



# Article Geopolymer-Based Nepheline Ceramics: Effect of Sintering Profile on Morphological Characteristics and Flexural Strength

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**Abstract:** The focus of this study is the fabrication of innovative and sustainable ceramic-based geopolymer with improved low temperatures performances. Kaolin was mixed with liquid sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) and 12M of sodium hydroxide (NaOH) solution using alkali activator ratio of 0.24 and solid-to-liquid ratio of 1:1 to synthesize kaolin geopolymer. The effect of the sintering profile on the microstructure, pore evolution and flexural strength were investigated. The heating exposure aided consolidation and created a fairly uniform microstructure, resulting in a smooth surface texture. In comparison to the unheated geopolymer, 3D pore distribution showed a significant increase in the range size of ~30  $\mu$ m with the appearance of isolated and intergranular pores. The flexural strength at 1200 °C with a heating rate of 5 °C/min and was increased by 146.4% to 85.4 MPa, as compared to the heating rate of 2 °C/min. The sintering process has an impact on the final microstructure formation thus improving the characteristic of geopolymer-based nepheline ceramic.

Keywords: ceramic-based geopolymer; geopolymer; nepheline; sintering; tomography

## 1. Introduction

Intriguing features of geopolymers, such as superior thermal characteristics, remarkable early strength, great acid resistance, low permeability and tunable kinetics of curing at low manufacturing cost, have drawn extensive attention in the past decade [1]. In general, geopolymers are large inorganic molecules synthesized by alkali treatment of aluminosilicates in a strongly alkaline solution at low temperatures [2]. Aluminate and silicate species were produced by binding together through a polycondensation reaction, resulting in the formation of a compact amorphous and semi-crystalline three-dimensional structure of polymeric Si-O-Al bonds [3,4]. The reactivity of aluminosilicate was affected by their chemical and mineral content, particle size distribution, morphology and crystalline/amorphous content. Moreover, the unique features of geopolymer can be utilized in a wide range of applications including building and construction materials [5–12], thermal insulation coatings [13], fire-resistant materials [14], repair and strengthening composites, low energy ceramic tiles [15] and refractory materials [16]. Another method of utilizing geopolymer technology is to reduce the high temperature necessary in the production



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). of ceramic materials. Sintering can transform geopolymer amorphous to semicrystalline phases into crystalline ceramic phases.

Materials classified as ceramics can be either crystalline or partly crystalline structures or amorphous (e.g., glass) [17]. Due to their high melting point, ceramics were frequently referred as refractory. Varying types of ceramics come with different performances, but, in general, they have high compression strength, high modulus, low heat conductivity, high hardness and are chemically inert [18]. Additionally, several factors were recognized as significant parameters that influence the characteristics and performance of ceramic materials such as the material utilized, method of fabrication and sintering profile. To facilitate the practical application of geopolymer, a considerable number of researchers have been devoted to studying the fabrication of ceramic materials using the geopolymer method. For instance, according to Xie et al., the sintering temperature of geopolymer above 1000 °C was adequate to consolidate the sample and begin the crystallization of leucite [19]. Another representative work was conducted by Peigang et al., in which a substantial amount of leucite was developed by sintering the geopolymer at 800 °C [20]. By directly sintering the geopolymer precursors at a heating rate of 2 °C/min, the bullet-shaped and plated-shaped leucite ceramic can be obtained without cracking.

Sintering processes, structural rearrangement and production of crystalline phases, such as nepheline, kalsilite and mullite, occurred as a result of high temperature exposure. The sintering profile will enhance the material densification, whereas diffusion acts as a mechanism to favor densification as well as grain growth [21]. Sintering and crystallization can occur sequentially or concurrently. If crystallization occurs before the sintering process is completed, the viscosity rises quickly and the sintering process ceases, leaving a porous material [22]. In some cases, the sintering process is faster than the crystallization process, depending on the heating rate. However, the final structure of the ceramic-based geopolymer was rarely reported to be affected by the heating rate. In the current work, the interaction between the heating rate, sintering temperature and the microstructure, flexural strength and pore evolution of the ceramic-based geopolymer was investigated.

#### 2. Materials and Methods

### 2.1. Sample Preparation and Experimentation

Fine particles of kaolin (0.7  $\mu$ m to 150  $\mu$ m) were used as geopolymer starting material, supplied by Associated Kaolin Industries Sdn. Bhd. Selangor. The chemical composition of kaolin is presented in Table 1. With a purity of 99% sodium hydroxide (NaOH) caustic soda micropearls that were marketed as Formosoda-P were supplied from Taiwan. Liquid sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) was supplied by South Pacific Chemicals Industries Sdn. Bhd. (SPCI), Pahang, Malaysia. It is composed of 30.1% of SiO<sub>2</sub>, 9.4% of Na<sub>2</sub>O, and 60.5% of H<sub>2</sub>O (modulus, SiO<sub>2</sub>/Na<sub>2</sub>O = 3.2).

Table 1. Chemical composition of kaolin (wt.%) obtained by X-Ray fluorescence.

Element	Wt. (%)
Silicon Oxide (SiO <sub>2</sub> )	54.0
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	31.7
Iron (II) Oxide (Fe <sub>2</sub> O <sub>3</sub> )	4.89
Potassium Oxide (K <sub>2</sub> O)	6.05
Titanium Dioxide (TiO <sub>2</sub> )	1.41
Manganese Oxide (MnO <sub>2</sub> )	0.11
Zirconium Oxide (ZrO <sub>2</sub> )	0.10
LOI	1.74

To obtain a fully dissolved alkali activator solution, the 12 M NaOH solution was mixed with  $Na_2SiO_3$  at a molar ratio of 0.24. The solution was prepared for 24 h prior to use. To prepare the geopolymer paste, an appropriate amount of prepared alkali solution with the kaolin powder was mixed and stirred using a mechanical mixer until workable

pastes were obtained. The fresh paste was then poured into HDPE mold and then placed in an oven for 24 h at 80 °C for curing. The cured geopolymer was crushed using a mechanical crusher (JHL., KL., Malaysia) and sieved using 150 microns to obtain a fine powder.

The fine powder was compressed using a 12-mm-diameter cylindrical stainless-steel die at 5 tons for 2 min using a cylindrical stainless-steel die (Ipoh, Perak, Malaysia). In an electrical furnace (LT Furnace Model HT4-1600-SIC, (Shanghai, China), the final green body was sintered at 900 °C, 1000 °C, 1100 °C and 1200 °C on an alumina plate. The soaking time used was 180 min at various heating rates of 2 °C/min, 5 °C/min and 10 °C/min. Then, the samples were allowed to be cooled in the furnace as shown in the sintering profile in Figure 1.



Figure 1. Sintering profile for fabrication of geopolymer-based nepheline ceramic.

#### 2.2. Investigation of Unsintered and Sintered Geopolymer

The chemical constituents of kaolin were examined by X-ray fluorescence (XRF). The microstructural changes of kaolin geopolymer were investigated by using JSM-6460LA model Scanning electron microscope (JEOL, Peabody, MS, USA), equipped with secondary electron and energy-dispersive x-ray spectroscopy. The samples were palladium-coated before imaging using Auto Fine Coater, model JEOL JFC 1600 (Peabody, MS, USA).

The mechanical properties of the geopolymer-based nepheline ceramics were determined in terms of flexural strength. The three-point bending fixture was used at dimensions of 7 mm  $\times$  5 mm  $\times$  52 mm, in accordance with ASTM C-1163b. The support span was 30 mm long, with a crosshead speed of 0.3 mm/min. To reduce random error, five samples were assessed, and the average flexural strength was computed.

Pore evolution of unsintered and sintered samples at 1200 °C with an optimum heating rate of 5 °C/min was tested using tomographic scanning using Synchrotron radiation X-ray tomographic microscopy (SXTM). SXTM was carried out at Synchrotron XTM beamline (BL1.2 W: X-ray imaging and tomographic microscopy), Synchrotron Light Research Institute, Thailand. To prevent displacement and dehydration, each sample was placed in a cylindrical sample holder filled with formaldehyde-soaked cotton. The sample projections included a 200-mm-thick YAG:Ce scintillator, a white beam microscope (Optique Peter, France) and a pco.edge 5.5 sCMOS camera (2560 × 2160 pixels, 16 bits). The total tomographic scan was discovered with a pixel size of 1.44 mm, providing a field of view ( $3.1 \times 3.7 \text{ mm}^2$ ). Two reconstruction tomographic volume scans were performed to identify the fine features of the samples. The first 180° scan was captured over on the vertical axis of rotation as shifted parallel and horizontally while the second 180° scan was flat-field corrected, stitched and reconstructed using Octopus software. Drishti software was used to render 3D images of tomographic volume.

#### 3.1. Microstructural Analysis

Figure 2 shows the SEM micrograph of raw kaolin and kaolin-based geopolymer. The SEM micrograph of surface particles of raw material indicates that the plate-like structure of kaolin contributed to a smaller surface area for the geopolymerization process. However, once the raw kaolin was activated by an alkali activator, the structure of kaolin has been altered and became more compact at the edges of plate-like particles. The continuous gel-like structure with pores confirmed the presence of a pure geopolymer binder that further improves the structure thus contributing to the overall strength [23].



Figure 2. SEM micrograph of (a) raw kaolin and (b) kaolin-based geopolymer.

Figure 3 displays the surface morphology of the fracture surface of sintered samples at 900 °C and 1200 °C with different heating rates. As the sintering temperature reached 900 °C, a neck connection was formed among the geopolymer particles. Additionally, the edge particles became smoother with more neck connections. More geopolymer particles bonded and agglomerated into large particles at a sintering temperature of 1200 °C due to excessive molten phase formation. Furthermore, the number of pores was also reduced. Dai et al. [24] found that sintering at high temperature causes the particles to diffuse well, resulting in the formation of broad sintered area thus exhibit a smooth structure.

Meanwhile, the heating rate also shows a great influence on the morphology of the samples. For the samples heated at a rate of 5 °C/min and below for both sintering temperatures, the structure is more compact and fewer pores has been observed. However, for the sample with a high heating rate of 10 °C/min, some pores existed. The usage of a high heating rate leads to a densification process at lower viscosity and raises the rapid densification rate thus resulting in high porosity caused by a lack of particle rearrangement during sintering. The result reveals that heating rate had a great effect on the morphology and the connectivity of the ceramic particles which can affect the final density and average grain size. Rui et al., reported that water that existed in micro-pores inside the geopolymer can be slowly removed with less crack formation by controlling the heating and drying process of sintering [25].



**Figure 3.** SEM micrograph of geopolymer-based nepheline ceramics sintered at 900 °C and 1200 °C with different heating rates of (a)  $2 \degree C/min$ , (b)  $5 \degree C/min$  and (c)  $10 \degree C/min$ .

#### 3.2. Flexural Strength

The effect of heating rate on flexural strength and the relative percentage change of geopolymer-based nepheline ceramics sintered at 900 °C and 1200 °C is shown in Figure 4. The result shows that the heating rate and sintering temperature give a significant effect on the flexural strength of geopolymer-based nepheline ceramics. The flexural strength increased with the increase in sintering temperature as well as heating rate. The lowest flexural strength was recorded at 13 MPa for the unsintered sample. The sample sintered at 1200 °C with 5 °C/min of heating rate having the highest flexural strength of 86.5 MPa. At high temperatures, the atoms of geopolymer particles continuously diffused and migrate in the matrix. The rearrangement of atoms in the lattice turns the quartz into liquid-fused silica [26]. This liquid phase will fill the gaps and occupy the pores at high temperatures resulting in a denser system. Therefore, the distance between the particles decreased, leading to densification and the system exhibited a relatively smooth structure in the matrix as shown in the microstructural analysis.

While the lowest flexural strength was noted at a sintering temperature of 900 °C at 2 °C/min. A low sintering temperature with a low heating rate was not sufficient to promote diffusion. Further increments of heating rate to 10 °C/min degrade the strength for both sintering temperatures. The time needed to reach the sintering temperature is decreased with a higher heating rate. The samples were kept for a short period at intermediate temperatures, which were not adequately high to encourage grain growth [27]. Grain growth is strongly time-dependent process. Furthermore, rapid grain growth also indicates a decrease in densification rate. Thus, the high heating rate would lead to the aberrance of the final pores resulting in decreasing in flexural strength. The schematic diagram of the sintering mechanism of geopolymer is illustrated in Figure 5.



**Figure 4.** (a) Flexural strength and (b) relative percentage change of unsintered and geopolymerbased nepheline ceramics sintered at 900 °C and 1200 °C with different heating rates of 2 °C/min, 5 °C/min and 10 °C/min.



**Figure 5.** Schematic diagram of sintering mechanism of geopolymer-based nepheline ceramics sintered at 900 °C and 1200 °C.

## 3.3. Porosity Evolution Analysis

Figure 6 illustrates the 3D pore structures, pore size distribution with pore content of kaolin-based geopolymer and geopolymer-based nepheline ceramics sintered at 900 °C and 1200 °C at 5 °C/min of heating rate. These samples were used in this study to reveal the pore structure due to the highest flexural strength. The unsintered kaolin-based geopolymer shows the distribution of small pores throughout the sample. The sintered samples at 900 °C had wider pores (Figure 6b) which indicated a purple color "air" space, compared to 1200 °C (Figure 6c). Both sintered samples had the most pore sizes ranging from 10 to 15  $\mu$ m, with counts of 1217 and 958, respectively (Figure 6d). Moreover, geopolymer-based nepheline ceramics sintered at 900 °C had larger pore sizes which contributed to a lower flexural strength as larger pores initiated the flexural failure. The decrease in pores size with the increase in sintering temperature from 900 °C to 1200 °C can be explained by the densification of the body, which promotes partial elimination of porosity at high temperature. The diffusion of atoms to points of contact created bridges, resulting in pore size reduction.



**Figure 6.** Three–dimensional pore structures with pore distributions of (**a**) unsintered kaolin-based geopolymer, geopolymer-based nepheline ceramics sintered at (**b**) 900  $^{\circ}$ C and (**c**) 1200  $^{\circ}$ C, and (**d**) pore contents and pore size counts.

The unsintered kaolin-based geopolymer had the lowest total pores of 0.14% with 0.05% of open pores and 0.09% closed pores (Figure 6d). The pores that appeared in the sample were classified as microstructural pores resulting from entrapped gas introduced

by the gas atomization process of powder particles. The geopolymer-based nepheline ceramics sintered at 900 °C had a drastic increase of 9.69% of total pores with 2.89% of open pores and 6.80% of closed pores compared to the unsintered sample. This increment was attributed to the thermal expansion caused by the vapor pressure resulting from the escaped residual water content as well as oxidation and crystallization of the oxides in the geopolymer [28]. For the sample sintered at 1200 °C, the total porosity recorded was 8.59% (4.41% open pores and 4.54% close pores). As the sintering temperature increased, more crystalline phases were formed, and pore structure was more refined compared with the SEM micrograph in Figure 3.

### 4. Conclusions

The microstructure, flexural strength and pore evolution of geopolymer-based nepheline ceramics at different sintering temperatures were investigated experimentally in this paper. The effect of heating rate on microstructure and flexural strength of geopolymer-based nepheline ceramics was examined. The microstructure analysis showed that a compacted and smooth surface was obtained at the sintering temperature of 1200 °C at a heating rate of 5 °C/min having the highest flexural strength of 86.5 MPa. Higher sintering temperatures attain structural consolidation, resulting in a relatively homogenous and smooth structure. High porosity derived from a lack of particle rearrangement during sintering was induced by the use of a high heating rate. This continuous diffusion and migration of atoms leads to promoting densification and grain growth as well as increasing flexural strength. The tomography analysis concludes that phase transformation taking place during the sintering process increases the appearance of pores from 0.14% to 9.69%, indicating a considerable contribution to the enhancement of characteristics. The knowledge of sintering temperature and profile is required in understanding the attempt to enhance the mechanical and microstructure properties of ceramic-based geopolymer

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