International Journal of Engineering & Technology, 7 (4.25) (2018) 109-113

International Journal of Engineering & Technology

Website: www.sciencepubco.com/index.php/IJET

Research paper

Effect of Particles Content and Size on Density, Void and Tensile Properties of Sokka-Clay/Phenolic Composite

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Abstract

Sokka clay particles, whose stability in mass and good inhibitor in flame, is capable to be used as filler in polymer composite. The investigation on tensile properties of Sokka-clay/phenolic composite at different particles content (0-50 vol%) and size (less than 74 µm up to 177 µm) has been performed. The analysis of chemical compound, voids fraction, density and fracture surface was conducted to complete the discussion. The Sokka-clay/phenolic composites have an increase in density along with an increase of particle content and a decrease of particle size. Voids also rise proportionally with an increases of particles content and size. The particles addition of 10 vol% on phenolic decreases its strength gradually. However, further addition of particles enhances its strength and modulus, and that is also similar with smaller particle addition. Otherwise, its addition causes a decrease of composite strain due to the lower strain of particles. The increase of strength is influenced by many factors, such as the amount of particle, particle size, movement of particle to fill empty space, effective contact of particle surface, and particle aggregation. The modified Sokka-clay particle has good potential to improve composite properties, and many explorations of its properties needed to fulfill the application requirements.

Keywords: *Sokka-Clay, Particle Content, Particle Size, Phenolic, Tensile*

1. Introduction

In 1979, Davidovits was the first researcher who introduced a new material of geopolymer to show a new class of alumina-silicate material. This material has driven many researchers to explore and optimize its mechanical and thermal properties. The geopolymer is polymer mineral produced from geo-synthesis or geochemistry of alumina-silicate polymeric and alkali silicate producing SiO_2 and Al_2O_3 which was bonded by *tetrahedral*. It can be use as coating, binder, matrix in composite, and cement in mortar [1]. The geopolymer material has many advantages, such as low density, good chemical resistance and fire resistance [2].

The development of composite material, which uses geomaterial (*alumino–silicate*) and polymer, is performed to get similar properties with geopolymer composite, for example clay-polymer composite. Many researchers have studied particulate composite to explore the optimization of its properties in accordance with the terms of application, such as the use of bentonite**,** magnesium hydroxide, aluminum hydroxide, clay, montmorilonite and carbon black [3, 4, 5, 6]. The composite has a good potential to be applied as car body panel or other structure which have high risk in fire. However, it should have good mechanical properties, durability, thermal stability and acid resistance [7].

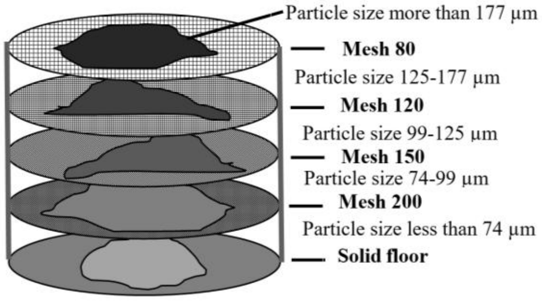
In subdistrict of Sokka in Kebumen, Central Java, Indonesia, there is a high quality local clay (Sokka-clay) which has been used to produce roof-tile with high mechanical properties by many Small Medium Enterprise (SME). In previous research, the compounds of the Sokka-clay were dominated by 54.59% of silica oxide (SiO_2), 19.62% of alumina oxide (Al_2O_3), and 13.03% of ferro oxide (Fe_2O_3), which can be applied as fire resistant [8]. It has been proved by Diharjo et. al. to enhance the fire risistance and reduce its cost of CFRP composite [9]. An amount of char coat which rise in burning test of Sokka-clay based composite provides an evidence that that the clay may act as a flame retardant [10]. Moreover, application of a certain number Sokka-clay on GFRP composite gives significant improvement in flame resistant [11]. The best method of fire protection is the incorporation of flame retardants to inhibit the combustion for resulting the satisfactory flame retardancy (12).

The studies of particulate composite material have been widely practiced, but there is no investigation of polymer composite filled with the local Sokka-clay particle. This study is focused on investigation of Sokka-clay/phenolic composite, especially on density, void content, tensile properties and fracture characteristic. All results are discussed comprehensively to meet the correlation of the properties.

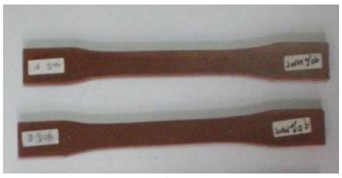
2. Experimental Method

2.1. Materials

The commercial bisphenolyc resin of LP-1Q EX was used as matrix to produce composite samples with additional methyl ethyl ketone peroxide (MEKP) of 2% (v/v) as hardener and promotor Cobalt P-EX of 0.5% (v/v) as promotor. The chemical materials were supplied by *PT. Justus Kimia Raya*, Indonesia. The used fire retardant was local clay particle made from rejected roof tiles with Sokka brand which was produced by Small Medium Enterprises (SME's) in Sokka, Kebumen, Central Java, Indonesia. The particles were crushed using a machine and sieved on mesh sizes of 80, 100, 120, 150 and 200. Then, the particle sizes were classified as 125-177 µm, 99-125 µm, 74-99 µm and less than 74 µm for mesh codes of -80/+120, -120/+150, -150/+200, and -200, respectively. The sieving process of the particles were illustrated in Figure 1a.



(a)



(b)

Fig. 1: (a) Scheme of sieving process and (b) tensile test sample

2.2. Specimen and Testing

The composite specimens were produced using a press mold method which controlled in thickness of 3 mm. The molding was prepared using flat glasses and its size is 165 x 19 x 3 mm. The Sokka-clay particles were finally dried in an oven at 105 ^{O}C for 35 minutes to evaporate its water content. Then, the particles were weighed using a digital balance to ensure its composition of each sample. Furthermore, the particles were mixed with phenolic resin, hardener and promotor using a stirrer at 70 rpm for 5 minutes. The materials should be poured in the molding before 15 minutes from the addition of hardener, and the molding was compressed with a weighting load. The composite was removed from the molding after 2 hours, and then it was cut to produce test samples. There are 2 kinds of specimen in this research, i.e. the composite varied with particles content for particle size of 74-99 µm and with particles size for particles content of 40 vol%. The size of tensile test samples was referenced to ASTM D 638 for type IV, as shown in Figure 1b. The tensile test samples have 115 mm overall long, 19 mm overall wide, 33 mm gage long, 6 mm gage wide and 3 mm thick.

The analysis of chemical compound was conducted with the use of secondary data obtained by XRF characterization of Sokka clay particle in previous research [8]. The evidence of void in the composite was analyzed by comparing the theoretical and measuring densities which were determined using Rule of Mixture Theory [13]. Besides that, the morphology of Sokka clay particle was also observed using Scanning Electron microscope (SEM).

The tensile test samples were conducted using a universal testing machine with speed of testing 5 ± 25% mm/min and rate of strain 0.15 mm/mm.min adjusted with ASTM D 638. The testing time of each sample was between 1.5 and 5 minutes and the distance of grips was maintained in 65 mm. The recorded data in this testing were of load and elongation.

3. Result and Discussion

3.1. Particle Characterization

According XRF analysis in previous research, the compound os Sokka-clay particles are dominated by 54.59% of SiO_2, 19.62% of Al_2O_3 and 13.30% of Fe_2O_3. They are good inhibitor of flame (flame retardant) due to the addition of particles producing a new advantage property in inhibition of flame [8, 11]. Based on Table 1, all compounds have high melting point due to the clay has been burned in tile processing at temperature more than 1000 °C which can evaporate all organic compound so that there in no change in mass of the material [6]. For example, the compounds of Fe_2O_3 and CaO have melting point at 1,535 ^{O}C and 845 ^{O}C, and similarly, the others are also have high melting point to give significant influence on flame inhibitor [15].

Table 1: Chemical compound of Sokka clay particle [10]

Compound	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	TiO_2	P_2O_5	SO_3	Cl
Content,%	54.59	9.62	3.30	3.55	3.03	2.25	1.40	0.69	0.37	0.35

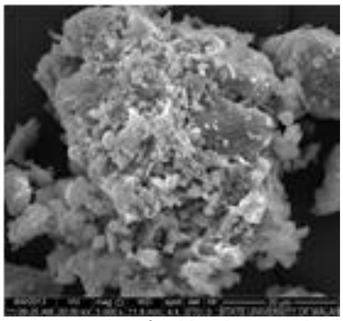
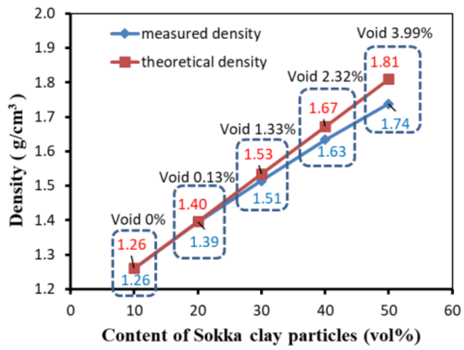


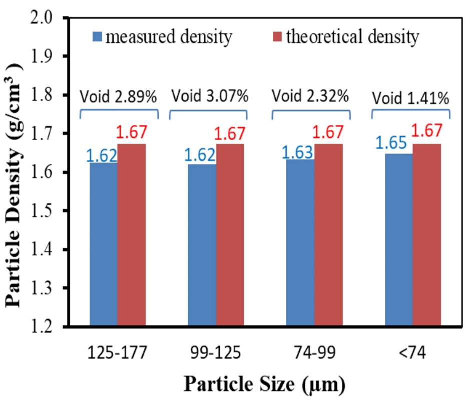
Fig. 2: SEM of Sokka clay particle

SEM of Sokka clay particles shows amorphous particles and its diameter is around 5 µm (Figure 2). The particle tends to agglomerate or adhere to the bigger agglomeration of particle. This particle can absorb water easily due to its agglomeration property [14]. The shape of particle is still classified as filler which can be activated to become active filler in further research.

3.2. Density and Void Content of Composite



(a)



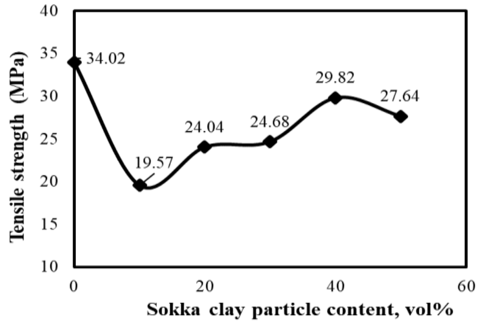
(b)

Fig. 3: Density of composite (a) varied in particles content for particles size of 74-99 µm and (b) varied in particles size for particles content of 40 vol%.

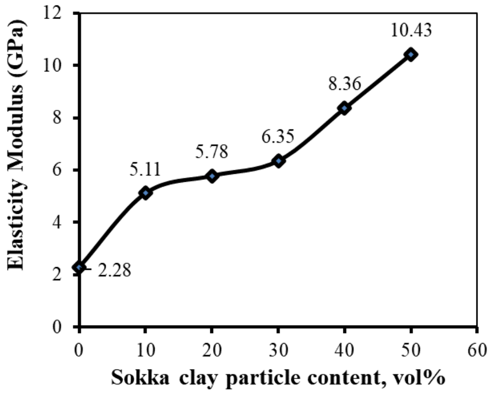
The composites have an increase of density proportionally with an increase of particles content. It may be caused by higher density of Sokka clay particles (2,5 g/cm³) compared with phenolic resin density (1,12 g/cm³). Based on rule of mixture (ROM) theory, the higher content of higher density material produces higher density of mixed materials (composites), as shown in Fig. 3a. The measured density has lower value compared to the theoretical density for 20-50 vol% of particles content (Figure 3a) and the voids increase along with increasing of particles content. The voids content of composites with 10, 20, 30, 40 and 50 vol% of particles are 0, 0.13, 1.33, 2.32 and 3.99 vol%. The rising of voids can be influenced by the reducing of phenolic which should bond all particles. As result, the layers of phenolic among particles in composite become thicker or are limited, and then the voids are formed [5].

In Figure 3b, the smaller particle size produces higher density of composite and its voids are reduced. Those are influenced by the capability of smaller particles to fill empty space among the particles. As result, the composite with smaller particles has higher mechanical properties [16, 17].

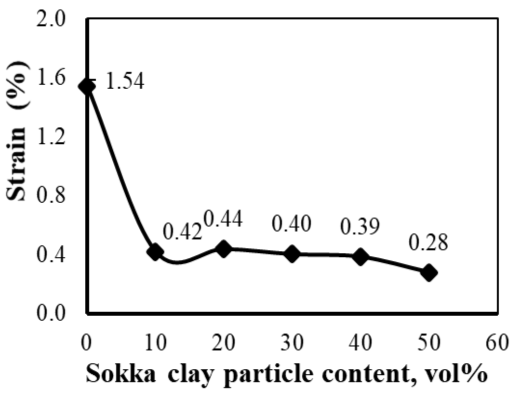
3.3. Tensile Properties



(a) Tensile strength

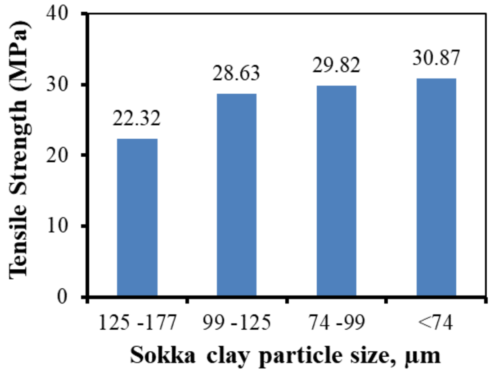


(b) Elasticity modulus

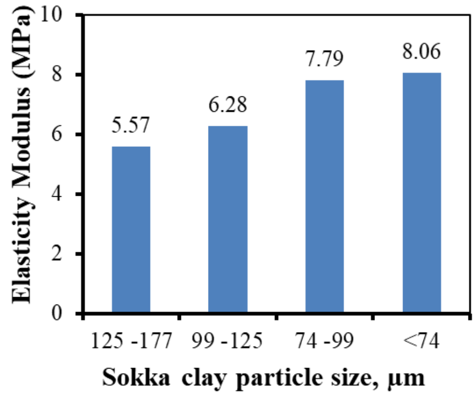


(c) Strain

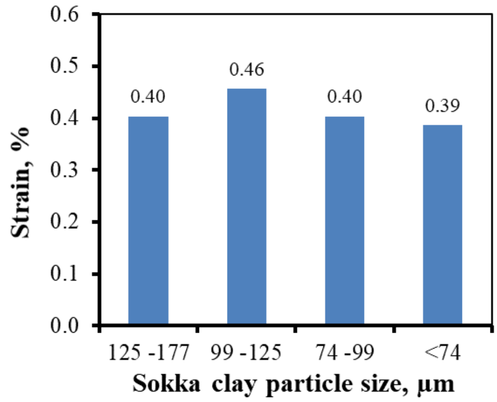
Fig. 4: Tensile properties of Sokka-clay/phenolic composite varied in particle content



(a) Tensile strength



(b) Elasticity modulus



(c) Strain

Fig. 5: Tensile properties of Sokka-clay/phenolic composite varied in particles size

Generally, the addition of Sokka-clay particles in phenolic based composite decreases its tensile strength, as shown in Figure 4a. The strength of composite gradually declines from 34.02 to 19.57 MPa for from 0 to 10 vol% of particles content. It may be caused by the presence of particles serves just as filler or as inclusion filler. Then, its strength enhances from 10% of particles content and achieves peak strength (29.82 MPa) at 40 vol% of particles content. In this period, the particles function serves as reinforcement filler. Although void rises at 10-40 vol% of particles content, but its strength increases consistently due to the sufficiency of phenolic bonds particles and it can be more improved by clay modifying become active clay. Furthermore, its strength decreases at 40 vol% of particles content. The limitation of phenolic to bond all particles or intercalation or exfoliation of particle producing voids is the cause of the decline [18, 19]. Besides that, the addition of too much particles may form particle aggregation and it reduces adhesion between phenolic and particles (Figure 6c). Only a certain amount particles can increase the composite strength [17].

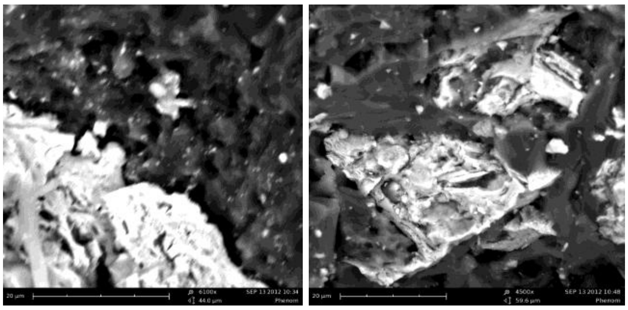
This case may be eliminated by improvement on process modification of clay and curing agent [18].

A decline of strain also occurs consistently as shown on Fig. 4c, and contrary, its elasticity modulus increases (Figure 4b). The increase of particles content, which has properties of harder, higher strength, higher density and higher modulus, restricts the movement of polymer molecules [19]. Theoretically, strain of composite depends on the substance with lower strain, especially clay particles in this research. It makes the reduction of strain and changes its properties become more brittle, stiffer and harder. As consequence, the significant reduction of strain which was determined by particles causes the increasing of elasticity modulus (Figure 4b). Based on this case, an increase of modulus is influenced by an increase of tensile strength and a decrease of strain.

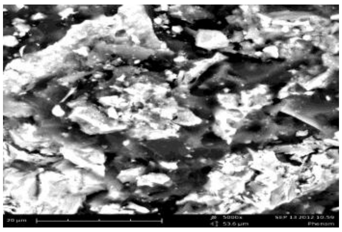
Effect of particles size shows that the smaller particles size, the higher tensile strength and elasticity modulus of composites (Figure 5a and Figure 5b). It may be caused by the easier movement of smaller particles to fill the empty space which can reduce of deffect and make better homogenuous of composites [20]. Beside that, the smaller particles with larger contact surface are also effective to increase its bonding with matrix. On other hand, the difference of particles size in this research does not give a significant influence on the strain (Figure 5c) due to the strain was determined by the lowest strain substance (particles). According to Hooke Law, its modulus increases proportionally with its strength. However, the addition of particle gives a significant decrease of strain compared to pure phenolic (Figure 4c and Figure 5c).

3.4. Fracture Surface Analysis

SEM images of fracture surfaces of Sokka-clay/Phenolic composites varied in particles content is shown in Figure 6. The composite with 40 vol% of particles has good bonding between the particles and phenolic which is evidenced by strong binding of phenolic covered clay particle, as shown in Figure 6b. The fracture surface of composite with 20% of particles in Figure 6a shows that the particles are not distributed overall on the matrix due to its limitation of particles number. However, the phenolic shows a strong bonding to keep the position of particle. The decline strength of composite with 50 vol% of particles are proved by the aggregation of particles which is no well bonded by phenolic (Figure 6c). This incident is source of discontinuity material producing stress intensity factor while loading. Furthermore, this strength declines significantly.



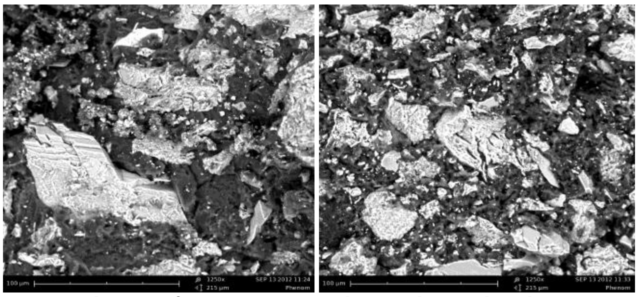
(a) 20 % of particle (b) 40% of particle



(c) 50% of particle

Fig. 6: SEM images of Sokka-clay/phenolic composite varied in particle content

The more homogenous distribution of smaller particle on the composites is proved in Figure 7. Figure 7b shows that the phenolic bonds all particles strongly and evenly on all surfaces. Its surfaces also have similar fracture characteristic due to a wider contact surface of all particles. It is evidenced by the presence all particles bounded strongly by the matrix. However, the fracture surface of composite with larger particles shows non-homogeneity of failure. There are many areas dominated by large particles and by rich matrix (Figure 7a) and its strength depends on the lower strength of the areas. When the large particles undergo aggregation, its strength is determined by the particles, and conversely.



(a) particles size of 125-177 µm (b) particles size less than 74 µm

Fig. 7: SEM images of Sokka-clay/Phenolic Composite varied in particle size

4. Conclusion

Sokka-clay particles, whose stability in mass and good inhibitor of flame, is capable to be used as filler in polymer composite, although it has disadvantage in term of its agglomeration property. The Sokka-clay/phenolic composites have an increase in density along with an increase of particles content and a decrease of particles size. However, there is an increase of voids proportionally with an increase of particle content and particle size. The particles addition of 10 vol% on phenolic resulted in a gradual decrease in tensile strength from 34.02 to 19.57 MPa. Further addition of particle content until 40 vol% enhances its strength and modulus, and similarly with the addition of smaller particles size. Otherwise, addition of particles causes a decrease of strain due to the lower strain of particles. The increase of tensile strength is influenced by the amount of particles, particles size, a possibility of particles to fill the empty space, effective width of contact surface of particles, and elimination of particles aggregation. The Sokka-clay/phenolic composite properties has good potential to be improved with modified Sokka-clay particles. Many improvements of the composite properties is also needed to fulfill its application requirements.

Acknowledgement

The authors would thank to the Ministry of Research, Technology and Higher Education, Republic of Indonesia, especially the Directorate General of Research and Development Strengthening for research financial support by scheme of *INSINAS* 2017-2018.

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