission scanning electron microscopy (FESEM) using Quanta FEG Type 250, FEI Electron Optics (SN: D9122), Netherlands. As seen in Figure. 1(a), the fly ash contained spherical shapes with relatively smooth outer surfaces of different sizes. Most particles were hollow and filled with smaller particles in their interior. The surface of the fly ash included some quartz particles with a size distribution between 930 nm to 25000 nm. FESEM micrographs of metakaolin (Figure. 1(b)) revealed vitreous unshaped fragments. It was clearly identified as non-crystallized, lamellar particles. The particle size distribution of metakaolin was 63 μm to 200 μm.
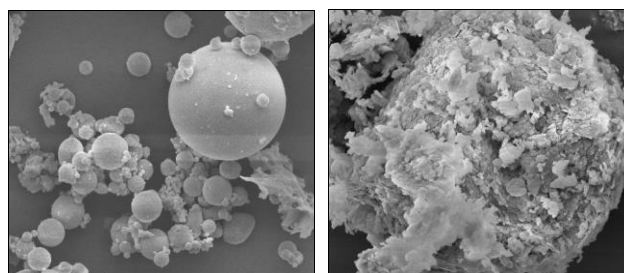
(a)   (b)

Figure 1 FESEM image of (a) fly ash with 5000x magnification and (b) metakaolin with 10000x magnification

Analytical grade sodium hydroxide in pellet form (NaOH with 98% purity) from Fisher Scientific UK Ltd, Bishop Meadow Road, Loughborough and sodium silicate solutions (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, were used as the alkaline activators. The foaming agent, hydrogen peroxide (H_2O_2) is a colorless liquid, slightly more viscous than water and has a density of 1.135g/cm^3 at 20°C. Wood particles, as an aggregate agent, were obtained from a local particleboard mill in Germany. The longish wood particles of mixed softwood, which were used as coarse core layer particles for particleboard manufacturing, were 3 to 5 mm in size and had 6.4% moisture content.

The liquid alkaline activator was prepared by mixing a 10 M sodium hydroxide solution with sodium silicate solution at a weight ratio of 2.5:1. The activator solution was thoroughly mixed and stored at ambient conditions for at least one day prior to use. Fly ash at 70 wt.% and 30 wt.% metakaolin were mixed dry with 10 to 60 wt.% wood particles in a pan mixer for 3 min to provide a uniform solid supply. Specimens without wood particles were also prepared as controls. The alkaline solutions 5wt% of hydrogen peroxide was added to the solids, which were thoroughly mixed for another 3 to 5 min to ensure homogeneity. The ratio of alkaline activator to solid material was increased due to the increasing percentage of wood particles in order to improve the workability between solid and fluid.

Table 2 Design Mix for Lightweight Concrete Wood Composites

Sample	Composition of Mixture				
Wood particles added (%)	Fly ash (%)	Metakaolin (%)	H_2O_2 (%)	Alum : Alkaline	Na_2SiO_3 : NaOH
0	70	30	5	2.0:1.0	2.5:1.0
10	70	30	5	2.0:1.0	2.5:1.0
20	70	30	5	2.0:1.0	2.5:1.0
30	70	30	5	2.0:1.0	2.5:1.0
40	70	30	5	2.0:1.33	2.5:1.0
50	70	30	5	2.0:1.33	2.5:1.0
60	70	30	5	2.0:1.33	2.5:1.0

Table 2 describes the mix design used in this study. The fresh mixture had a stiff consistency and was glossy in appearance. The resulting pastes were cast in 50 mm cubic steel molds and were immediately wrapped by plastic film to avoid moisture evaporation during curing. The samples were cured at two different conditions: room temperature (20 °C) for 7 days and at 60 °C in an oven for 24 h; for each condition, they were removed from the molds after 24 h. After curing, the 50 mm cubic samples were stored in plastic bags for 24 h at room temperature to complete the reaction and to avoid drastic changes in environmental conditions before testing.

Before testing, the test specimens were weighed to determine the density of the LWC. The compressive strength of the specimens was performed on cubic samples (50 mm × 50 mm × 50 mm) at different curing conditions in accordance with the ASTM C109 / C109M-12 [8] standard. The average values from the five samples were reported. The compressive strength test was performed on a Mohr and Federhaff AG testing machine Hamburg, Germany using a speed rate of < 10 mm/min.

The porosity of each cube sample was measured according to the ASTM C642-06 [9] standard with some modification. Three specimens from each batch were oven-dried at 105 °C ± 5 °C for 24 h to achieve a constant weight before testing. The porosity was calculated using Eq. 1:

$$\text{Porosity (\%)} = (W_s - W_d) / W_d \times 100 \qquad (1)$$

Where W_s is the saturated weight (g) and W_d is the oven-dry weight (g).

The change in the chemical structures in each sample was analyzed using Fourier-transform infrared spectroscopy (type Vector 33 and Hyperion 1000/2000, Bruker, US). The IR spectra were determined in the region of 100 cm^{-1} to 4000 cm^{-1} employing the KBr pellet technique. Pelletized disks containing 2 mg of the sample and 298 mg KBr were prepared. The microstructure of the samples was determined using Quanta FEG Type 250, FEI Electron Optics (SN: D9122), Netherlands. Specimens were crushed into small pieces before being coated with a thin layer of gold (to avoid charging).

3.0 RESULTS AND DISCUSSION

Density and Porosity

The porosity and density of LWC wood composite at different curing conditions are shown in Figure 2 and 3. The results showed the reduction of density values at increasing levels of inclusion of wood particles. The lowest density results were found in the mixture with 60 wt.% wood particle aggregates, which were 413 kg/m^3 at 80 °C and 372 kg/m^3 at room temperature. Generally, an increase in wood particle aggregates led to a lower density. These results were in agreement with the work done by Alomayri *et al.* [6] Abdul-Kader *et al.* [1], and Timakul *et al.* [24] where the density of cement composite was decreased by the addition of natural fiber. The density reduction of LWC wood composite might be attributed to a lower specific gravity and density of wood particles itself.

An increase in the curing temperature increased the density of the LWC wood composite. Figure 2 represents the influence of curing temperature on the density of the samples which can indicate that higher curing temperature makes the hardened

structure of LWC wood composite denser, and hence more compact. Wu and Sun [26] also found that an increase in the curing temperature increased the density of the lightweight samples. This could be attributed to the rapid formation of hard structure at elevated temperature. When the process of hardening proceeds too quickly, it results in less ordered structure with relatively uneven pores sizes distribution compared to LWC wood composite cured at room temperature.
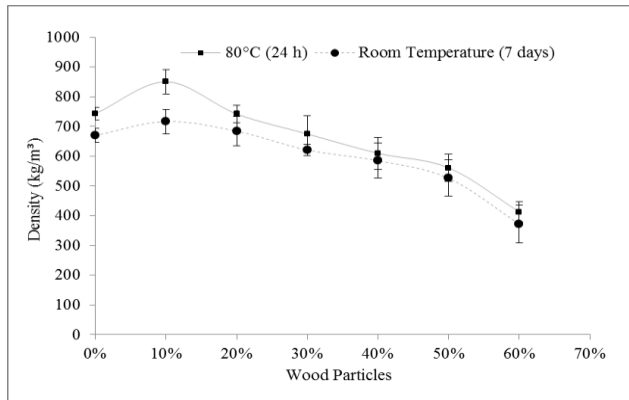

Figure 2 Variation of density as a function of wood particle content at two different curing conditions
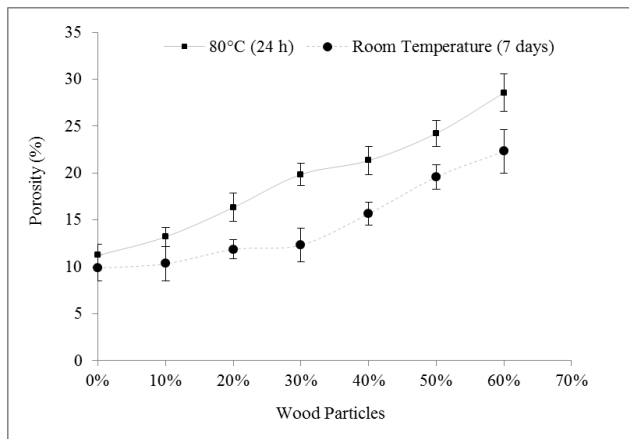

Figure 3 Variation of porosity as a function of wood particle content at two different curing conditions

The value of the increase in porosity with the increases in the weight percent of wood particles aggregates is shown in Figure. 3. The lowest value of porosity (11.23% for room temperature curing and 15.23% for 80 °C curing) was found in control sample that contained no wood particles aggregates, whereas the lightweight sample containing the highest amount of wood particles (60%) had the highest porosity of 52.17% for 80 °C curing and 45.16% for room temperature curing.

The liquid content had the most influence on the density and porosity. To improve the workability between the geopolymer mixtures while increasing the percentage of wood particles, a high ratio of

alkaline activator to solid material was required, this caused an increase of porosity in the resulting LWC wood composites. A study done by Alomayri *et al.* [7] showed that the addition of extra liquid results in large amounts of "free" liquids that become trapped in inter-granular spaces or in large pores; after geopolymerisation and evaporation during curing and extended ageing, there are many inter-granular pores in the microstructures which contribute to the increased porosity of LWC wood composite.

At high curing temperature, the porosity of LWC wood composite was higher compared to room temperature curing. It is possible that the higher temperatures results in uneven structures of pore volume and pore size in hard structure of LWC wood composite. In addition, the wood particle itself had a potential to be clumping together during mixing resulted in undesirable for achieving a uniform microstructure.

Compressive Strength

The compressive strength was measured on the lightweight sample cured at 80 °C for 24 h and on the sample cured at room temperature for seven days (Figure. 4). The highest compressive strength, found in the 80 °C cured composites, was 14.37 MPa with 10 wt.% wood particles addition, whereas the compressive strength of the room temperature cured sample was 11.19 MPa with 10 wt.% wood particles. Compared with the samples without wood particles, the 10 wt.% wood particle samples had more than 60% improvement in the compressive strength for both curing conditions. It can be seen that the compression strength of LWC wood composite increase with higher wood particle content up to 10wt% and then decreases thereafter. This could be attributed to the presence of wood particles at the optimum content which can effectively carry more compression load and thus delay the growth of the microcracks. However, further increase of the wood particles content induces poor workability and wood particle agglomeration, resulting in nonuniform wood particles dispersion, heterogeneous and less dense structures. These flaws may lead to stress concentrations and degrade the compressive strength of LWC wood composite. Similar trend was also reported by Alomayri *et al.* [7] and Timakul *et al.* [24] for fly ash based geopolymer reinforced with natural fibers. In addition, the inclusion of wood particles is not to increase the compressive strength (sometimes it may decrease the compressive strength), but to control the cracking of the reinforced composite by bridging across the cracks and providing post-cracking [22].
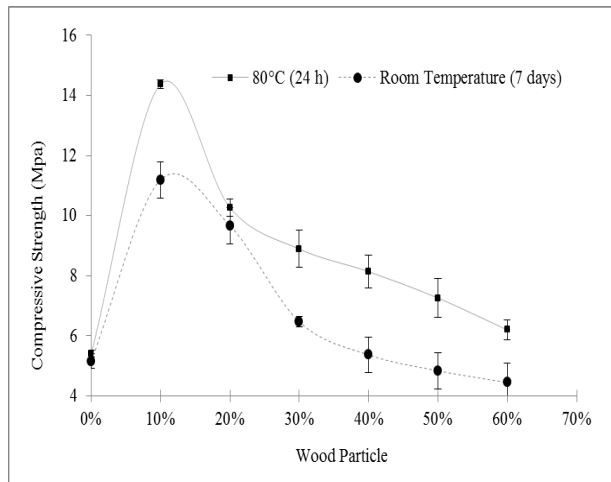
Figure 4 Compressive strength of LWC wood composites with different wood particle contents and curing conditions

Figure 5 FESEM of lightweight concrete with 0 wt.% wood particles at 600x magnification

Both the lightweight samples and the control samples showed a positive enhancement of strength when they were cured at an elevated temperature (Figure. 4). The compressive strength development seems to depend mainly on the binder (alkali activated fly ash and metakaolin) despite amount of wood particles itself. Wu and Sun [26] stated that the strength of the fly ash-based lightweight inorganic polymer develops rapidly when it is cured at higher temperature.   This effect was attributed to the geopolymer, which gains higher compressive strength at an elevated temperature due to the hydration products that are formed [3, 4, 26].

Microstructures and FT-IR Analysis

Microstructures in the lightweight samples were observed by FESEM (Figures. 5 and 6). The FESEM analysis was performed only on 0 wt.% and 20 wt.% wood particle samples. The synthesized lightweight samples were compact, but they presented micro-porosity and nano-porosity, which could have formed during curing or polycondensation. As expected, the existence of these pores resulted in decreased weight, which led to the lightweight concrete classification.
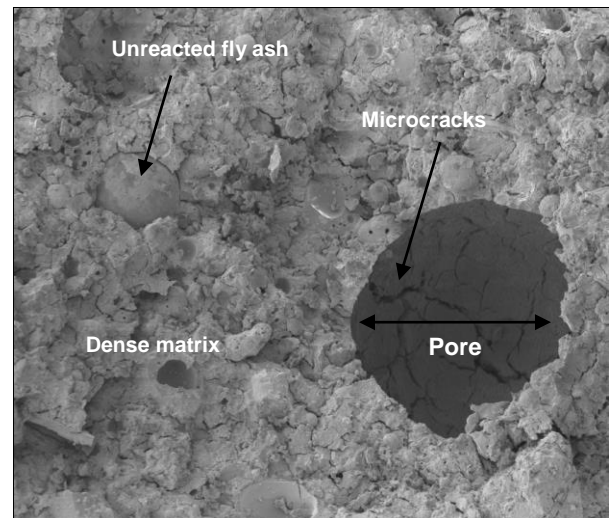
Dense matrix area in Figure 5 and 6 showed that the alkaline activator and the aluminosilicate; fly ash and metakaolin had already reacted. However, at high magnifications, some small areas (200 µm) had undissolved solid particles of fly ash. Unreacted fly ash was present in samples with and without wood particles. Fly ash, with its original spherical shape, was very well bonded within the amorphous geopolymeric framework of aluminosilicates (Figure. 6). The interfaces of unreacted fly ash comprised the most sensitive areas of the lightweight samples during the compression strength test. As shown in Figure. 5, the grains around the unreacted fly ash were detached from the amorphous framework, which explained the failure of geopolymeric materials. Both lightweight samples also contained microcracks, which contributed to their lower compressive strength and increased porosity.
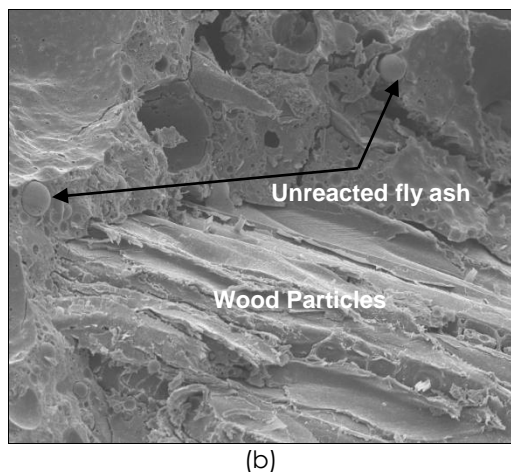

(a)

(b)
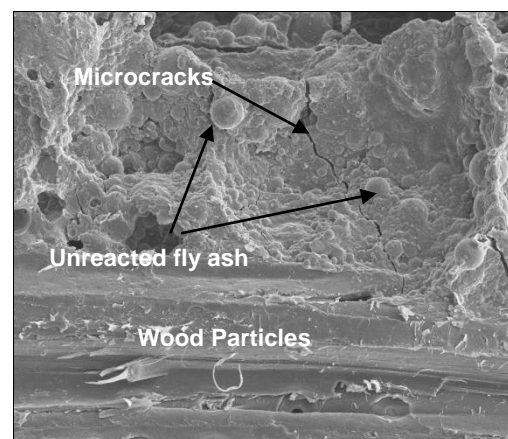
Figure 6 FESEM magnification of lightweight concrete with 20 wt.% of wood particles at (a) 1500x and (b) 1200x magnification

Figure 7 FT-IR spectra of (a) metakaolin, (b) class F fly ash, and (c) geopolymer pastes in the region of 100 to 4000 cm^{-1}

Figure 6 contains micrographs of the lightweight samples with wood particles. There was good adhesion between the wood particles and the geopolymer matrix at the interfacial zone. The bonding between wood particles and the geopolymer matrix was crucial to the properties of the concrete composite. The wood particles may control cracking and increase the fracture toughness of the brittle matrix through a bridging action during the micro- and macro-cracking of the matrix [12, 22]. In this study, the lightweight samples with 10% wood particles had better compressive strength (Figure. 4). This result suggested that the inclusion of wood particles strengthened and toughened the geopolymer concrete, which can effectively carry more compression load and thus delay the growth of the microcracks.

Figure 7 shows the FT-IR spectra of metakaolin, the class F fly ash, and the geopolymer pastes after curing. All spectra contained broad absorption bands at about 3427 cm^{-1} and 1705 cm^{-1} resulting from hydration water (-OH) and bending (H-O-H) vibrations of water, respectively [17,20]. The metakaolin spectrum showed sharp peaks at about 1080 cm^{-1} and 582 cm^{-1}, which were assigned to the internal vibration of Si-O-Si asymmetrical stretching and Al-O-Si bending vibrations, respectively [14]. The band at about 797 cm^{-1} was ascribed to Al(VI)-OH stretching vibrations in kaolinite [25].
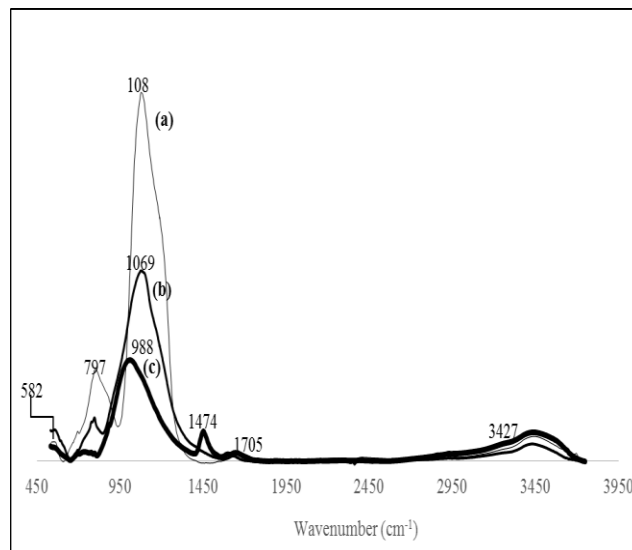
The FT-IR spectrum of fly ash shows the main absorption band at about 1069 cm^{-1} due to the Si-O-Si and Al-O-Si asymmetric stretching vibration [13]. The main absorption spectrum for both fly ash (1069 cm^{-1}) and metakaolin (1080 cm^{-1}) were shifted to the lower frequency in the geopolymer pastes (988 cm^{-1}), which indicates the formation of a new product (the amorphous aluminosilicates) due to the dissolution of fly ash and metakaolin in the alkaline activator [4,23]. The band at about 1474 cm^{-1} was attributed to the stretching of the O-C-O bands, demonstrating the presence of carbonate trace that occurs due to the atmospheric carbonation of a high alkaline NaOH aqueous phase, which was diffused on the geopolymeric materials surface [11, 18, 23].

4.0 CONCLUSION

From this study, the following conclusions can be reached:

(1) Lightweight concrete wood composite can be made from fly ash, metakaolin and wood particles. With 10% wood particles, significantly improved the compressive strength, which was 62% higher than without wood particles LWC. The addition of more than 30 wt. % of wood particles did not shown a major effect on the compressive strength improvement when compared to 10 wt. % wood particle addition, which might due to the poor workability and led to wood particle agglomerations.

(2) Obviously, the density of lightweight concrete wood composite decreased with an increase in wood particle content.

(3) Strength of the lightweight concrete wood composite increased rapidly when it is cured at

higher temperature than at room temperature. It is recommended that during curing, the samples should be wrapped to preserve moisture, and to prevent shrinkage cracks.

(4) The inclusion of wood particle in the concrete decreases workability, thus demanding more amount of alkaline activators, NaOH and sodium silicate.

Acknowledgement

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