The Mechanical Properties and Microstructure Characters of Hybrid Composite Geopolymers-Pineapple Fiber Leaves (PFL)

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Abstract. The objective of this research is to study the influence of organic fibers on the mechanical properties and microstructure characters of hybrid composite geopolymers-pineapple fibers (PFL). Geopolymers were synthesized by using alkali activated of class C-fly ash added manually with short pineapple fiber leaves (PFL) and then cured at 60ºC for 1 hour. The resulting composites were stored in open air for 28 days prior to mechanical and microstructure characterizations. The samples were subjected to compressive and flexural strength measurements, heat resistance as well as acid attack (1M H_2SO_4 solution). The microstructure of the composites were examined by using Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS). The measurement showed that the addition of pineapple fibers was able to improve the compressive and flexural strength of geopolymers. The resulting hybrid composites were able to resist fire to a maximum temperature of 1500ºC. SEM examination showed the presence of good bond between geopolymer matrix and pineapple fibers. It was also found that there were no chemical constituents of geopolymers leached out during acid liquid treatment. It is concluded that hybrid composite geopolymers-pineapple fibers are potential composites for wide range applications.

1. Introduction

The term of hybrid composite implies material contained more than one type of material such as organic fibers which act as reinforcement [1]. This has a potential to provide improved properties such as toughness, strength and ductility, and fire resistance [2]. Basically, composite comprise of two parts, matrix and aggregate. In recent years, organic fibers have received numerous attention due to their excellent properties such as high tensile and compressive strength, and difficult to crack [3]. Organic fiber used in this research is pineapple fiber extracted manually from pineapple leaves. Pineapple is a well-known fruit all over the world. Pineapple leaves have attracted global attention because it creates environmental problems. New efforts are needed to overcome the environment impact due to the abundance of pineapple leaves dumped on the ground especially around the traditional markets. In recent years, for example, pineapple leaves have been used in the production of rough textile [4]. The addition of fibers as aggregate in the production of composite is intended to reduce the volume of the matrix [5]. Besides that, fibers show good adhesion properties and capable to control micro crack along the matrix [3]. Pineapple fibers have high cellulose and it is known that the quality of fibers depends on the amount of cellulose [4, 6]. The tensile strength of pineapple fiber is about 1088.60 MPa and its modulus Young is around 6441,6 GPa [7]. The length and the content of fiber in composite were found to increase the tensile and the impact strength of the material [8]. Other study showed that pineapple fiber leaves (PFL) exhibited superior mechanical properties due to its nature high-cellulose content and relatively low

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micro-fibrillar angle (14°) [9]. The matrix used in this study was geopolymer synthesized trough alkali activated fly ash (class-C). Geopolymers have a potential for a wide variety of applications, whether use pure, with fillers or reinforced [10]. A number of research have utilized fly ash as matrix and inorganic fibers as aggregate in the production of geopolymer composite (particularly concretes) and reported good mechanical properties, low thermal conductivity and high fire resistance [11, 12, 13].

2. Experimental Method

Geopolymers paste were synthesized by using class-C fly ash activated with alkaline solution. The molar ratio of the mixture were SiO_2/Al_2O_3 = 3.0, Na_2O/SiO_2 = 0.2, and H_2O/N_2O = 10.0. Pineapple fiber leaves (PFL) was extracted manually and soaked into 0,5M NaOH for 1 hour, dried and cut to about 5.0 mm in length. The composites were produced by varying the mass of aggregate relative to the mass of fly ash with a variation of 0%, 0.2%, 0.4%, 0.6%, 0.8% and 1.2%. The mixture of geopolymer paste and PFL was molded and cured at 60ºC/1 hour. The resulting materials were demolded and left in open air for 28 days before conducting any measurements. The size and the shape of the sample were customized in accordance with the measurement and characterization requirement such as flexural strength, fire resistance, acid attack, and SEM-EDS.

The compressive strength of the samples were measured by using universal testing machine with a maximum load of 50 ton, and the flexural strength was measured by using three bending flexural strength method. The level of crystallinity and chemical compositions of fly ash were measured by using Rigaku Mini Flex II X-Ray Diffraction operated at 30 kV and scan speed 0.2°/s. The morphology and the element compositions of fly ash was observed by using Tescan Vega 3 SB Scanning Electron Microscope couple with Bruker Energy Dispersive Spectrometer (EDS). The fire resistance of the composite was examined by exposing the sample surface on fire for 20 – 30 minutes and measured the temperature around 2.0 cm from the fire spot by using a thermocouple. The acid resistance of the samples was measured by immersing the sample into H_2SO_4 solution for 7 days.

3. Result and Discussion

Figure 1. Shows the sample of hybrid composite PFL-geopolymers with a size of (6.0 x 3.0 x 3.0) cm produced in this study. Physically, the sample appear glossy and no apparent cracks observed on the surfaces of the samples.

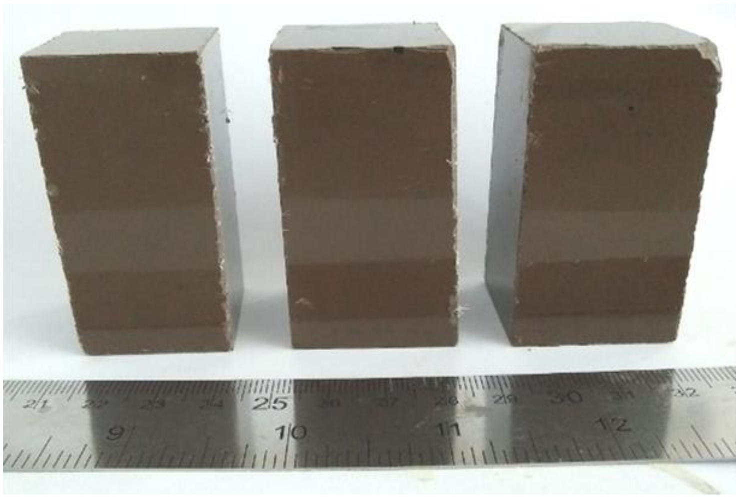
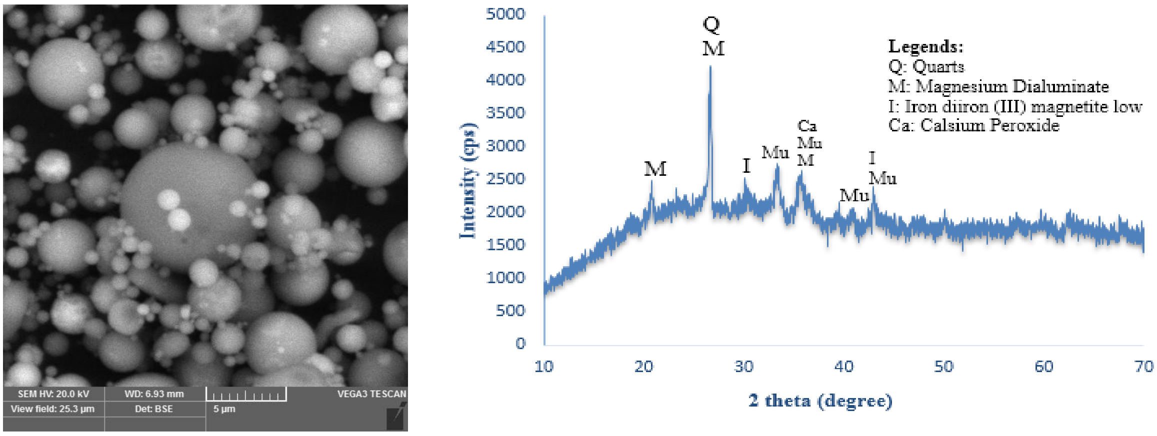


Figure 1. Example of hybrid composite geopolymers-PFLproduced in this study.

Figure 2 (a) and (b) shows the SEM image and diffractogram of fly-ash, respectively. The image shows the spherical shape of fly ash morphology with a particle size smaller than 10 μm. The size of fly ash particles suggest that its surface area is large and hence the reactivity of fly ash and sodium silicate solution is high. The diffractogram showed that the crystallinity level of fly ash is fairly high and the main chemical compositions of fly ash were quarts, magnesium, iron diiron magnetite and calcium peroxide. The wt% of elements and oxides of fly ash were examined by using Energy Dispersive Spectroscopy (EDS) as shown in table 1.



(a) (b)

Figure 2. SEM image (a), and X-ray diffraction pattern (b) of fly ash used in this study

Table 1. The wt% of elements and oxides of fly ash measured with EDS

No	Element	Atom (at. %)	Compound norm.	wt %
1	Oxygen	58.50		0.00
2	Sodium	0.76	Na_2O	0.99
3	Magnesium	3.65	MgO	6.20
4	Aluminium	10.36	Al_2O_3	22.25
5	Silicon	11.89	SiO_2	30.10
6	Sulfur	0.15	SO_3	0.51
7	Potassium	0.70	K_2O	1.39
8	Calcium	5.90	CaO	13.95
9	Titanium	0.36	TiO_2	1.20
10	iron	7.73	FeO	23.41

Table 1 shows that the molar ratio of SiO_2 and Al_2O_3 is in the range of good geopolymers production. The high content of CaO however make the geopolymers paste to set in a very short time and difficult to control in large quantity.

Table 2 shows the results of mechanical measurements of the resulting composites. It can be seen that the density of geopolymers paste was around 2,02 g/cm^3 and slightly decrease as the mass of fibers increases. The magnitude of compressive strength increase as the concentration of fibers increase. However, as the mass of fibers reached 1.2% relative to the mass of fly ash the bond between the matrix and the fibers weaken and as a result the compressive strength decrease significantly as in sample NN 04. The flexural strength of all composites increase as the mass of the fiber increase. This is understandable since the presence of fibers will enhance the ability of the geopolymer matrix to resists external load by sharing the stress-strain with the fibers.

Table 2. The average value of density, compressive and flexural strength of the samples.

NO	Sample	Density (g/cm^3)	Compressive Strength (MPa)	Flexural Strength (MPa)
1	NN 01	2.02	23.33	6.00
2	NN 02	1.97	41.57	6.83
3	NN 03	1.97	58.24	7.07
4	NN 04	1.99	25.20	7.09

Fire resistance measurement was conducted by using flame torcher and a thermocouple was inserted around 2.0 cm from the flame spot (Figure 3) to register the temperature as a function of time. The temperature of the flame used can reach 1500°C and the sample was exposed to fire for 30 minutes. The measurement was conducted to investigate the ability of geopolymers matrix to protect the organic fibers from intense fire or heat.

Figure 3. Fire resistance measurement on the surface of the composite

The rise of the temperature at the point 2.0 cm from the fire spot was monitored every 2 minutes. Figure 4 shows the graph of temperature as a function of time for the fire source (a) and the temperature of the sample surface.

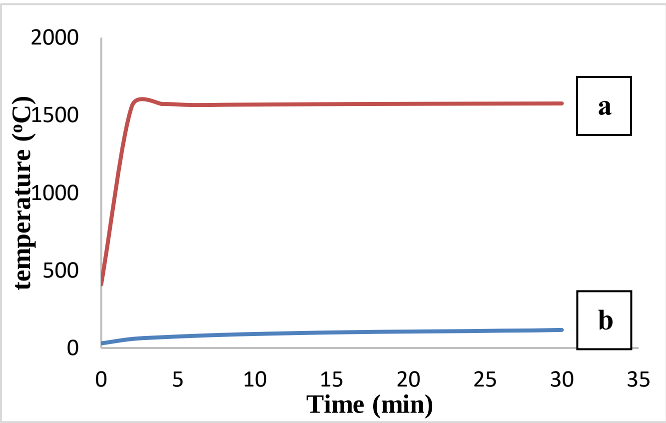


Figure 4. Temperature vs time (a) fire source (b) sample surface (2 cm from the fire)

Figure 4 shows that the temperature difference between the fire source and 2.0 cm from the fire spot is very high. This explains that geopolymer behave as excellent heat isolator and able to protect the fibers from burning. A research has been conducted to compare heat resistance between calcium silicate and geopolymer and reported that geopolymers are suitable for fire resistance materials [14]. Figure 5 show the condition of the samples after exposed to fire for 30 minutes. It can be seen that the sample did not show substantive damage or extensive cracks except on the area of exposed fire. This results confirmed that the addition of PFL on geopolymers did not change the properties of geopolymer as fire and heat resistance materials which suitable for high temperature ceramics and composite [15]. The thermal resistance of the sample was then observed by examining cracks development on the surface of the materials [16].

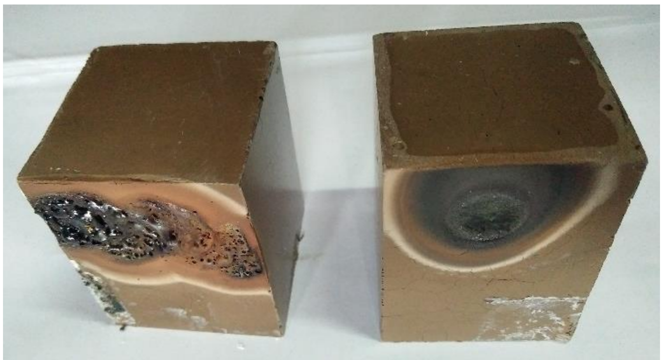


Figure 5. The surface of hybrid composite PFL-geopolimer after fire resistance measurement

The acid resistance of the composites was conducted by immersing the samples into 1M H_2SO_4 solution for 7 days. This measurement was conducted to investigate any damage on the surface of the samples or fibers due to the intrusion of strong chemical into the bulk of geopolymers. The physical condition of the samples after removing from the bath is shown figure 6. It can be seen that after 7 days immersed into strong acid solution, the samples remain strong although their color changed.

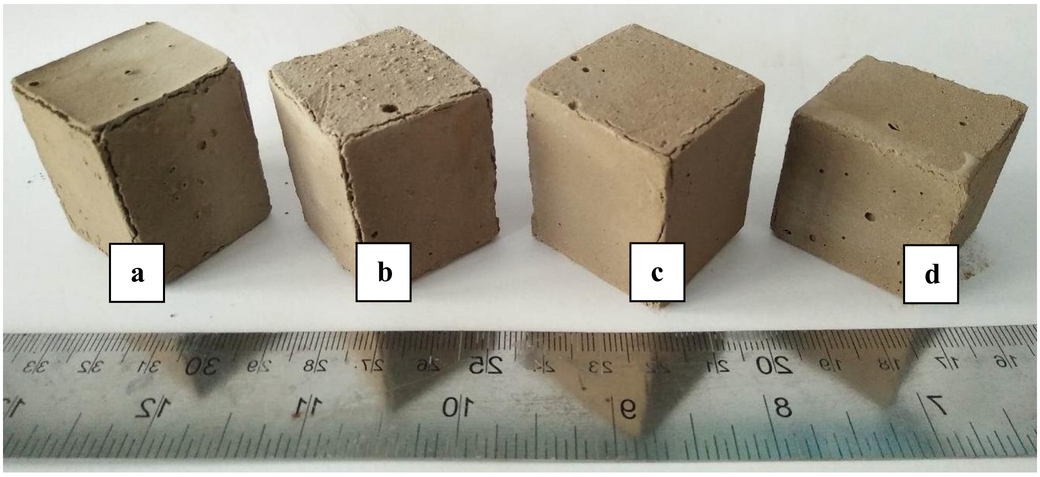


Figure 6. The samples after 7 days immersing in 1M H_2SO_4 solution (a) NN 01, (b) NN 02, (c) NN 03, (d) NN 04

It was observed that after 7 days, the pH of the acid solution remain 1 indicating that the sodium silicate in the network of geopolymer did not leached out and react with H_2SO_4 solution and hence increase the pH of the solution. It is worth mentioned that the samples contained higher volume of PFL were the most resistant to acid attack as there were no cracks observed on their surfaces. Extensive crack was observed on sample NN01 which is pure geopolymer. Altough it is not clear, apparently this result indicates that the PFL plays important role in preventing physical damage of the composite due to acid attack. Further study need to be conducted to confirm this finding. This finding is similar to a previous research on geopolymers concrete immersed in H_2SO_4 and found that there were no mass loss or physical damage observed [17, 18, 19].

Figure 7 shows SEM characterization of hybrid composite geopolymers-PFL. Figure 7(a) is the morphology of geopolymer without pineapple fiber. The surface of geopolymer consists of unfully reacted fly ash particles and other minerals originated from the raw materials

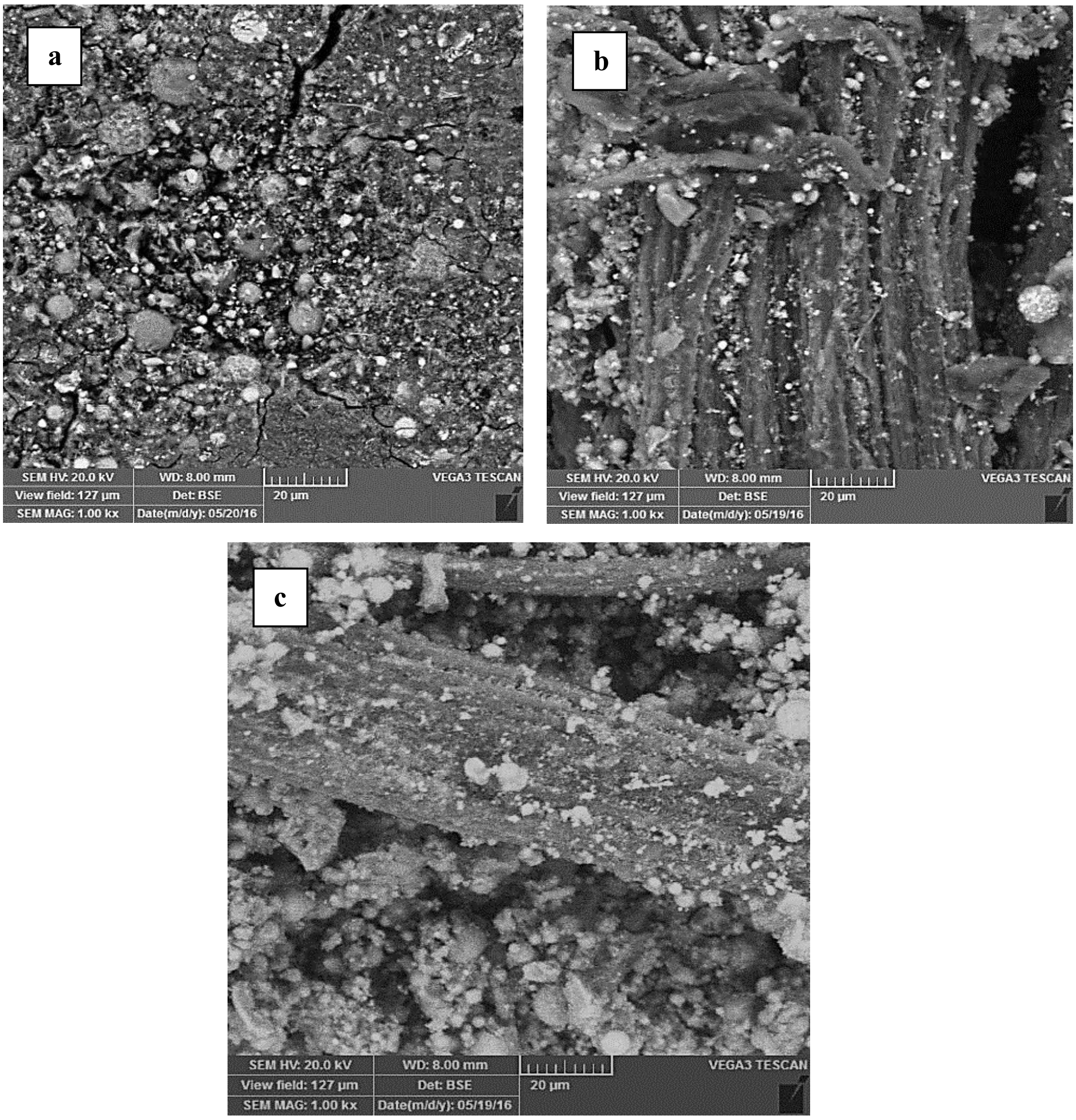


Figure 7. SEM image of hybrid composite PFL-geopolymer (a) 0% PFL (b) 0.4% PFL (c) 1.2 % PFL

Figure 7 (b) is SEM image of the sample contain 0.4% PFL in which the fibers surrounded by the matrix of geopolymer and the remaining fly ash particles. The space between fibers maybe as a result of polishing the sample prior to SEM examination. Figure 7 (c) is the image of the sample which contain 1.2% PFL. It can be seen that the volume of unreacted fly ash particle increase as the volume of fibers increase. This condition was undesirable since a strong between matrix and aggregate can only be achieved in more homogenous matrix. This is the main reason why the compressive strength of this sample is much lower than the other samples. Further work is needed to improve the chemical bond between the matrix and the fibers aggregate.

4. Conclusion
The addition of pineapple fibers into the geopolymers matrix produced a new type of inorganic hybrid composite. The fibers were found to improve the mechanical properties, compressive and flexural strength of the composites. The addition of pineapple fibers was also found did not change the nature of geopolymers as a fire or acid resistance material. Microstructure examinations show that the space between the geopolymers matrix and pineapple fibers reduce the mechanical strength of the resulting composite.

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