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Research Article

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# Characterization of Geopolymer–based Materials Synthesized from Fly Ash and Rice Husk Ash

**Nguyen Hoang Luong Ngoc**

Faculty of Chemical Technology, Ho Chi Minh City University of Food Industry, Viet Nam

**Abstract** Investigations on geopolymer-based materials are always interesting topics for researchers. Geopolymer is environmentally friendly material which has been potential applications for many different fields such as technical materials, building materials, insolation or refractories, and others. This study used fly ash (FA) and rice husk ash or rice hull ash (RHA) as raw materials for geopolymerization process to develop novel materials with high porosity. Both FA and RHA are industrial waste of coal fired thermal power plants and agricultural wastes, respectively that need to be managed to reduce their negative impact to the environment. FA and RHA contain high alumino-silicate resources were mixed with sodium hydroxide solution for 10 minutes to obtain the geopolymer pastes. Sodium hydroxide solution was used as an alkaline activator to form geopolymer paste. The geopolymer paste was filled into 5-cm cube molds according to ASTM C109/C109M 99, and then cured at room temperature for 28 days. These products were then tested for compressive strength and volumetric weight. Results indicated that the material can be considered lightweight with a compressive strength at 28 days that are in the range of 18.3 to 35.5 MPa, volumetric weight around 800kg/m$^3$ and water absorption is under 215.65 kg/m$^3$. The properties of raw materials and geopolymer products were also determined by analytical techniques that included chemical composition by X Ray Flourescence (XRF), mineral composition by X Ray Diffraction (XRD), and structural bonds by Infrared spectroscopy (IR).

**Keywords** Lightweight Materials, Geopolymer, Fly Ash, Rice Husk Ash, Sodium Hydroxide Solution, Alkaline Activator, Engineering Properties

## Introduction

Geopolymer, originally named as "soil cement", is a kind of synthetic alumino-silicate material that is found to have several applications including as a material for high-performance composites, ceramics, as well as, as a replacement for Portland cement [1-4]. Geopolymer-based materials are capable of setting rapidly with high final compressive strength and they are highly resistant to chemical attack. Thus, it is predicted that this new material would be popularly applied as a sustainable construction material in the future [1].

Since the raw materials of geopolymer are mainly composed of aluminium oxide, silicon dioxide, and other oxides, its mechanical properties are influenced by the development of its microstructure. Geopolymerization is based on a chemical reaction between different alumino – silicate oxides ($Al^{3+}$) with silicates under highly alkaline conditions, yielding polymeric Si–O–Al–O bonds [5]. The thermal and chemical stability of these bonds are hypothesized to be determined by the nanostructure and molecular structure within the gel phase [1, 5]. In the raw materials, these oxides are believed to be in amorphous or semi – crystalline phases that participate in geopolymerization process whereas the crystalline phases do not take part in forming the geopolymers [1, 5-8].

This research presents the utilization of fly ash and rice husk ash as raw materials to produce a geopolymer-based material. These raw materials constitute the blend of the alkali-activated binder in this study. FA was used as the primary source of reactive alumina and silicate. It is an industrial waste of coal-fired power plants, which is estimated to be over 650 Mt/year worldwide [9-11]. Rice husk ash was used as the primary source of reactive silica. It is a by-product of burning agri-waste particularly rice husk, with an estimated generation rate of over 30 million metric tons per year worldwide [12-14]. It is highly porous, lightweight material with very good pozzolanic properties which is used to produce cheap insulating refractory materials (e.g., see [11]).

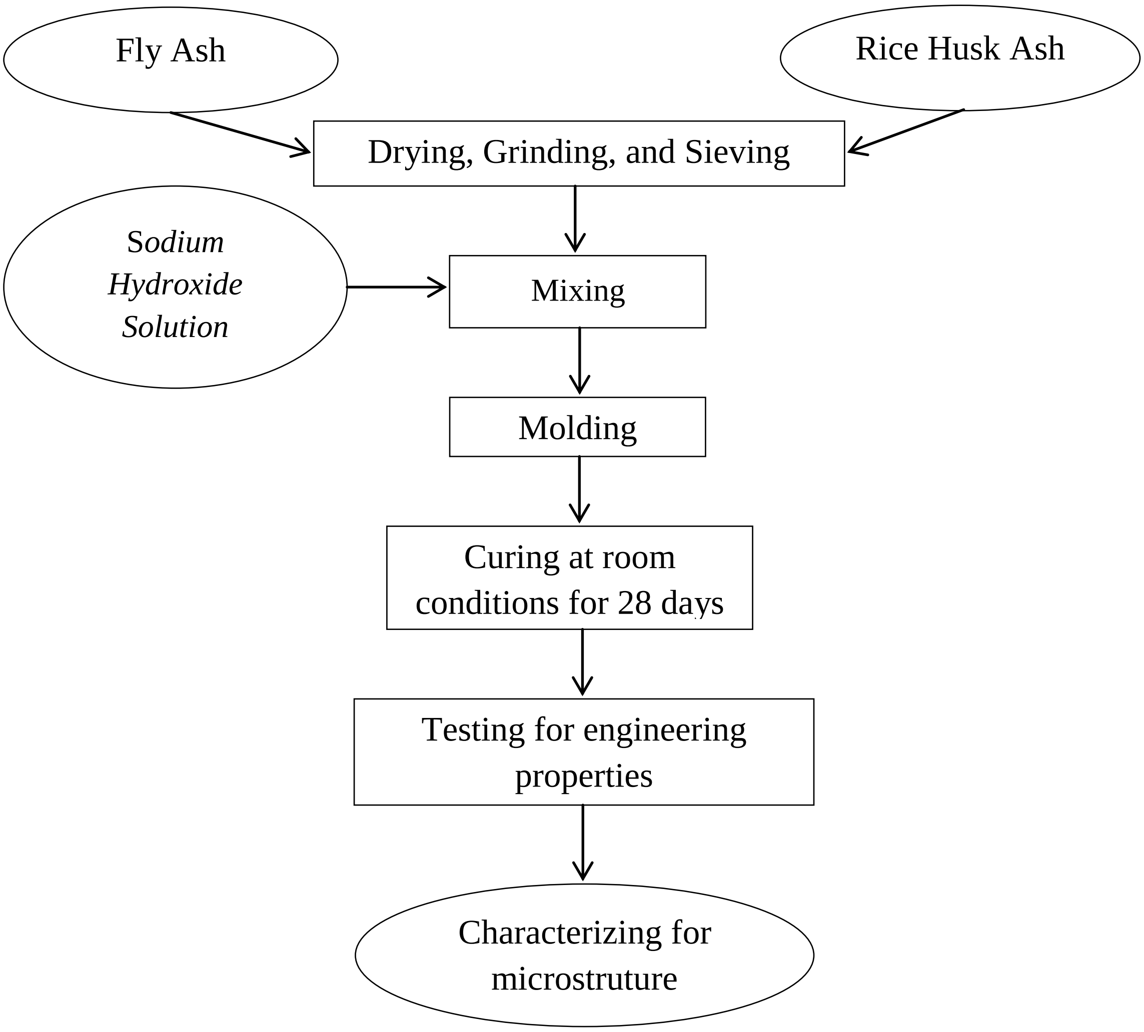


*Figure 1: The flow of experimental process*

In Viet Nam, There has been no study research on geopolymer using FA and RHA as raw materials and no evaluations for effects of alkaline activators to formation of the geopolymer-based materials. Especially in Vietnam, this is the first study which is carried out for production of Geopolymer-based materials with using the different concentration of sodium hydroxide solution (SHS).

**Materials and Methods**

In this research, both FA and RHA from Vietnam were used to produce the geopolymer mixture. They are the raw materials in geopolymerization reactions to create the mechanical strength for product. FA waste came from Vinh Tan coal-fired thermal power plant, Binh Thuan Province, Vietnam was dried at 110 $^oC$ for 24 hours and then passed 90µm-siever to obtain the raw material. On the other hand, rice husks from Mekong delta, South of Viet Nam were burned at 650$^o$C for 2 hour at Laboratory of Ceramic Materials to obtain rice husk ash. RHA was ground using ball miller for 30 minutes, and then passed 90µm-siever to obtain the raw material in powder. All of the raw materials were characterized for chemical and mineral compositions using XRF and XRD, respectively. On the other hand, *sodium hydroxide solution (SHS) was a commercial product from Bien Hoa Chemical Factory, Dong Nai province, Viet Nam.*

**Table 1:** Mix proportions used in the design of experiments

| Mixture (Sample) | Proportion of solid powders (%wt) | | Concentration of SHS (M) |
|---|---|---|---|
| | FA | RHA | |
| GeoNa0 | 50 | 50 | 0 |
| GeoNa2 | 50 | 50 | 2 |
| GeoNa4 | 50 | 50 | 4 |
| GeoNa6 | 50 | 50 | 6 |
| GeoNa8 | 50 | 50 | 8 |
| GeoNa10 | 50 | 50 | 10 |

In this study, FA and RHA powder were mixed with SHS concentration from 0 to 10M, ratio of solid powder per liquid solution was at 0.4 (in weight) using a laboratory cement mixer. **Table 1** showed the mix proportions of FA, RHA, and SHS concentration for producing of geopolymer-based materials. The fresh pastes of the geopolymer were formed in standard cubic molds with size of 50mm and cured for 28 days at room conditions (25 $^{o}$C, 85% humidity). Engineering properties of the geopolymer products were evaluated by testing for compressive strength (MPa), volumetric weight (kg/m$^3$), and water absorption (kg/m$^3$). From the experimental data, this paper conducts for evaluation on effects of SHS to engineering properties of the geopolymer products. All of the tests for engineering properties were carried out according to ASTM C109/C109M [19] and ASTM C140 [20]. Moreover, the geopolymer sample with the highest strength and low water absorption and volumetric weight was characterized microstructure using XRD and SEM.

## Results and Discussion

### Properties of Raw Materials

The raw materials of FA and RHA were tested for physic-chemical properties and the results as shown in Table 2. In which FA has moisture content of 6.52%, bulk density of 0.91 g/cm$^3$, apparent density of 2.42 g/cm$^3$, mean particle size of 48.2 µm, and LOI value at 3.82%. For the chemical composition using X-ray fluorescence (XRF), FA *contains* 35.36% of $Al_2O_3$, 47.74% of $SiO_2$, 7.02% of $Fe_2O_3$, and others. Figure 2 shows XRD pattern of FA which is in amorphous phases of alumina and silica suitable for geopolymerization reactions at high alkaline condition. For mineral compositions, FA has quartz, mulite, silimanite, and hematite.

For RHA, it has moisture content of 0.59%, bulk density of 0.59 g/cm$^3$, apparent density of 2.17 g/cm$^3$, mean particle size of 66.84µm, and LOI value at 1.79%. For the chemical composition using x ray fluorescence (XRF), RHA *contains high silica with* 93.2% of $SiO_2$, 0.43% of $Al_2O_3$, and others. Figure 3 shows XRD pattern of RHA which is in amorphous phases of silica suitable for geopolymerization reactions at high alkaline condition. For mineral compositions, RHA has cristobalite and tridymite.

**Table 2:** Physico-chemical properties of DE and WGS

| Physico-chemical properties | FA | RHA |
|---|---|---|
| $Al_2O_3$ | 35.36 | 0.43 |
| $SiO_2$ | 47.74 | 93.20 |
| $Fe_2O_3$ | 7.02 | 0.33 |
| $Na_2O$ | 0.69 | 0.22 |
| $K_2O$ | 2.61 | 0.41 |
| Others | 2.76 | 3.62 |
| L.O.I | 3.82 | 1.79 |
| Moisture content (%) | 6.52 | 0.59 |
| Bulk density (g/cm$^3$) | 0.91 | 0.59 |
| Apparent density (g/cm$^3$) | 2.42 | 2.17 |
| Mean particle size (µm) | 48.20 | 66.84 |

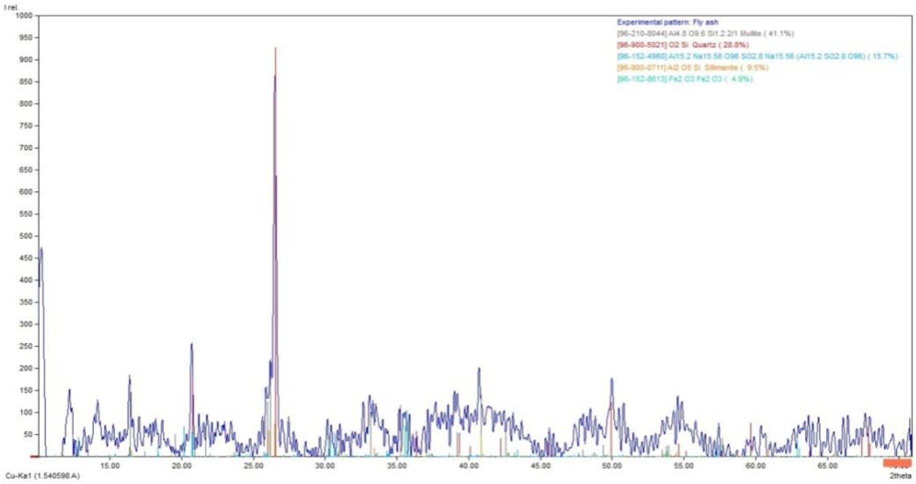

*Figure 2: XRD patterns of FA*

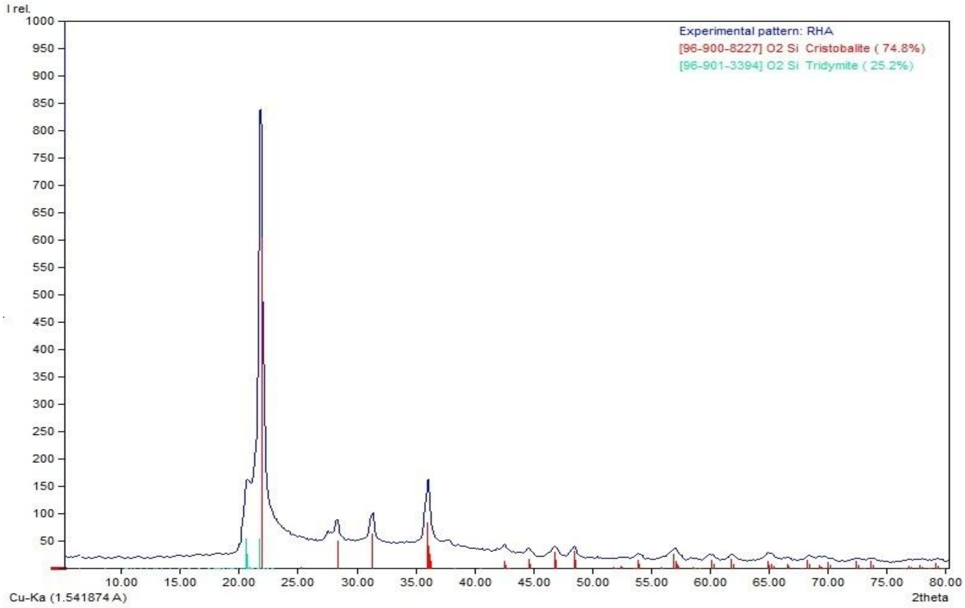


*Figure 3: XRD patterns of RHA*

**Effects of Alkaline Activator to Engineering Properties of Geopolymer Products**

The values of volumetric weight for all geopolymer-based materials are in range of 779 to 812 kg/$m^3$ which are less than the prescribed volumetric weight (1680 kg/$m^3$) for a lightweight concrete brick in ASTM C55-99 and ASTM C90-99a [21-22]. This property is inherited from the porous material of diatomite as described in term of 3.1. Moreover, the geopolymerization processes and the evaporation of water during the cured process were also produced micro-pores in the geopolymeric structures [1-2, 4-5, 15-16].

**Table 3:** Engineering properties of geopolymer specimen

| Samples | Volumetric weight (kg/$m^3$) | Compressive strength (MPa) | Water absorption (kg/$m^3$) |
|---|---|---|---|
| GeoNa0 | 784 | 0.46 (unformed when exposed in water) | - |
| GeoNa2 | 792 | 18.33 | 215.65 |
| GeoNa4 | 812 | 28.68 | 204.44 |
| GeoNa6 | 798 | 35.51 | 179.64 |
| GeoNa8 | 806 | 32.45 | 180.52 |
| GeoNa10 | 779 | (Swelling solid with macro-cracks) | 186.18 |

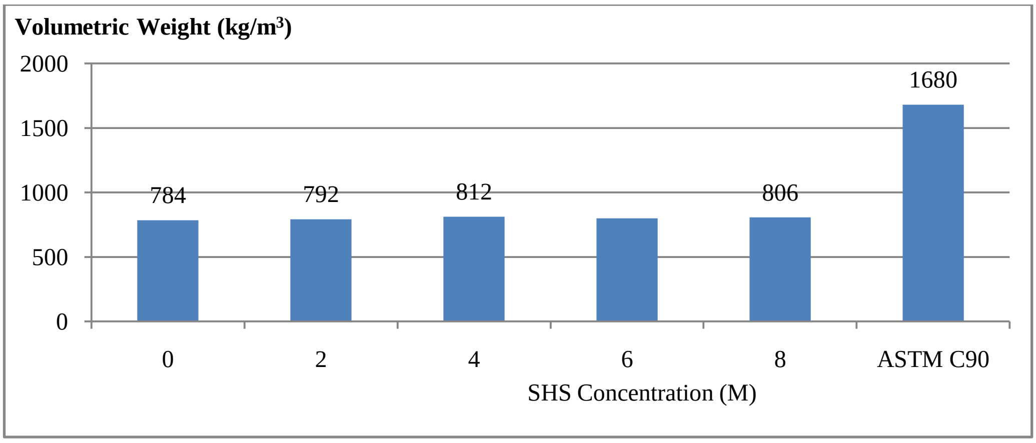


*Figure 4: The lower values of volumetric weight compared with ASTM C90 for lightweight concrete brick*

Table 3 showed that the geopolymer sample of GeoNa0 without SHS or alkaline activator had no strength and it was failed in water (test of water absorption), because the activated alumino-silicate resources were impossible to react geopolymerization without alkaline condition. The Geopolymer-based geopolymer specimens with SHS 10M appeared phenomena of swell and macro-cracks. This is related an increase of temperature in high sodium geopolymer paste because of enthalpy of dissolution of the formed NaOH [17, 18]. High temperature in fresh geopolymer pastes may cause thermal stress that produce cracks in the sample. In fact, the geopolymer specimen with SHS 8M was decreased compressive strength at 32.45MPa and increased water absorption at 180.52 kg/m$^3$. For SHS concentration from 2 to 6M, the geopolymer specimens increased significantly the compressive strength from 18.33 to 35.51MPa and decreased steadily water absorption from 215.65 to 179.64 kg/m$^3$. These are effects of SHS concentration or alkaline activators to engineering properties of the geopolymer-based materials which are interested in investigations. Thus, the SHS concentration should be optimized around 6M to obtain the geopolymer-based materials prescribed limit according to ASTM C55 or C90 [21-22] requirements for lightweight concrete brick materials.

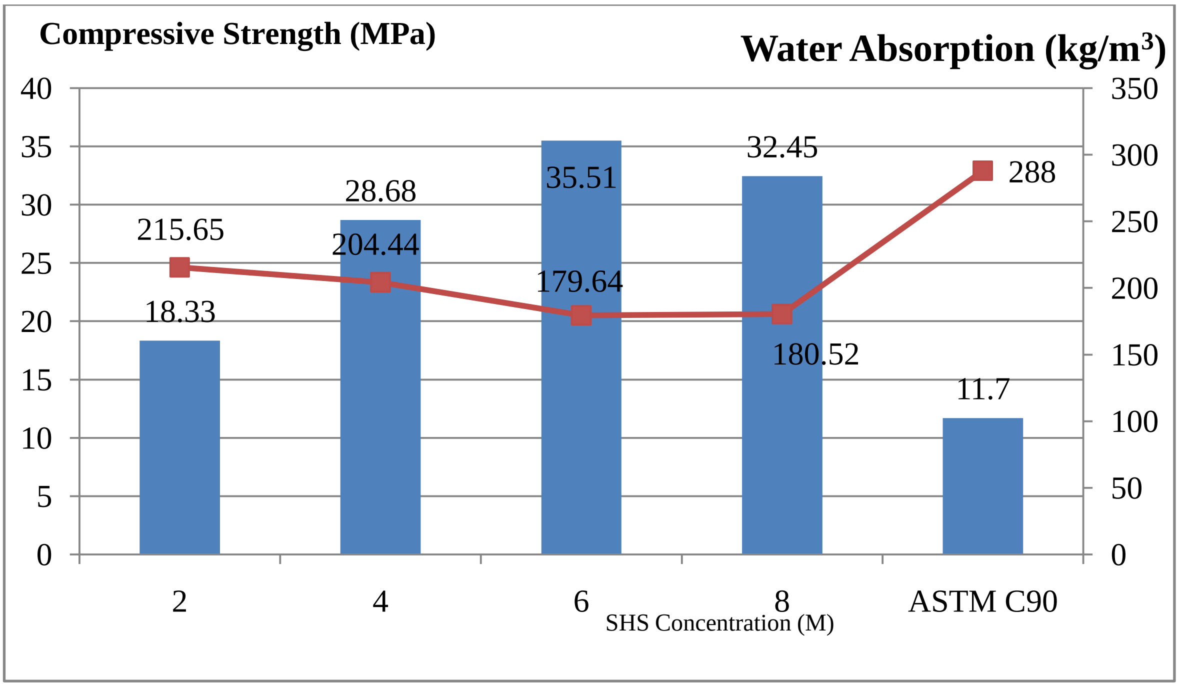


*Figure 5: Relationship among SHS concentration (%, in weight) and engineering properties of geopolymer materials compared with lightweight concrete brick in ASTM C90.*

**Engineering Properties of Geopolymer Products**

The sample of geopolymer-based geopolymer of GeoNa6 had the best engineering properties on low volumetric weight and water absorption (kg/$m^3$) and the highest compressive strength was carried out for analysis of microstructure using XRD and SEM.

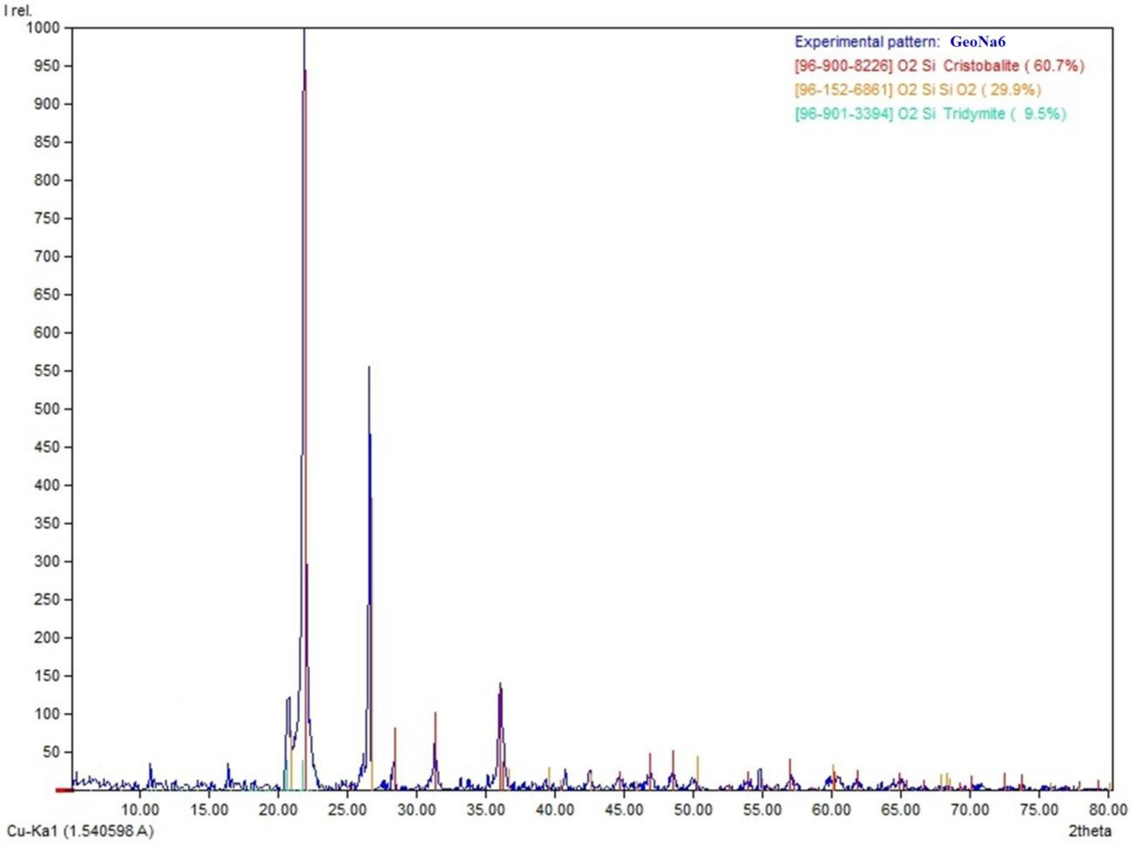


*Figure 6: XRD pattern of the geopolymer-based material with SHS 6M*

The XRD pattern of the geopolymer-based material (GeoNa6) showed differences among structures of raw material (FA and RHA) and the geopolymer product. Many key peaks of clay minerals in FA (Fig. 2) were decreased intensity in the geopolymer (Fig. 6) such as mulite, silimanite, and hematite. This is explained that the geopolymerization reactions were dissolved the alumino silicate to from alumino-slicate networks in amorphous phases. The crystal phases of quartz, cristobaliteand tridymite were dissolved with low concentration and continuously existence in the geopolymer in crystal structures. As a result, the crystal structures appeared in XRD pattern with high intensity among background of amorphous phases in the ash-based geopolymer material.

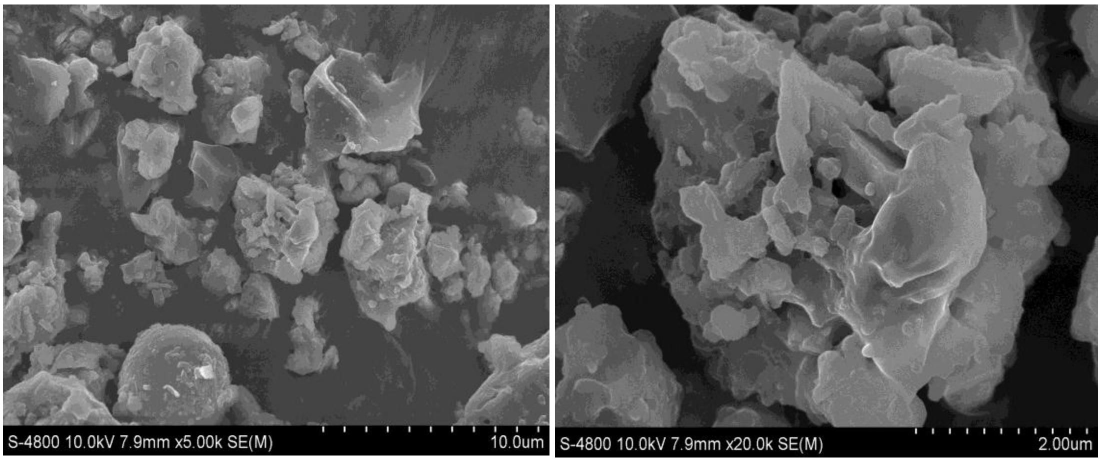


*Figure 7: SEM image of GeoNa6 sample*

Microstructures of the geopolymer-based material (sample of Na6) had changed with appearance of new structure dissolved in background as shown in Figure 6. The porous structures of alumino-silicate have existed with high concentration. This is easy to realize with magnification at 20,000. Thus, SEM images are explanation for the experimental data of volumetric weight and compressive strength in terms of 3.2.

**Conclusions**

This paper presents an experimental study to produce and optimize a light-weight geopolymer-based material from a blend of coal fly ash waste and rice husk ash. The ash-geopolymer based materials with a solid powder mix of 50% FA and 50% RHA and alkaline-activator with concentration of SHS at 6M produced geopolymers with an average 28-day compressive strength of 35.51MPa, water absorption of 179.64.9 kg/m$^3$, volumetric weight of 798 kg/m$^3$. These values were in good agreement with the required values of the ASTM C55 and C90 for lightweight concrete brick. The ternary-blended geopolymer can thus be potentially used as lightweight material for masonry walls or partitions. Future studies will consider chemical resistance of the material and other thermal properties such as thermal conductivities, thermal expansion coefficient. Microstructure of these geopolymers will also be studied further to understand the relationship among composition, microstructure and macroscopic properties of such materials.

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