Characteristics of Commercial SiC and Synthetic SiC as an Aggregate in Geopolymer Composites

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Abstract. This main objective of this study is to investigate the effect silicon carbide (SiC) as an aggregate on the mechanical strength and microstructure of the geopolymer composites. The geopolymers binder were produced by using alkaline activation method of metakaolin and cured at 70°C for 2 hours. In this study commercial and synthetic SiC were used as aggregate to produce composite structure. Synthetic SiC was produced from rice husk ash and coconut shell carbon calcined at 750°C for 2 hours. The addition of SiC in geopolymers paste was varied from 0.25g, 0.50g to 0.75g to form geopolymers composites. The chemical compositions and crystallinity level of SiC and the resulting composites were measured by means of Rigaku MiniFlexII X-Ray Diffraction (XRD). The microstructure of SiC and the composites were examined by using Tescan Vega3SB Scanning Electron Microscopy (SEM). The physical and mechanical properties of the samples were determined based on apparent porosity, bulk density, and three bending flexural strength measurements. The results showed that the commercial and synthetic SiC were effectively produced geopolymers composites with different microstructure, physical and mechanical strength.

1. Introduction
Geopolymers is a class of aluminosilicate material activated by using alkaline solution at low temperature [1]. Numerous research have been conducted on this promising material for a wide range applications. Various raw materials were used to produce geopolymers such as metakaolin, red clay (laterite soils), fly ash and furnace slags [2]. The unique properties of geopolymers has attracted researchers to investigate the addition of various type of aggregate to enhance the strength and flexibility of geopolymers. Metakaolin has been considered as a standard raw material to study the basic properties of geopolymers including the addition of new aggregate. In this research, geopolymers were produced from metakaolin and added with SiC particulate as aggregate. Mineral SiC has been known as a stable and fire resistance material with a lot of applications such as in medical sector, semiconductors technology, space industry, and house utensils. Basically, SiC is used as aggregate in the form of fiber to enhance the flexural strength of the composite, including geopolymers composites. A research to investigate the ability of SiC particulate as a forming agent of porosity in the bulk of geopolymers was reported in reference [3]. This research was conducted to study the influence of commercial and synthetic SiC as aggregate on the mechanical and microstructure of geopolymers-SiC (geo-SiC)
composites. Synthetic SiC was produced from a mixture of rice husk ash (RHA) and coconut shell carbon calcined at 750°C for 2 hours.

2. Method

Geo-SiC composites were synthesized by using alkali activation method of metakaolin and cured at 70°C for 2 hours. Geo-SiC composites were produced into 6 different samples by varying the mass of SiC relative to the mass of metakaolin. Commercial SiC used in this research was taken from Rigaku MiniFlexII XRD standard materials and synthetic SiC particle was produced by mixing purified RHA and coconut shell carbon (C) and calcined at 750°C for 2 hours [4]. Geopolymer composites were produced by mixing geopolymers paste with commercial or synthetic SiC particles. The mass of SiC were varying from 0.25g, 0.50g and 0.75g keeping the mass of metakaolin constant. The mixture was poured into sealed mould and cured at 70°C for 2 hours. The samples were stored at open air for 7 days before conducting any measurements. The microstructure characters of the resulting composites were examined by means of Scanning Electron Microscopy (SEM) coupled with Energy Dispersive Spectroscopy (EDS) for microanalysis. X-ray diffraction was used to examine the phase of the starting as well as the resulting materials. The physical characteristics of the samples were examined by means of bulk density and apparent porosity measurements, and thermal conductivity. The mechanical properties of the composite were measured by using three bending flexural technique using the following equation [5],

$$\sigma = \frac{3F}{2bd^2}L$$

Where F = maximum load (N), L = the length of span (support) (mm), b = width of the sample (mm) and d = sample thickness (mm).

3. Results and Discussion

In this research, there were 6 samples of Geo-SiC produced with different compositions. There were 3 samples synthesized contained commercial SiC, and 3 samples contained synthesized SiC. The synthesis of SiC was performed by adopting the following A.J Acheson [6,7] with the ratio of RHA:C = 1:3,

$$\text{SiO}_2(s) + 3C(s) \rightarrow \text{SiC (s)} + 2\text{CO (g)}$$

Figure 1 show the representative of Geo-SiC composites produced in this study. It can be seen that the surface of the samples comprising substantial apparent closed porosity or air bubbles and increasing with the increase of SiC content.

![Figure 1](image_url)

**Figure 1.** Geopolymer sample with commercial SiC (sample A, B, and C) and synthetic SiC (sample D, E, and F) with a concentration of 0.25 g; 0.50 g; 0.75 g, respectively.
The presence of air bubbles can be seen clearly on the surface of sample C which contain 0.75 g commercial SiC. This result is in good agreement with those reported by Skvara, F. et.al [3]. The presence of air bubbles in geopolymers surface or bulk is due to the air trap during the polycondensation process in which the interior of the sample release water reaction (residue) to the surface of the material [9]. Figure 2 shows the SEM images of Geo-SiC surfaces with different type and concentration of SiC. The surface morphology of the samples consists of geopolymers matrix, SiC particles, pores, unfully reacted metakaolin, and cracks.

Figure 2. SEM images of Geo-SiC with commercial SiC (A, B, and C) and synthetic SiC (D, E, and F) with a composition of 0.25 g; 0.50 g; 0.75 g, respectively.

The SEM micrographs shows that the quantity and the size of porosity is larger on the sample contain synthetic SiC. It can also be seen that synthetic SiC particles and unreacted metakaolin dominates the surface of the sample creating inhomogeneous morphology. The particle size of commercial SiC is also smaller than that of synthetic SiC and it may be one of the reason why the surface of composite with commercial SiC is more homogenous. The bulk density and apparent porosity of the samples were measured by using Archimedes method. Table 1 shows that the density of composite decrease with the increase of commercial SiC content, while remain fairly the same for synthetic SiC.

| SiC Content (g) | Bulk Density (g/cm³) | Porosity (%) |
|----------------|----------------------|--------------|
|                | Synthetic SiC | Commercial SiC | Synthetic SiC | Commercial SiC |
| 0.25           | 1.68             | 1.72          | 25.9          | 36.4          |
| 0.50           | 1.68             | 1.58          | 29.1          | 39.6          |
| 0.75           | 1.71             | 1.51          | 22.9          | 38.4          |
The apparent porosity of composite contain commercial SiC are higher than that of synthetic SiC. Although the surface morphology of composite with synthetic SiC appear consist of more pores, the physical measurement revealed different result. It is well known that area examined in SEM is very small compare to physical measurement for bulk density and apparent porosity determination. This result indicate that the commercial SiC worked well as pores forming agent [8].

The thermal conductivity of the composite was measured by using a steady state method to examine the influence of the addition of commercial and synthetic SiC on the thermal properties of composites. Table 2 shows that the magnitude of thermal conductivity of the samples contained commercial and synthetic SiC are the same within the error of measurement. The magnitude of thermal conductivity of the composite Geo-SiC observed in this research is higher than the thermal conductivity of geopolymers paste or geopolymers with quartz or granite aggregate [9]. This indicates that SiC increase the thermal conductivity of geopolymers which is suitable for certain application.

Table 2. The thermal conductivity of composite contained commercial and synthetic SiC.

| SiC Content (g) | Thermal Conductivity (W/mK) |
|-----------------|-----------------------------|
|                 | Synthetic SiC | Commercial SiC |
| 0.25            | 0.77          | 0.79           |
| 0.50            | 0.73          | 0.73           |
| 0.75            | 0.71          | 0.71           |

Figure 3(a) an (b) shows diffractogram of as-prepared geo-SiC for synthetic and commercial SiC, respectively. The picture shows that the structure of geopolymers is amorphous with a hump peak between 20° – 30° 2θ. The addition of SiC did not change the structure of geopolymers. The presence of SiC start to be visible at the addition of 0.75 g commercial SiC (figure 3(b)). This result indicates that geopolymers paste and SiC form a good mechanical bond as a composite.

Figure 3. Diffractograms of Geo-SiC (a) synthetic, (b) commercial
Geopolymers has been known as a heat and fire resistance material which can withstand temperature up to 1200°C[10]. In order to investigate the influence of SiC addition to geopolymers paste, the resulting composites were calcined at 750°C for 2 hours. Figure 4(a) and (b) show the diffractogram of calcined composite for synthetic and commercial SiC.

Figure 4. Diffractograms of calcined Geo-SiC at 750°C, (a) synthetic SiC, (b) commercial SiC.

It can be seen that calcination of the samples at 750°C caused the peaks of amorphous hump shifted into a small 20 indicating restructure of geopolymers networks although the phase of geopolymers remains the same [7]. The presence of SiC in the network od geopolymers paste become more discernible at the addition of 0.75 g commercial SiC (Figure 4(b)).

The mechanical properties of Geo-SiC was examined by means of three bending flexural measurement. Table 3 show the flexural strength of Geo-SiC for synthetic and commercial SiC.

| NO | Load (F_N) | Flexural Strength (MPa) |
|----|------------|-------------------------|
|    |            | Synthetic SiC | Commercial SiC |
| 1  | 2.5        | 8.87         | 13.89         |
| 2  | 7.5        | 10.20        | 6.22          |

Table 3 shows that the magnitude of flexural strength for both type SiC additions are higher than the magnitude of flexural strength of pure geopolymers made from metakaolin[10]. The flexural strength of Geo-SiC using synthetic SiC increase with the increase of SiC concentration. In contrast, the increase of commercial SiC reduce the magnitude of flexural strength of SiC. The difference may be attributed to the higher porosity of Geo-SiC due to the addition of commercial SiC as tabulated in table 1.

4. Conclusions
Geopolymers composites made from metakaolin added with SiC (commercial and synthetic) activated through alkali activation and cured at 700°C for 2 hours have been produced and characterized. SiC particles used as aggregate were consisted of commercial and synthetic SiC. Synthetic SiC was produced from rice husk ash and coconut black carbon calcined at 750°C for 2 hours. The bulk density and porosity of composites increase with the increase of SiC concentration. The volume of pores on the surface of the composites is higher in composites produced from commercial SiC. The particle size of commercial SiC is smaller than that of synthetic SiC as measured by using SEM. The XRD examination of the composites showed that the addition of SiC did not change the amorphous structure of geopolymers. The addition of commercial and synthetic SiC particles were found to improve the magnitude of flexural strength of composites.
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