Innovative Materials and Technologies

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Innovative Materials and Technologies

Edited by Mohd Mustafa Al Bakri Abdullah Zarina Yahya Mohd Arif Anuar Mohd Salleh

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Edited by

Mohd Mustafa Al Bakri Abdullah, Zarina Yahya and Mohd Arif Anuar Mohd Salleh



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Characterization and Microstructure of Kaolin-based Ceramic using Geopolymerization

Nur Ain Jaya^{1, a},Mohd Mustafa Al Bakri Abdullah^{1,2,b}, Che Mohd Ruzaidi Ghazali^{1,2,c}, Mohammed Binhussain^{3,d}, Kamarudin Hussin^{1,2,e}, Romisuhani Ahmad^{1,f}

¹Center of Excellence Geopolymer and Green Technology, School of Materials Engineering, Universiti Malaysia Perlis (UniMAP), 01007, P.O Box 77, D/A Pejabat Pos Besar, Kangar, Perlis, Malaysia.

²Faculty of Engineering Technology, Universiti Malaysia Perlis (UniMAP), 01007, P.O Box 77, D/A Pejabat Pos Besar, Kangar, Perlis, Malaysia.

³King Abdul Aziz City Science & Technology (KACST), P.O. Box 6086, Riyadh 11442, Kingdom of Saudi Arabia.

^aainjaya@gmail.com, ^bmustafa_albakri@unimap.edu.my, ^cruzaidi@unimap.edu.my

Keywords: Geopolymer; Ceramic; Sintering; Powder Metallurgy; Microstructure

Abstract. Geopolymers can be transformed into ceramics upon sintering. This paper reports the effect of temperature on the physical, mechanical properties and characteristics of kaolin geopolymer ceramic. The nepheline ceramic was fabricated through geopolymerization. The geopolymer samples were exposed to temperature from 900 °C up to 1200 °C. Kaolin geopolymer undergo shrinkage upon temperature exposure. Unheated kaolin geopolymer appeared to be amorphous and crystalline nepheline was the major phase after sintered to high temperatures as depicted by XRD analysis. Microstructural analysis showed formation of denser structure as the temperature increased. The maximum flexural strength of 86 MPa is achieved at temperatures of 1200 °C.

1. Introduction

Geopolymers are synthetic alkali aluminosilicate material produced through a reaction between aluminosilicate materials with alkali or alkali silicates under highly alkaline conditions [1-3]. The commonly used aluminoslicate materials are fly ash [4, 5], volcanic ash [6] [7], metakaolin [8] and natural minerals [9] containing Si and Al can be used to form geopolymers with superior physical and mechanical properties. Alkaline solution aids in the dissolution process of raw material. The most common alkaline activator used is a mixture of hydroxide (NaOH or KOH) and liquid silicates (Na₂SiO₃ or K₂SiO₃).

Geopolymerization occured by putting the aluminosilicate materials in contact with the alkaline activator solution which gives as a result, the presence of the polymeric chain of Si-O-Al-O bonds. Geopolymer synthesis involves dissolution, hydrolysis and condensation of aluminate and silicate species to form geopolymeric structure [9, 10] which occurs at room temperature. Water was expelled from the reaction during the curing process and extended drying periods. This gives benefits to the performance of geopolymers as the water leaves behind nano-pore structure in the matrix [11]. Hence, geopolymers often regarded as cementitous materials. However, at high temperature crystalline phases are formed and sintering reactions result in the formation of ceramic products [12].

Geopolymerization has emerged as an alternative way to ceramic formation by using clay based material such as kaolin. Huge effort has been done in converting this high strength monolith of Si/Al ≤ 2 into ceramics. Generally, geopolymers are amorphous to semi-crystalline [13] and comprise of cross-linked AlO₄ and SiO₄ tetrahedra, where charge balance is supplied by hydrated alkali metal cations. The amorphous to semi-crystalline behavior of geopolymer will change into

crystalline ceramic phases upon heating [14]. The shaped green body is heated to a temperature that is typically 0.5 - 0.9 % of the melting point. Formation of liquid phase produces joining of the particles and reduction of the porosity [15]. The behavior of geopolymers at high temperature exposure is dependent entirely on the chemical composition of the binder. The type of alkali ion, Si/Al ratio and crystal structure themselves are the important factors included [16-18].

Kaolin is used as the aluminosilicate source in this study. The selection of kaolin offers a few advantages towards the common source materials used in geopolymer such as metakaolin. Dehydroxylation is a reaction of decomposition of kaolinite crystals to a partially disordered structure to produce metakaolin. This reaction requires calcining of kaolin at temperatures between 500 to 900 °C [19, 20]. Calcining kaolin limits the commercial application of metakaolin due to the added cost and consume much energy. Previous work done by He, et al. [21] has been produced metakaolin based geopolymer and obtained flexural strength of 70 MPa. This study has come to be crucial in order to produce kaolin based geopolymers with excellent properties as to metakaolin based geopolymers.

The aim of this work was to prepare powder pressed geopolymer ceramic using kaolin with various sodium hydroxide concentrations and then sintered at 900, 1000, 1100 and 1200 °C. The effects of sintering temperature on the physical, mechanical properties and characteristics of kaolin geopolymer ceramic are studied comprehensively.

2. Experimental method

2.1 Materials

Kaolin used in this study was supplied by Associated Kaolin Industries Malaysia as Si-Al sources materials. The chemical composition of kaolin was determined by X-ray fluorescence shown in Table 1. The particle size distribution of aluminosilicate materials was obtained using a Malvern particle size analyzer as follows: $d_{(0.1)} = 2.836 \mu m$, $d_{(0.5)} = 9.465 \mu m$ and $d_{(0.9)} = 25.697 \mu m$. Sodium hydroxide (NaOH) powder used to be of caustic soda micropearls, 99% purity with brand name of Formosoda-P made in Taiwan. Sodium silicate (Na₂SiO₃) solution was supplied by South Pacific Chemicals Industries Sdn. Bhd. Malaysia with a chemical composition of 30.1% SiO₂, 9.4% Na₂O and 60.5% H₂O.

2.3 Sample Preparation

The ratio of kaolin to alkaline activator used is 1.0 with Na₂SiO₃ /NaOH ratio of 0.24. The details of the mixtures involved were presented in Table 2. The mixture then were mixed well for a few minutes and were cured at 80 °C for 24 h. To obtain fine powder, kaolin geopolymer were crushed using mechanical. In order to produce geopolymer ceramic, the sieved powders were then compacted using a 12-mm-diameter cylindrical stainless steel die at 5 ton for 2 min. The final green body was sintered in a high temperature furnace to 900 °C - 1200 °C with 3 h soaking time and a cooling and heating rate of 5 °C/ min.

Table 1 : Chemical composition of kaolin as determined by X	R	J	Ē
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Chemical	Wt. (%)
SiO ₂	54.0
Al_2O_3	31.7
Fe_2O_3	4.89
TiO ₂	1.41
ZrO_2	0.10
K_2O	6.05
MnO_2	0.11
LOI	1.74

NoOH No SiO /	Kaalin	No SiO	NoOU	Total molar ratio			
malarity	NaOH	Kaulili (g)	132310_3	(α)	Na ₂ O/	SiO ₂ /	$H_2O/$
morarity	NaOII	(g)	(g)	(g) (g)	SiO ₂	Al_2O_3	Na ₂ O
6	0.24	300	58.06	241.94	0.2428	3.2022	18.2024
8	0.24	300	58.06	241.94	0.2951	3.2022	15.1505
10	0.24	300	58.06	241.94	0.3472	3.2022	13.0288
12	0.24	300	58.06	241.94	0.3950	3.2022	11.5730

 Table 2: Mix design details

2.4 Tests and Analysis Method

X-ray fluorescence spectroscopy is used for the determination of bulk elemental composition. XRF spectroscopy of the powders in this study was conducted by a commercial laboratory (Ultra Trace Geoanalytical Laboratories). The samples were fused in a silicate glass disc (method code XRF 202) and analyzed on a Philips PW 2404 x-ray spectrometer with a 4 kW rhodium tube. Results were reported as oxides. Flexural strength measurements of kaolin geopolymer ceramics were conducted on specimens (7 mm x 5 mm x 52 mm) using a three-pointbending fixture on an instron-500 tester, with a span length of 30 mm at a crosshead speed of 0.3 mm/min. A minimum of five samples for each group was subjected to flexural strength testing in accordance with ASTM C1161-02c [22]. The effect of sintering temperature on the microstructure of sintered samples was investigated using scanning electron microscopy (SEM) on polished surfaces. Polishing was conductive materials, samples were coated with platinum as a conductive layer to obtain better quality of SEM images. XRD patterns were collected using XRD 6000, SHIMADZU diffractometer with Cu-K α radiation. The diffraction scans were performed from 10° to 80° 20 at a rate of 2°/min, with a step size of 0.02°.

3. Results and discussion

3.1 Shrinkage

This work studies the effect of sintering on the physical, mechanical properties and characteristics of kaolin geopolymer ceramic. During sintering, geopolymer undergo some transformations which are removal of water and newborn of crystalline phase growth. As shown in Fig. 1, green kaolin geopolymer body shows reduction in size after sintering. This is because during sintering process, the surface energy decreases due to the cohering together of incoherent particles. Thus, reducing the total surface area. Further increment of sintering temperature to 1250 °C caused the samples to melt because it exceeds the melting point of the kaolin geopolymer.





Fig. 1: Effect of sintering on the shrinkage of the kaolin geopolymer ceramic.

3.2 Flexural strength

The flexural strength of kaolin geopolymer ceramic with various NaOH concentrations sintered at 900 °C up to 1200 °C is shown in Fig. 2. Table 2 concludes the mechanical properties of 12M0.24 kaolin geopolymer ceramic at different sintering temperature. The flexural strength increased with increasing sintering temperature and NaOH concentration. The flexural strength at 6 M is expected to be the lowest among the other NaOH concentration due to lower alkalinity. This might contribute to the excess mixing water for a lower NaOH concentration. Hence, this showed that water plays an important part in transporting the ions during geopolymerization process [23].



Fig. 2: Flexural strength of kaolin geopolymer ceramic of 6 M to 12 M with 0.24 Na₂SiO₃/NaOH at elevated temperature.

Sintering temperatures (°C)	Density (g/cm ³)	Flexural strength (MPa)
900	2.67	40.35
1000	2.50	48.46
1100	2.39	50.68
1200	2.13	86.83

Table 2: Mechanical properties of 12M0.24 kaolin geopolymer ceramic at different temperatures

The maximum flexural strength of 86 MPa was achieved at 1200 °C using 12 M kaolin geopolymer. It proved that higher temperature (up to 1200 °C) does help to enhance the mechanical properties. Further sintering at 1250 °C leads to melting of the geopolymer body. Thus, the flexural strength cannot be measured. The increasing strength at elevated temperature might be contributed by the low diffusion coefficient of alkali metal used which result in higher melting temperature of the geopolymer [24]. Geopolymer body contains portions of unreacted particles remained from the dissolution process. Exposure to temperature caused the sintering of these particles which leads to increasing strength by forming stronger bonds and countering the thermal damage at elevated temperatures [25]. Daniel L.Y. Kong [26] has reported that the strength increment in geopolymer might be attributed by the combination of polymerization and sintering. This high temperature was also sufficient to consolidate the sample and for the crystallization to occur which later enhance the mechanical properties [27].

3.3 X-ray diffraction analysis

Generally, kaolin consists of kaolinite as main mineral. From the Figure 3, kaolinite showed strong diffraction peaks at 2 Θ value of 24.9° and less strong peaks at 12.5°, 19.9°, 20.4°, 21.4°, 24.9°, 31.9°, 36.0°, 37.8°, 38.5°, 43.8°, 50.2°, 56.7°, 60.1°, 62.1°, 68.4° and 70.3°. Other than kaolinite, quartz also can be found in kaolin at 2 Θ value of 26.7°, 38.5°, 45.6° and 51.2°. Illite peaks were also present at 2 Θ value of 17.89° and 70.29°. Dickite phase present at 2 Θ value of 23.17°, 28.59°, 36.04°, 39.37° and 56.75°. The presence of only kaolinite and quartz suggested that kaolin used in this study is a pure phase as it is predominantly kaolinite.

The transformation of amorphous state of kaolin geopolymer to crystalline phase upon heating were determined by XRD is shown in Fig. 4. A broad peak of the amorphous range from 10° to 40 ° can be depicted from the unheated kaolin geopolymer pattern. Formation of zeolites in unheated geopolymer originated from the hydrothermal setting conditions [3] whereas the kaolinite and quartz peak were remained from the original kaolin (Fig. 3). The broad hump of amorphous phase disappeared when geopolymer undergoes heat treatment as sharp diffraction peaks appeared. On heating to higher temperatures (900 – 1200 °C), the zeolites peak were reestablished and rearranged into nepheline structure [28]. All of the samples heated to 900 – 1200 °C shows the appearance of the same nepheline peak but with increasing intensity. The increasing intensity of nepheline resulted in the increased flexural strength of the sample. Crystalline nepheline is typical to occur in sodium aluminosilicate for glass- ceramics [29]. The crystalline product formed is to show a relationship with the sodium-based activator used to produce the sample in this study.



Fig. 4: XRD diffractogram of unheated and geopolymer sintered at 1200 °C

3.4 Scanning electron microscopy analysis

Sintering in ceramics is known to have a direct relationship with microstructure. SEM micrographs for unheated and heated kaolin geopolymer were shown in Fig. 5. Unheated kaolin geopolymer has plate-like structure which originates from the kaolin used in this study. The temperature exposure causes the appearance of pores and voids (Figure 6) in the microstructure due to the binding of the particles. The appearance of small pores throughout the sample denotes the transformation of amorphous to nepheline crystalline ceramic [12]. After treating to higher temperature, the structure experienced advanced growth where the grains of clay minerals coalesce and the joining of particles [30]. The pore size increase significantly due to the fact that high temperatures transformed the liquid water present in the material to water vapour which tended to escape, generating pressure on the pore walls and growing inter-pore connections to the loss of smaller pores. The formation of voids which lowered the density is due to the evolution of gas originates from the chemical composition and structural water of the geopolymer [31].



Fig. 5: SEM micrographs of kaolin geopolymer and kaolin geoplymer ceramic sintered at 1200 °C



Fig. 6: SEM micrographs showing voids in geopolymer as temperature increases

4. Conclusion

The effects of sintering temperature on the physical, mechanical properties and characterization of kaolin geopolymer ceramic are studied comprehensively. This study concludes that the flexural strength of 6 to 12 M NaOH concentration increases with sintering temperature. The result reveals that the maximum flexural strength of 86 MPa is achieved at 1200 °C. Upon heating to high temperatures, amorphous geopolymer transform to crystalline nepheline ceramic. Microstructural analysis of samples with the highest strength depicted that higher temperature aids in increasing the consolidation and facilitating a fairly uniform microstructure. This study showed that geopolymer can be used as a precursor to ceramic forming.

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