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INFLUENCE OF SINTERING TEMPERATURE ON THE PORE STRUCTURE OF AN ALKALI-ACTIVATED KAOLIN BASED GEOPOLYMER

Kaolin-based geopolymers are alternatives for producing high-strength ceramics for construction materials. Creating high-performing kaolin ceramics utilizing the regular technique requires a high handling temperature (higher than 1200°C). Thus, the structure and properties such as pore size and distribution are affected at higher sintering temperatures. Along these lines, information with respect to the sintering system and related pore structure is essential for advancing the properties of the previously mentioned materials. This study investigated the microstructure and the density of a kaolin-based geopolymer at various sintering temperatures. The unsintered sample has the highest density of 1610 kg/cm^3, while the samples sintered at 1100°C haves the lowest density of 1203 kg/cm^3. The result also shows that increasing the sintering temperature to 1100°C resulted in increasing the water absorption of the kaolin-based geopolymer ceramic.

Keyword: Geopolymer; sintering; pore; density; water absorption

1. Introduction

Geopolymer is an amorphous three-dimensional (3D) aluminosilicate activation of suitable precursor raw material [1]. Geopolymers are amorphous to semi-crystalline three-dimensional silico-aluminate materials synthesized at low temperatures in a short time. However, with the increasing temperature crystalline phase begins to appear. Kaolin is an inorganic material that has been identified as geopolymer compatible with excellent performance. As a result of excellent properties, geopolymers have been investigated for potential application in refractory and insulating areas industries. Wang et al. [2] reported that the structure of kaolin was majorly influenced by the sintering temperature. The change of aluminium species affected the structural changes of geopolymer after heating at 900°C.

In order to use the geopolymer in commercial application, it is important to understand the sintering process and the pore structure of kaolin geopolymer. Sintering can be defined as the thermally-activated adhesion processes, which increase the contact between the particles and their respective coalescence [3]. Sintering closes some of the open pores, decreasing the water absorption rate and increasing its strength. The porosity of geopolymer could change because of the evaporated of water during sintering process. The dense heated geopolymer has a glassy phase, basically making it a ceramic. Traditionally, the ceramic vitrification commonly initiates from 900°C which marked by the melting of several solid phase that binds the present solid particles and lead to enhance the bonding strength [4,5]. Geopolymers are known to have inflammability at high temperature during sintering. Thus, their behaviour at elevated temperature exposure is becoming an interest in the means of mechanical properties. The mechanical properties of geopolymers are said to improve greatly when exposed to elevated temperatures.

In this study, the SEM microstructure has been used to investigate the effect of sintering on the amount of pore of kaolin-based geopolymer. The porosities and water absorption after the sintering process also has been investigated to study the effect of sintering on the pore structure. This work successfully characterised and investigated the pore structure of kaolin-based geopolymer.

2. Experiment Details

The kaolin utilized in this work was provided by the precursor sources of kaolin (Associated Kaolin Industries Sdn.

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Bhd, Malaysia). The sodium silicate (Na_2SiO_3) solution was supplied by South Pacific Chemical Industries Sdn. Bhd., Malaysia. The kaolin consists of SiO_2 (30.1%), Na_2O (9.4%), and H_2O (60.5%), with SiO_2/Na_2O = 3.2. The NaOH clear solution was mixed with sodium silicate solution and cooled to ambient temperature a day before mixing. The kaolin materials were mixed with an alkaline activator solution for 5 mins; then, the homogenised mixture was poured into a mould. After curing for 14 days, the kaolin-based geopolymer was sintered at 900°C and 1100°C and soaked for 2 hours at a heating rate of 10°C/min in an electrically-heated furnace.

The unsintered and sintered examples were imaged utilizing the JSM-6460LA Scanning Electron Microscope (JEOL, Peabody, MA USA), outfitted with auxiliary electron identifiers. The voltage and working distance were fixed at 10 kV and 10 mm, separately. The surface region and pore volume were estimated utilizing Brunauer-Emmet-Teller (BET) gear (TrisStar 3000, Micrometrics Instrument Corporation, GA, USA). The adsorbed amount is connected to the particles' absolute surface regions and pore volume in the unsintered and sintered examples. The density was calculated and water absorption tests were conducted per ASTM C642-13 [ASTM C642-13, Standard Test Method for Density, Absorption, and Voids in Hardened Concrete, ASTM International, United States (2013)]. The weight of the sample after and before the sample was immersed in water was recorded, and the percentages of water absorption for the samples after sintering at 900°C and 1100°C were determined.

3. Results and Discussion

3.1. Microstructure analysis

The microstructure of kaolin geopolymer unsintered and sintered was investigated by SEM as shown in Fig. 1(a) unsintered, (b) 900°C, and (c) 1100°C.The microstructure reveal that unsintered kaolin shows the presence of well-defined clay platelets and an incomplete reaction of kaolin, per Fig. 1(a). After sintering at 900°C and 1100°C, the images clearly show the presence of pores and cracks in all of the heated kaolin-based geopolymer ceramic. The kaolin-based geopolymer surface became increasing glassy and glossy when sintered at 900°C (Fig. 1b). The microstructure change can be attributed to the hydration of moisture and phase transformation, as reported by Dudek et al. [6]. It can also be seen in Fig. 1(c) that the kaolin-based geopolymer ceramic sintered at 1100°C have a higher porosity, alongside cracks and voids.

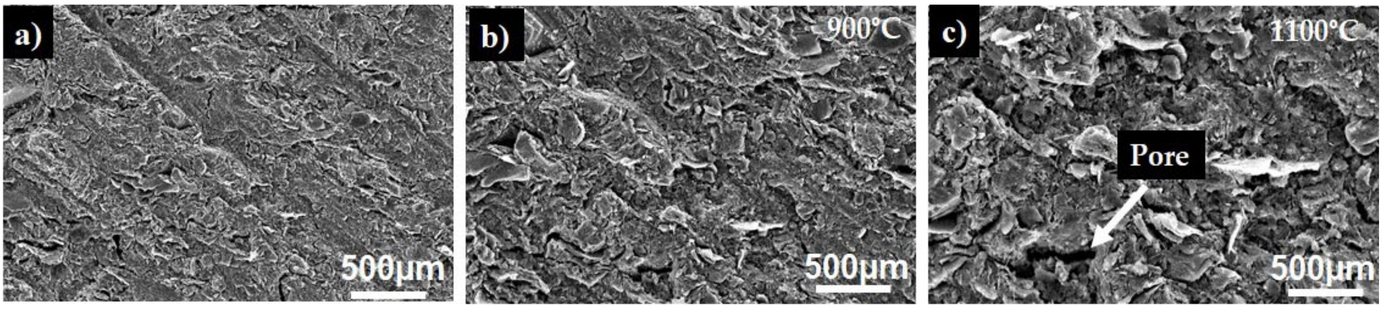

Fig. 1. SEM micrograph of (a) unsintered, (b) sintered at 900°C and (c) sintered at 1100°C

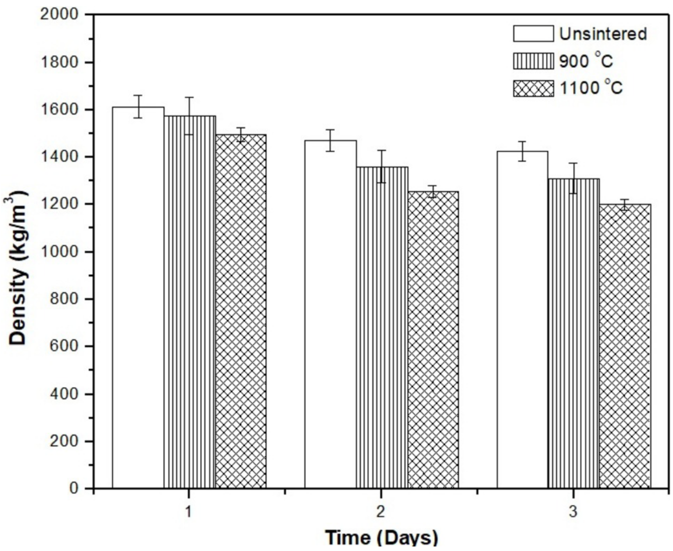

Fig. 2. The density of kaolin geopolymer for unsintered and sintered at 900 and 1100°C for 3 days

3.2. Density and Water Absorption Analysis

The density and water absorption of the kaolin-based geopolymer sample have been investigated to examine the pore structure in kaolin based geopolymer. The densities of the unsintered and sintered kaolin at 900°C and 1100°C for 3 days are shown in Fig. 2. The densitiesy of the unsintered and sintered samples at all temperatures decreased with increasing time. The unsintered sample has the highest density of 1610 kg/cm^3, while the samples sintered at 1100°C haves the lowest density of 1203 kg/cm^3.

Therefore, it is theorized that the formation of large pores created kaolin at 1100°C resulted in the lowest density, while sample sintered at 900°C resulted in the formation of small pores in the kaolin-based geopolymer sample. Likewise, the unsintered sample contained small and open pores, while the sintered sample had large and closed pores, which translated into high material density [7].

Fig. 3 shows the percentage water absorption of kaolin-based geopolymer ceramic after sintering at 900°C and 1100°C for 3 days. From the graph, it was found that the highest value percentage of water absorption was found after 3 days sintering at 1100°C. Basically, the trend was continuously increased with increasing sintering temperature and time. It is believed that the higher sintering temperature exposed to the kaolin geopolymer, resulting larger pores size due to the water removal. Hence, increased the pore size can affect to the increasing the capacity of the water been absorbed to the geopolymer sample. The sintering temperature could affect the pore size and density of geopolymer ceramic and the mechanical testing is needed to further investigation regarding this finding.

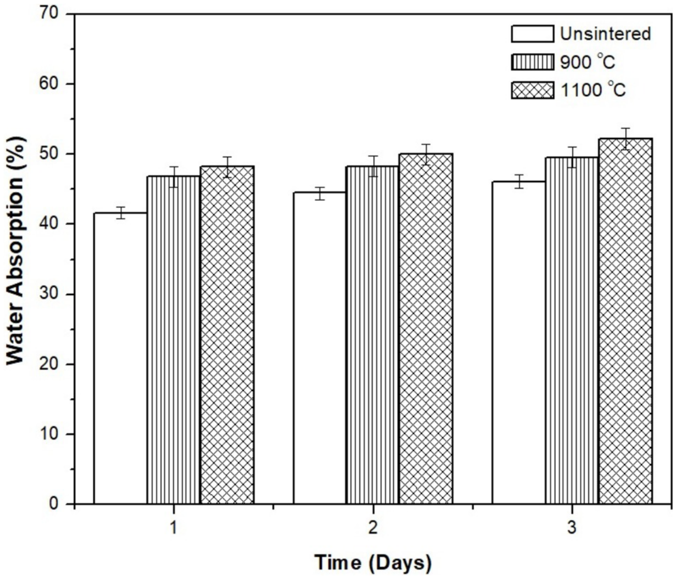

Fig. 3. Average percentage of water absorption kaolin geopolymer sintered up to 1100°C after 3 days

4. Conclusions

The pore properties in kaolin geopolymer ceramic has been investigate and were analysed in this paper. Several conclusions has been made as below:

1. The effect of sintering temperature to the pore were investigated as the microstructure analysis show the development of surface densification and less pore within the geopolymer matrix.
2. The density and water absorption confirmed the presence of pores after the sintering process. The unsintered sample has the highest density of 1610 kg/cm^3, while the samples sintered at 1100°C haves the lowest density of 1203 kg/cm^3.
3. The microstructural analyses showed that sintering at 1100°C resulted in large pore sizes relative to its unsintered sample.

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