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Synthesis of Zeolite from Geothermal Waste

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Abstract. The high content of silica in geothermal waste can be used as an alternative source of amorphous silica for the production of silicon based materials. In this research, geothermal waste was used as silica sources in hydrothermal reaction of zeolite synthesis. Zeolite was synthesized by unstirred hydrothermal process. Hydrothermal process was conducted at variation temperature of 100, 110 and 120 °C during 5 hours. In order to study the effect of holding time on hydrothermal product, hydrothermal process was performed for different holding times of: 1, 3 and 5 hours at the temperature 120 °C. All of hydrothermal products were characterized by XRD. The results showed hydrothermal process successfully converts geothermal waste into zeolite A and sodalite.

Introduction

In recent year, production of worldwide electricity based on geothermal sources has increased significantly. As a country that located at the ring of fire, Indonesia has a high potential for geothermal energy. The Indonesian Geological Agency reported geothermal potential for power plant in all Indonesian areas is about 27,441 MW. However, the use of geothermal as energy resources produces geothermal waste. The geothermal power plant produces geothermal sludge and geothermal brine. Most of geothermal waste is of environmental concern, since geothermal waste contains elevated amounts of boron, ammonia, arsenic, and heavy metals compared to ordinary soil [1]. Such amount of geothermal waste cannot directly be disposed into a landfill without prior treatment [2]. Characterization of geothermal waste reported that the major constituent of geothermal waste is silica [3].

The problem of the waste resulted from geothermal power plants is now considered as an opportunity to encourage the precipitation of useful silica by product. The silica has a relatively large specific surface area, which makes it become interesting for several applications. The high content of silica in geothermal waste, was used as partial replacement of Portland cement [4,5]. Utilization of geothermal waste in producing new useful materials is here with the new research area that will expand the positive reuse of this abundant material. It's helping to reduce the environmental and economical impacts of its disposal.

Zeolite is a crystalline microporous solid that has regularity of cavities, channels with molecular dimensions and the diverse framework chemical compositions. There are two kind of zeolite, natural zeolite and synthesized zeolite. Synthesized zeolite is generally produced from hydrothermal reaction of sodium aluminosilicate gel prepared from various silica and alumina [6]. Factors that determine the type of synthetic zeolite produced are time of reaction, temperature, pressure, and synthesis conditions (like the order of mixing, gel aging, and stirring).

The main problem of synthesized zeolite production is the availability and cost of raw material specifically the silica source [7]. For reducing the production cost of synthesized zeolite, many researchers considered alternative materials as silica source. Zeolites A, X and hydroxysodalite (HS) were successfully synthesized from a natural Iranian kaolinite [8]. Wajima et.al (2006) also synthesized zeolite at low temperature (90 °C) from paper sludge ash, with addition of diatomite [9]. Na-X zeolite and X zeolite was also synthesized from solid by-product of oil shale processing [10]. High amount of silica in rice husk were used by many researcher as silica source at zeolite synthesized [11,12]. Coal fly ash was also an interesting materials as starting precursor of synthesized zeolite [13,14]. Previous experiments reported that geothermal waste contains silica.

The present of crystalline and amorphous silica in geothermal waste is potential materials as silica source at zeolite synthesis. Geothermal waste can be used as a cheap alternative source of amorphous silica for the production of silicon based materials with industrial and technological interests. This paper proposes a novel approach to take advantage the geothermal waste with synthesized of zeolite by hydrothermal reaction.

Material and Methods

The geothermal waste was obtained from Geo Dipa geothermal power plant, Dieng, Indonesia. To burn the unexpected materials such as sulphur and other volatile materials that present in geothermal waste, the geothermal waste was calcinated at temperature 850 °C for 3 hours at atmospheric condition. Geothermal waste was characterized by AAS, SEM and XRD. Composition of calcinated and un-calcinated of geothermal waste were analyzed by atomic absorption spectroscopy (AAS) as shown in Table 1. Fig. 1 shows scanning electron micrograph of geothermal waste. Powder X-ray diffraction (XRD) patterns were taken on a Rigaku X-ray diffractometer using CuK α radiation (40 kV, 30 mA). XDR analysis of calcinated geothermal waste and un-calcinated (as receives) geothermal waste were shown in Fig. 2.

Table 1. Composition of calcinated and uncalcinated of geothermal waste

Compound	Un-calcinated (% wt)	Calcinated (% wt)
SiO ₂	50.20	81.26
Al ₂ O ₃	0.05	0.14
Fe ₂ O ₃	0.20	0.45
Na ₂ O ₃	0.62	0.76
others	until 100 %	until 100 %

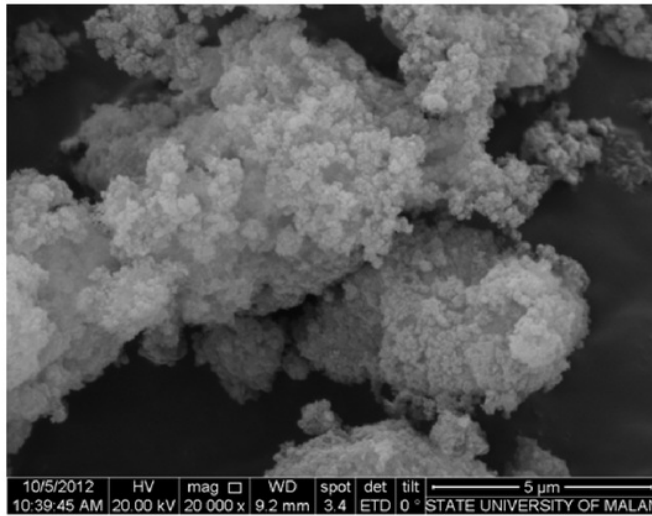


Fig.1. Scanning electron micrograph of geothermal waste

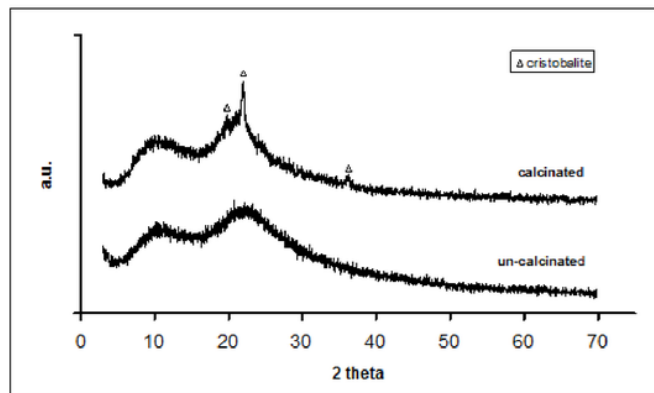


Fig. 2. XRD result of calcinated geothermal waste and un-calcinated (as receives) geothermal waste

Starting precursor for zeolite synthesis was made by: reacted of 3 gr calcinated geothermal waste with 30 mL NaOH 5 M and 30 mL natrium aluminate and stirred at 200 rpm speed rotation at temperature 30 °C for 2 hours. This process results natrium silicate. Hydrothermal reaction was conducted on un-stirred hydrothermal reactor at temperature: 120 °C for 3, 4, 5 hours and at temperature 100°, 110°, 120° C for 5 hours. The zeolitic material obtained from different experiments were filtered and washed with distilled water. Synthesized products were examined by X-ray powder diffraction (XRD) using Cu K α radiation. Various crystalline phases present in the samples were identified with the help of JCPDS. The morphological structures of the synthesized zeolitic materials were obtained by using scanning electron micrograph.

Results and Discussions

The chemical compositions of geothermal waste samples used in the present study are given in Table 1. The geothermal waste contain SiO₂ about 50 %, calcinating process increases % wt SiO₂ in geothermal waste into about 80 %. Fig. 1 shows XRD analysis of un-calcinated and calcinated geothermal waste. It shows that the un-calcinated geothermal waste is mainly composed of SiO₂, Fe₂O₂, and Al₂O₃ in the form of amorphous matter. XRD patterns of calcinated geothermal waste show the presence of cristobalite phase. Calcinated process transforms in part of amorphous silicon into cristobalite. The X-ray diffraction patterns of zeolite synthesized from geothermal waste at variation of hydrothermal temperature and holding time are shown in Fig. 3 and Fig. 4 respectively. From XRD analysis in Fig. 3 and Fig. 4, it can be concluded that hydrothermal process of geothermal waste produces zeolite A and sodalite. The XRD peaks at: 14.091, 24.502, 31.819 and 42.253 indicates that synthesized product contains sodalite. The present of zeolite A is shown with the XRD peaks at: 13.761, 18.665, 23.881 and 27.055. Crystallization of the zeolitic materials occurs through nucleation reaction and crystal growth. Heat energy causes rearrangement of amorphous gel and from nucleus a crystal embryo. The higher hydrothermal temperature will promote the formation of crystal nucleus. This results match well with the work of Bayati et.al. that also reported that the reaction temperature has significant effect on the crystallinity [15].

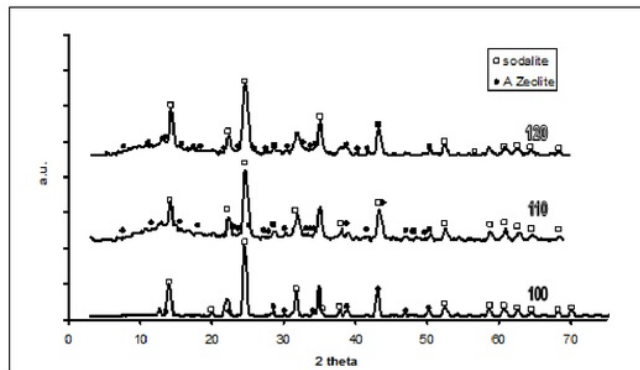


Fig. 3. XDR patterns of hydrothermal product of geothermal waste at variation of temperature

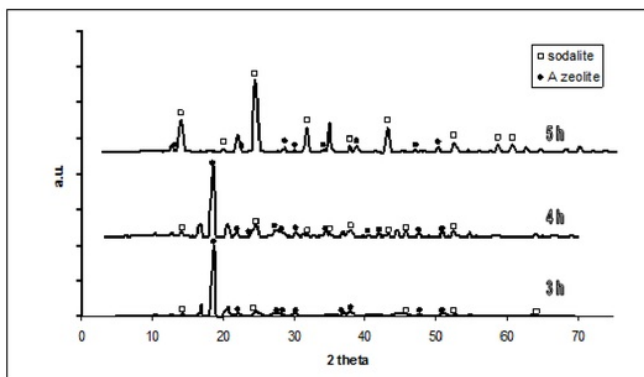
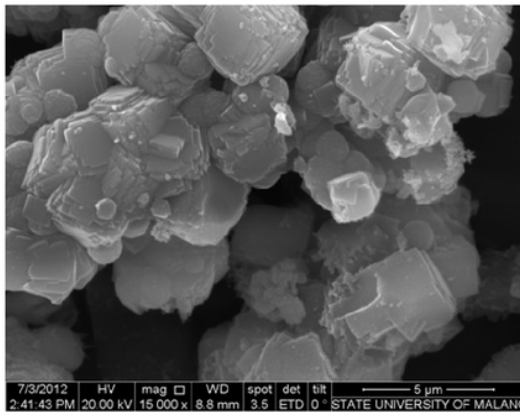
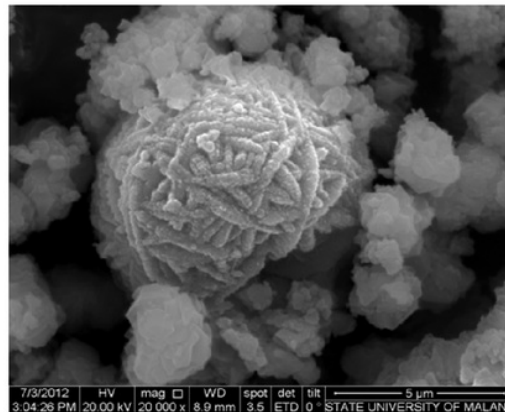


Fig. 4. XDR patterns of hydrothermal product of geothermal waste at the variation of holding time

Fig. 5(a) shows scanning electron micrograph zeolite A. It shows that hydrothermal product is in cubic form. Fig. 5(b) shows SEM photomicrographs of sodalite. SEM photomicrographs show differences of morphology of raw material and hydrothermal product.



(a)



(b)

Fig. 5. Scanning electron micrograph of (a) A zeolite and (b) sodalite

Conclusion

Hydrothermal process has successfully converts geothermal waste into zeolite A and sodalite. At holding time 5 hours, increasing temperature process will increase crystallinity of zeolite product.

The rise of holding time of hydrothermal at temperature process of 120 °C will decrease zeolite A, however at the same time sodalite produced from hydrothermal will increase.

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