

Properties and Microstructural Characteristic of Kaolin Geopolymer Ceramics with Addition of Ultra High Molecular Weight Polyethylene

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Abstract. In this paper, the mechanical properties and microstructure of kaolin geopolymer ceramics with addition of Ultra High Molecular Weight Polyethylene were studied. Inorganic polymers based on alumina and silica polysialate units were synthesized at room temperature from kaolin and sodium silicate in a highly alkaline medium, followed by curing and drying at 80 °C. Alkaline activator was formed by mixing the 12 M NaOH solution with sodium silicate at a ratio of 0.24. Addition of Ultra High Molecular Weight Polyethylene to the kaolin geopolymer are fabricated with Ultra High Molecular Weight Polyethylene content of 2, 4, 6 and 8 (wt. %) by using powder metallurgy method. The samples were heated at 1200 °C and the strength and morphological were tested. It was found that the flexural strength for the kaolin geopolymer ceramics with addition of UHMWPE were improved and generally increased with the increasing of UHMWPE loading. The result revealed that the optimum flexural strength was obtained at UHMWPE loading of 4 wt. % (92.1 MPa) and the flexural strength started to decrease. Microstructural analysis showed the samples appeared to have more number of pores and connected of pores increased with the increasing of UHMWPE content.

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1. Introduction

Since the concept of geopolymer was introduced by Joseph Davidovits in 1978, geopolymer have become familiar and prevalent materials all over the world due to their specific performance of high temperature resistance, high compressive strength, low density, low permeability and so on [1]. With the many advantages offered, geopolymers are considered as fine engineering materials with a broad application [2 – 5]. It can be used as fire and heat resistant fibre composites, sealants, concretes, ceramics, etc., depending on the chemical composition of the source material and the activators. It also can be used as waste encapsulation to immobilize toxic metals. Geopolymer are normally made by mixing materials that contain aluminosilicate, such as kaolin, metakaolin and fly ash, into alkaline solution and curing the mixture at a certain temperature [6 – 7]. Theoretically, any alkali and alkali earth cation can be used as an alkali element in this exothermic process, geopolymerization. Besides, any material composed of alumina and silica can be alkali-activated. In this study, kaolin was used as aluminosilicate material to prepare geopolymer.

The strength of geopolymers influenced by the nature of the source materials. Geopolymers that are synthesized using calcined source materials, e.g., fly ash, slag, and metakaolin, have higher compressive strength than those synthesized using non-calcined materials, such as kaolinite, and naturally-occurring minerals [8 – 10]. Several studies have focused on the use of metakaolin [11 – 13] because of its higher reactivity, which contributes to optimal performance, slightly than the less reactive of kaolin [14]. Besides, the use of metakaolin instead of other materials has been found to produce more homogeneous geopolymers [15]. Thus, there is a lack of study on green polymeric composites by using kaolin. In the development of geopolymer ceramic, kaolin is a potential material to be used since it can be calcined at high temperature [7].

The amorphous to semi-crystalline behaviour of geopolymer will change into crystalline ceramic phases upon heating [11, 16, 17]. Therefore, geopolymer technology offers a new route to produce advance ceramics of profitably low cost and near net-shape forming. As a result of their excellent physical and mechanical properties, geopolymer ceramic composites are expected to emerge as potentially stronger and tougher materials that can meet the demanding requirements of material applications. Jonathan et al. 2009 [11] also proved that it was possible to produce pollucite ceramic from the natural metakaolin-based geopolymer in the formation of pollucite ($\text{Cs}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 4\text{SiO}_2$). A geopolymer that consist of a mixture of metakaolin and cesium silicate solution were prepared using an IKA overhead mixer. Then the resultant slurry was cured at 50 °C for 24 h. The pollucite crystallization occurred gradually during heating at temperatures in the range of 900 - 1250 °C. While He et al. 2010 [12] studied the influence of temperature on microstructure and mechanical properties of carbon fibre reinforced geopolymer composites that heat treated those materials between 1000 °C and 1400 °C. They found that the mechanical properties of these composites can be greatly improved by heat treated at 1400 °C and tend to fracture in a very brittle manner owing to the seriously degraded carbon fibres together with the matrix melting and crystal phases dissolve.

A new approach of addition of polymer (UHMWPE) into geopolymer ceramic materials prepared by using powder metallurgy method and sintered at 1200 °C. Since the melting point of UHMWPE is low and expected to decompose when heated at high temperature, the effect of various UHMWPE loadings on flexural strength and microstructure were studied.

2. Experimental

2.1 Materials

Kaolin used in this study was supplied by Associated Kaolin Industries Malaysia as Si-Al sources materials. Table 1 summarised the chemical composition of kaolin analysed by X-ray fluorescence (XRF). The particle size distribution of aluminosilicate materials was obtained using Malvern particle size analyser. Results showed that kaolin consists of very fine particles with the most abundant particle size in the range of 90 - 120 μ .

Sodium hydroxide (NaOH) powder used was of caustic soda micropearls, 99% purity with brand name of Formosoda-P made in Taiwan. Sodium silicate (Na_2SiO_3) chemical composition are 30.1% of

SiO₂, 9.4% of Na₂O and 60.5% of H₂O (modulus, SiO₂/Na₂O = 3.2), specific gravity at 20 °C = 1.4 g/cm³ and viscosity at 20 °C = 0.4 Pa s.

The powdered formed UHMWPE, with a molecular weight of 5 X 10⁶ g/mol and a density of 0.94 g/ml was used as a binder in kaolin geopolymer ceramics and purchased from Ticona Engineering Polymer, China.

Table 1: Chemical composition of kaolin.

Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	TiO ₂	MnO ₂	ZrO ₂	LOI
Percentage (%)	54.0	31.7	4.89	6.05	1.41	0.11	0.10	1.74

2.2 Samples preparation

Alkaline activator was formed by mixing the 12M of NaOH solution with Na₂SiO₃ at a ratio of 0.24. The solution was then allowed to mature for 24 hours in order to dissolve the silica completely. The geopolymer paste was prepared by mixing kaolin powder with alkaline activator with the solid-to-liquid ratio of 1.0 and were stirred well for a few minutes by using a mechanical mixer. The details of the mixtures involved were presented in Table 2. This ratio was chosen based on our previous work, as it allowed for optimal mechanical and thermal properties. The samples were then cured at 80 °C for 24 h.

To obtained fine powder, kaolin geopolymer were crushed using mechanical crusher and sieved manually by using 150 μ siever. The kaolin geopolymer powder were then mixed with UHMWPE by using planetary mill in dry condition method. The mixing process was done for 4 minutes at 100 rpm with reverse direction to get the homogeneous mixture. The mixing formulae for UHMWPE and kaolin geopolymer are shown in Table 3.

The mixture was then compacted using a 12-mm-diameter cylindrical stainless steel die at 5 ton for 2 minutes. The final green body was sintered in a high temperature Carbolite furnace 1200 °C with 3 h soaking time and a cooling and heating rate of 5 °C/ min. All samples were sintered on an alumina plate as it can withstand high temperature firing.

Table 2: Mix design details.

S/ L	NaOH molarity	Na ₂ SiO ₃ / NaOH	Total molar ratio		
			Na ₂ O/ SiO ₂	SiO ₂ / Al ₂ O ₃	H ₂ O/ Na ₂ O
1	12	0.24	0.3950	3.2022	11.5730

Table 3: Mixing formulation of UHMWPE/kaolin geopolymer at different content.

Materials	Formulation
Kaolin geopolymer (wt. %)	100
UHMWPE (wt. %)	0, 2, 4, 6, 8

2.3 Testing

2.3.1 Flexural Strength

Flexural strength measurements of clay geopolymer ceramics were conducted on specimens (7 mm x 5 mm x 52 mm) using a three-point-bending fixture on an instron-500 tester, with a span length of 30 mm at a crosshead speed of 0.5 mm/min. A minimum of five samples for each group were subjected to flexural strength testing in accordance with ASTM C1161.

2.3.2 Morphology Analysis

JSM-6460LA model Scanning electron microscope (JEOL) was performed to reveal the microstructure of kaolin geopolymers at S/L of 1.0. The specimens were prepared and coated by using Auto Fine Coater; model JEOL JFC 1600 prior to examination.

3. Results and Discussion

3.1 Flexural Strength

The effect of UHMWPE content on the kaolin geopolymer ceramics heated at 1200 °C is shown in Figure 1. The flexural strength of kaolin geopolymer ceramics has been improved after incorporation of UHMWPE. As compared to the kaolin geopolymer ceramics without the addition of UHMWPE, the flexural strength increased at 2 wt. % of UHMWPE content and started to decrease when the UHMWPE content exceeded 4 wt. %. Kaolin geopolymer ceramics with addition of UHMWPE at 4 wt. % having the highest strength of 90 MPa. This is due to the decomposition of UHMWPE at high temperature attributed to increasing of carbon in the geopolymer system. As reported from previous researchers the fine distributed excess of carbon can be advantageous for the resistance against crystallization up to high temperatures and at the same time generates a complex microstructure for carbon-rich of geopolymer ceramics hence improve in flexural strength [18]. However, the flexural strength decreased at the optimum value were due to the porosity increased to a large extent hence reduced the strength [19]. Xu et al., (2015) observed the similar findings in their research on the effect of dolomite and spodumene for the performances of andalusite composite ceramics. They found the addition of dolomite has improved the mechanical properties of the andalusite composite ceramics and the excessive of dolomite decreased the bending strength [20].

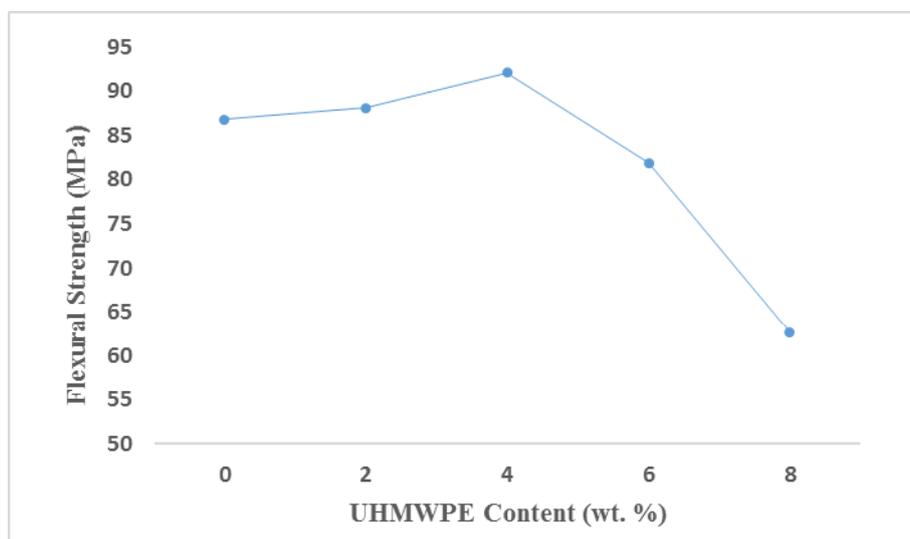


Figure 1: Flexural Strength of Kaolin Geopolymer Ceramics with various UHMWPE loading sintered at 1200 °C.

3.2 Morphology Analysis

Figure 2 shows SEM micrograph for raw kaolin while figure 3 kaolin geopolymer ceramics sintered at 1200 °C. Raw kaolin (Figure 2) has plate-like structure which contributes to the low reactivity compared to fly ash or any other aluminosilicate materials [21]. The appearance of small pores throughout the sample of kaolin geopolymer ceramics sintered at 1200°C denotes the transformation of amorphous to nepheline crystalline ceramic [22]. Figure 4 shows SEM micrographs for kaolin

geopolymer ceramics with various UHMWPE content sintered at 1200 °C. The microstructure of fractured surfaces of kaolin geopolymer ceramics at various UHMWPE content showed that the microstructure of the investigated materials was considerably affected by the content of the UHMWPE. The number of pores and connected of pores increased with the increasing of UHMWPE content. As mention above that the more UHMWPE addition would result in higher porosity, the flexural strength decreases with the increasing of UHMWPE content. The strength is highly related to the size, quantity and distribution of pores, as well as the morphology and size of crystals [23].

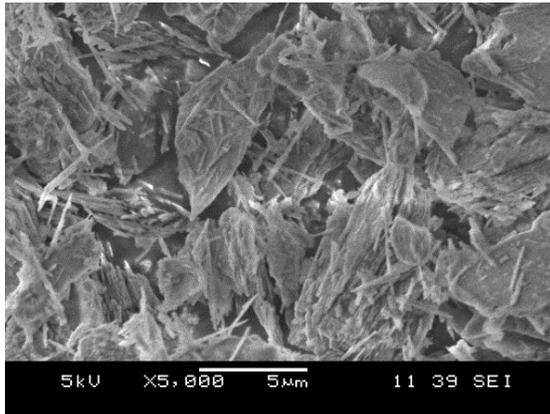


Figure 2: SEM micrograph of raw kaolin

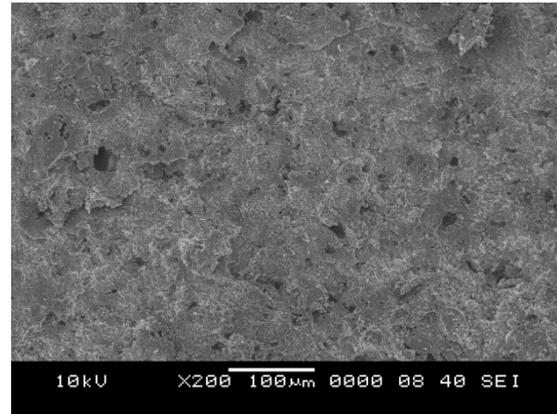


Figure 3: SEM micrograph of kaolin geopolymer ceramic sintered at 1200 °C.

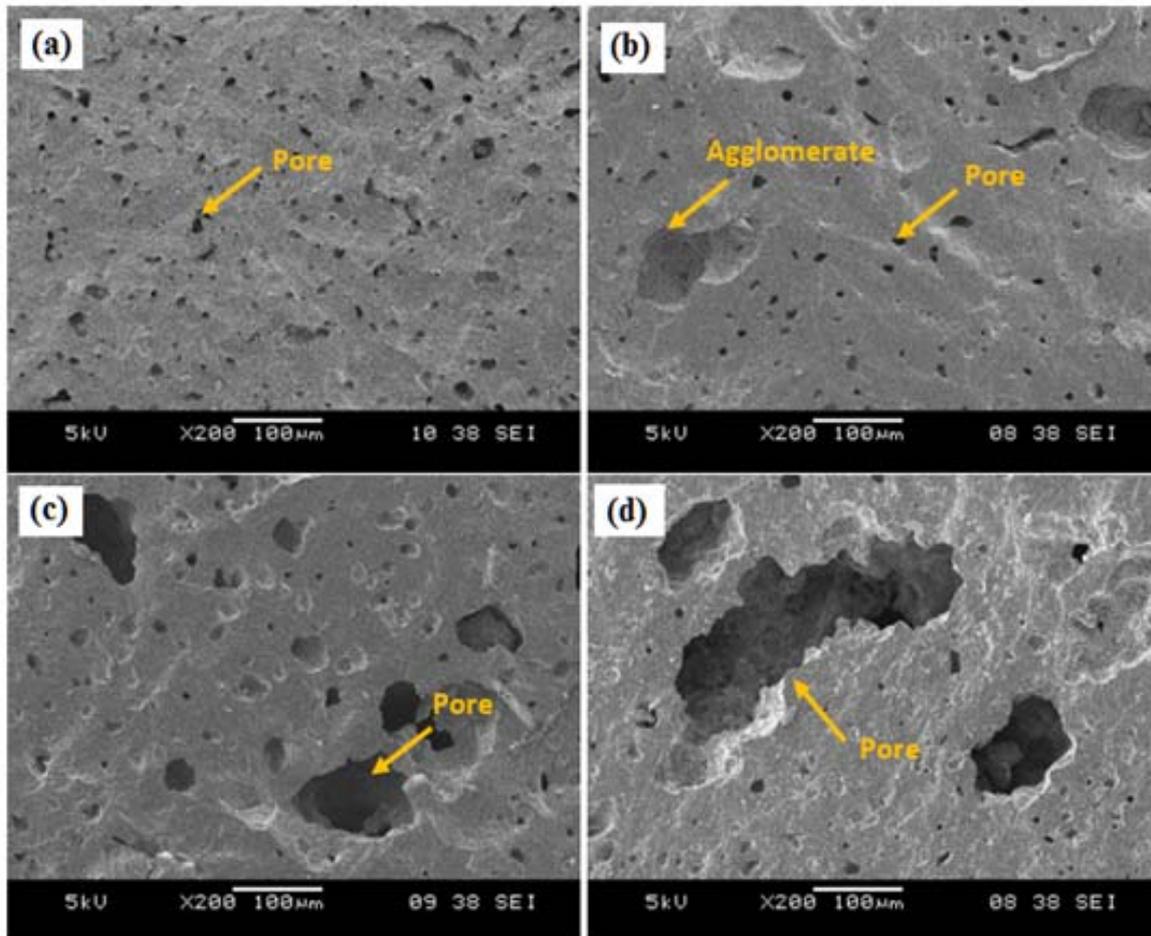


Figure 4: SEM micrograph of kaolin geopolymer ceramics at various UHMWPE loading (a) 2 wt.%, (b) 4 wt.%, (c) 6 wt.%, and (d) 8 wt.%.

4. Conclusion

The aims of this paper is to study the effect of addition of Ultra High Molecular Weight Polyethylene (UHMWPE) on kaolin geopolymer ceramics. The results concluded that the addition of UHMWPE affected the strength of the kaolin geopolymer ceramics. The flexural strength of kaolin geopolymer ceramics with addition of UHMWPE increased compared to the kaolin geopolymer ceramics without addition of UHMWPE. Kaolin geopolymer ceramics with addition of 4 wt. % UHMWPE give the highest strength which is 92.1 Mpa. However, the strength started to decrease due to the excessive content of UHMWPE lead to the increasing of pores size appeared in microstructure of kaolin geopolymer ceramics.

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