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Synthesis and Characterization of Zeolites from Coal Fly Ash Waste

Mita Fitriani[1], Ramlan[1], Yohanes Edi Gunanto[2*], Jan Setiawan[3]

[1] *Physics department, Faculty Mathematics and Natural Science University of Sriwijaya, Indonesia.*

[2] *Dept. of Physics Edu., Faculty of Education, University of Pelita Harapan, Karawaci, Tangerang, Indonesia.*

[3]*Center for Advanced Materials Research (PRMM), National Research and Innovation Agency (BRIN), Indonesia.*

Corresponding Authors E-mail: yohanes.gunanto@uph.edu

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Abstract

South Sumatra had coal production through PT Bukit Asam Tbk, which had coal production since 1950. Tanjung Enim Steam Power Plant (PLTU) is the largest coal ash producer because coal is the primary fuel. Coal combustion by-products include coal fly ash (CFA) and coal bottom ash (CBA). The current utilization of the CFA Tanjung Enim Steam Power Plant is for a cement mixture of PT Semen Baturaja and planting media. This work attempts to optimize zeolite synthesis from CFA by examining the effects of hydrothermal duration on reducing coal waste. This research studies the effect of hydrothermal time with time variations of 5, 12, and 24 on the morphology and phase of zeolite obtained. CFA from the Tanjung Enim Steam Power Plant contains SiO_2 and Al_2O_3, which account for 47.7% and 28.7% of the total composition, respectively. The SEM characterization result shows that the synthesized zeolite forms aggregates with a particle size of about 8-15 µm. Based on XRD characterization of CFA hydrothermal time of 5 hours, the dominant phase is the gibbsite phase, but there is a sodalite phase. The 12-hour hydrothermal time showed the formation of quartz, gibbsite, and sodalite phases. The 24-hour hydrothermal time shows that the dominant phase is sodalite, but there are gibbsite and quartz phases. The peak of the quartz phase decreases the longer the hydrothermal time. In this study, the duration of the hydrothermal process affects the formation of the zeolite phase.

Introduction

Indonesia's abundant natural resources include coal, and it is the world's largest exporter of coal [1]. PT Bukit Asam Tbk, established in 1950, is the main coal producer in Sumatra [2]. Tanjung Enim Steam Power Plant (PLTU) is the largest producer of coal ash generated from coal combustion. Coal combustion by-products include coal fly ash (CFA) and coal bottom ash (CBA). CFA is a fine residue (comprising dust, ash, and soot) generated when coal is burned.

It enters the exhaust gas stream, which includes gaseous by-products, from the furnace to the top. Bottom ash consists of minerals left over from burning coal that settle on the walls and bottom of the boiler [3]. The morphology and dimensions of the constituent particles are the main differences between CBA and CFA. CFA particles are mostly spherical. CBA has irregular shapes [4]. The relatively large coal ash can cause dangerous pollution impacts, and if it is not utilized, it will pollute the environment. Tanjung Enim Steam Power Plant utilizes CFA as a mixture for PT Semen Baturaja cement [2] and planting media [5].

Coal ash must be optimally utilized by turning it into raw material in zeolite synthesis [6–10]. CFA contains many chemical elements. These include alumina, iron oxide, silica, calcium, carbon, magnesium, and sodium [11]. The geographical location of the coal affects its chemical composition, but the same dominant chemical elements are SiO_2 and Al_2O_3. CFA contains crystal structures such as quartz, magnetite, mullite, and hematite [12].

Zeolites are included in aluminosilicate minerals with three-dimensional structure [13–16]. Zeolites can be defined as aluminosilicate compounds made of tetrahedral alumina and silica by bonding oxygen atoms [17]. Zeolite-A, zeolite Na-P1, and zeolite-X are zeolites that from CFA can be synthesized [18]. Zeolites have nanoporous material characteristics and can lose and absorb water in more than 30% of their dry weight [19, 20]. The advantages of synthetic zeolites compared to natural zeolite application relate to their greater stability reaction in the environment [21], larger pore sizes in synthetic zeolite, and larger molecules' absorption [19, 21, 22]. In applications involving removing radioactive contaminants and adsorbing heavy metal ions, synthetic zeolites exhibit removal efficiencies that are several times higher than those of natural zeolites [19, 23].

Zeolites synthesized with CFA can be synthesized using several methods, namely hydrothermal, ultrasonic, and microwave hydrothermal [14]. The hydrothermal method is a widely used method for zeolite production primarily due to its minimal energy consumption, reduced air pollution, and straightforward solubility control [24]. There are various wastes and natural resources that can be used as raw materials for zeolite synthesis, such as industrial waste [25], CFA [12], CBA [26], rice husk ash [27], kaolin [15], and natural zeolite [28].

The methodology used in this research is by reacting sodium aluminate solution and sodium silicate solution. The mixture of solutions that form this gel is then transformed using hydrothermal, which can form zeolite crystals [7, 29]. Hydrothermal time is an essential factor in the formation of zeolite material. Thus, this research's main objective is to analyze hydrothermal time's effect on the characteristics of the synthesized zeolite products formed. Characterization tests include X-ray fluorescence, scanning electron microscopy, and X-ray diffraction.

Experimental Method

The equipment used in this research is a 200 mesh sieve, oven, pH indicator, hydrothermal reactor (stainless steel autoclave), crucible, hot plate, and magnetic stirrer. Characterization tests were used to determine the elemental composition of the raw materials by X-ray fluorescence characterization. Phase identification is done through X-ray diffraction characterization while knowing the morphology of zeolite synthesis through scanning electron microscopy characterization.

The materials used were CFA from PLTU Tanjung Enim, 5 M NaOH, 1 M HCl, $Al(OH)_3$, and demineralized water.

The zeolite synthesis process uses raw materials in the form of CFA from the Tanjung Enim PLTU. It was produced by the hydrothermal method depicted in Figure 1.

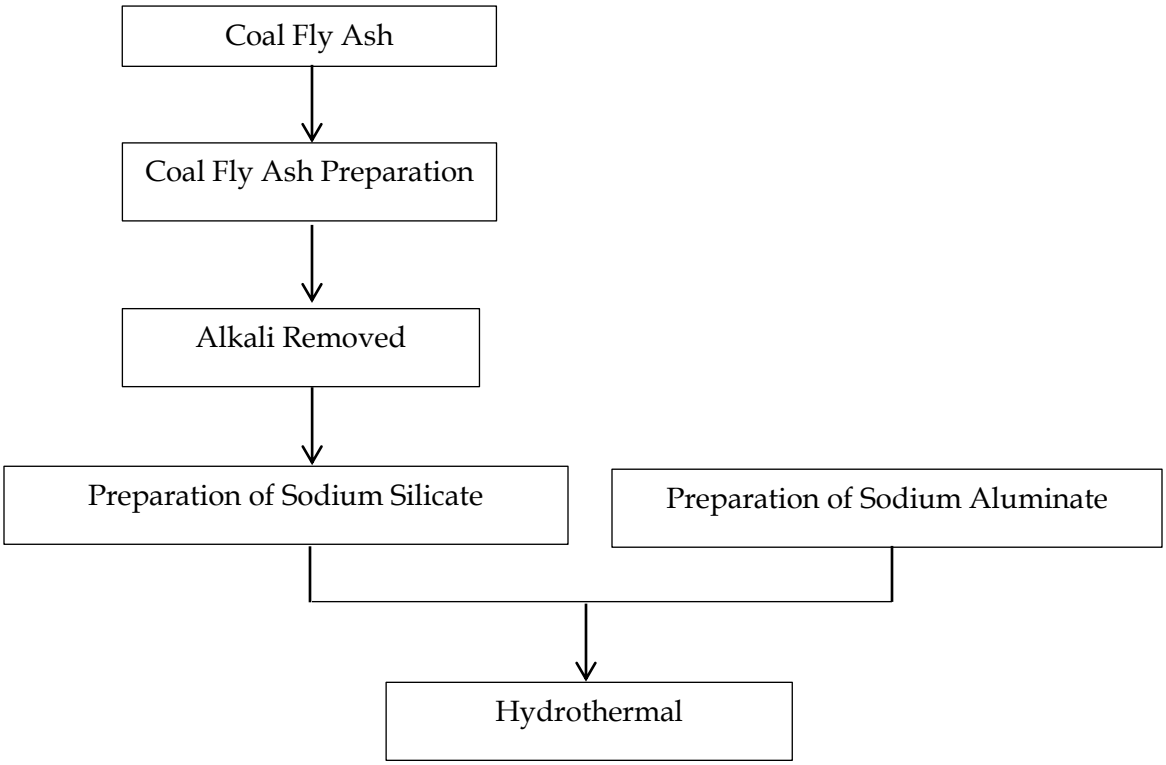

Figure 1. Zeolite synthesis process

Coal Fly Ash Preparation

CFA is filtered using a 200 mesh sieve, which aims to equalize the size and increase the solubility of coal fly ash and can produce CFA with high chemical reaction efficiency [30]. The screening process produced the fly ash, and then a temperature of 100 °C was used to dry the sample. For CFA that has been dried, a sample of 50 grams is taken to be washed with 1 M HCl [11]. The acidified sample was then neutralized with demineralized water and used at 70 °C for 5 hours for drying. The prepared CFA samples were then subjected to X-ray fluorescence (XRF) analysis to determine the differences in the chemical composition of CFA from PLTU Tanjung Enim and coal ash samples leached with 1 M HCl.

Zeolite Synthesis

CFA samples were washed with 1 M HCl and added to NaOH. The mass ratio of NaOH/CFA was 1:1. The mixture was then melted at 550 °C for 1 hour. The objective of utilizing bases is to facilitate the dissolution of the crystalline phase. This process will change some of the crystal phases of CFA to aluminosilicate [12]. In addition, smelting CFA produces a light grey powder,

which indicates that the carbon content in the smelting has disappeared, so the zeolite that will be synthesized has high purity without the element carbon. Mixing CFA and NaOH that has been made will form sodium silicate, and the following reaction equation occurs:

$$SiO_2 + 2NaOH \longrightarrow Na_2SiO_3 + H_2O$$

CFA melted with NaOH is then made into suspension by adding 12 ml/gram of demineralized water and stirring for 2 hours. Supernathan is then made into a slurry during the hydrothermal process. The preparation of sodium aluminate solution is made by reacting $Al(OH)_3$ powder with NaOH, with a molar ratio of Si/Al of 1,24, which is homogenized using a magnetic stirrer at a temperature of 100 °C for 30 minutes until a white solution is obtained. Sodium aluminate is used as a source of Al. The reaction that occurs is as follows:

$$Al(OH)_3 + NaOH \longrightarrow NaAlO_2 + 2H_2O$$

The sodium aluminate solution was mixed with the supernatant from the alkaline fusion in the hydrothermal process. Mixing sodium alumina solution with supernatant from alkali melting for 1 hour and then hydrothermal process for 5, 12, and 24 hours at 100 °C [31]. In the crystallization process of zeolite, the following reactions occurred [11]:

$$Na_2SiO_3 + 2NaAlO_2 \longrightarrow [Na_x(AlO_2)_y(SiO_2).NaOH.H_2O]$$
$$[Na_x(AlO_2)_y(SiO_2).NaOH.H_2O] \longrightarrow Na_{12}[(AlO_2)_{12}(SiO_2)_{12}.27H_2O$$

The hydrothermal process lasted 5, 12, and 24 hours at 100 °C. Then, the sample was filtered, and the residue was dried for 3 hours at 100 °C. In addition, XRD characterization testing was done to ascertain the achieved phase, and SEM testing was done to determine the morphology of the synthesized zeolite findings.

Result and Discussion

This research uses CFA from the Tanjung Enim PLTU. The CFA was first tested to determine the chemical elements in the CFA of PLTU Tanjung Enim; thus, X-ray fluorescence (XRF) characterization was carried out. Determining chemical elements is an important step in zeolite synthesis to calculate the percentage composition of silicon and aluminum, which are essential components in this process [32]. The XRF characterization results of the raw materials are shown in Table 1.

Table 1. Chemical composition of Tanjung Enim PLTU CFA.

Element	WT%	Element	WT%
SiO_2	47.7	K_2O	0.5
Al_2O_3	28.7	TiO_2	1
Fe_2O_3	9	SO_3	0.9
CaO	7.5	P_2O_5	0.8
Cl	-	MgO	4.1
Na_2O	2.1	SO_3	0.9

From the X-ray fluorescence (XRF) results, CFA is known to have a fairly high SiO_2 and Al_2O_3 content, which is 47.7% and 28.7%, respectively. CFA with $SiO_2 + Al_2O_3 + Fe_2O_3$ content ≥ 70 wt% is categorized as F-class fly ash. F-class fly ash is a good precursor for synthesizing zeolites [18].

CFA is given acid treatment with 1 M HCl solution. The results of CFA that have been treated with acid are then washed until neutral and dried. Based on research [11], washing using HCl reduces impurities that are easily soluble in HCl.

$$Fe_2O_3 + 6HCl \longrightarrow 2FeCl_3 + 3H_2O$$

$$Al_2O_3 + 6HCl \longrightarrow 2AlCl_3 + 3H_2O$$

In this study, CFA was washed using strong acid, and 1 M HCl solution was used. The characterization results after washing are presented in Table 2.

Table 2. Chemical Composition of CFA after Acid Washing.

Element	WT%	Element	WT%
SiO_2	45.6	K_2O	-
Al_2O_3	26.8	TiO_2	1.4
Fe_2O_3	6.4	SO_3	-
CaO	6.8	P_2O_5	0.5
Cl	4.4	MgO	2.9
Na_2O	-	SO_3	0.9

XRF characterization shows that CFA has a different chemical element composition before and after washing with 1 M HCl. This washing process aims to remove impurities that are easily soluble in HCl. The amount of iron oxides and alkaline in fly ash is reduced during the washing process with HCl solution [11, 30]. The acid treatment helps to remove impurities from the CFA [33]. Based on the XRF results, acidification has little effect on CFA.

The results of the characterization by scanning electron microscopy (SEM) in Fig. 2 show that the synthesized zeolite forms aggregates with a particle size of about 8-15 µm. XRD characterization aims to identify a material's crystallinity and analyze desired and unwanted phases. The results of the XRD analysis show a diffractogram pattern.

The XRD results were analyzed using the Match! 3 application in determining the phase. Figure 3 displays the XRD pattern of zeolite. At 5 hours of hydrothermal time, a CFA sample found gibbsite and sodalite phases. The sodalite phase was found at 2θ 13.922°, 24.202°, 31.569°, and 34.546°. The gibbsite phase emerged as the dominant phase based on XRD analysis. The hydrothermal time of 12 hours resulted in the formation of quartz, gibbsite, and sodalite phases, as indicated by the XRD pattern of the zeolite. The sodalite phase was found at 2θ 13.879°, 24.267°, 32.069°, 34.416°, 37.871°, and 42.762°. The 24-hour hydrothermal time shows that the dominant phase is sodalite, but there are gibbsite and quartz phases. The sodalite phase was found at 2θ 13.791°; 24.158°; 31.417°; 34.503°; 37.524°; 42.588°; 51.889°; 57.931°. In this study, quartz is always present in the synthetic samples. The peak strength of quartz decreases with increasing hydrothermal time, indicating that long hydrothermal time can increase the conversion of quartz into zeolite crystals. Based on this discussion, the duration of the hydrothermal process affects the formation of zeolite phases [34].

All three zeolite synthesis results yield the sodalite zeolite phase, which can be formed using a Si/Al molar ratio of less than 2 [25]. Sodalite is a material that is widely used as an effective

adsorbent and catalyst support due to several favorable characteristics, including hydrothermal strength, resistance to bases, uniform pore structure, and accessible surface area [9]. This synthesis results from the sodalite phase, as the NaOH concentration significantly influences the CFA hydrothermal reaction. [33].

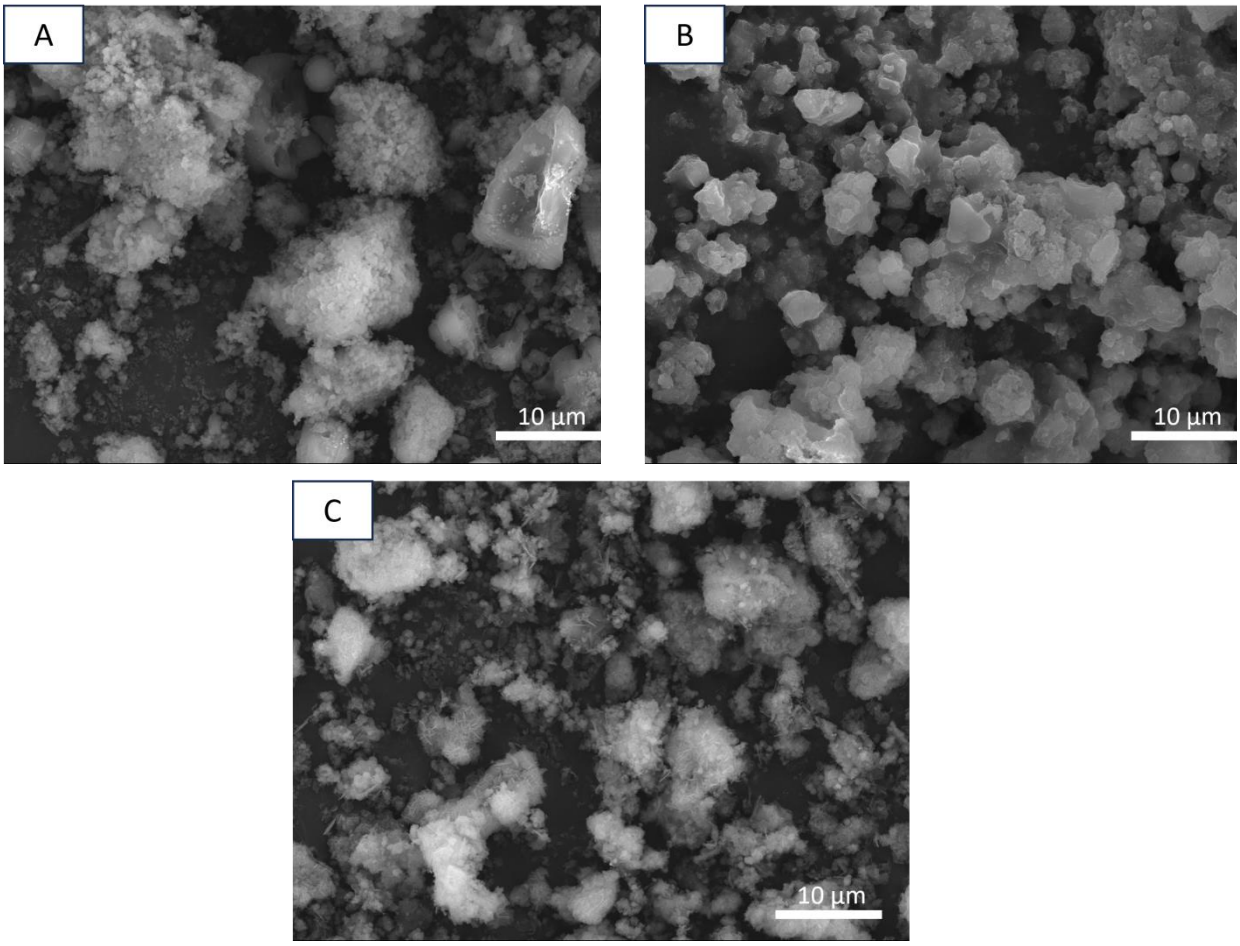

Figure 2. The morphology of synthetic zeolites (a) hydrothermal time of 5 hours, (b) hydrothermal time of 12 hours, (c) hydrothermal time of 24 hours

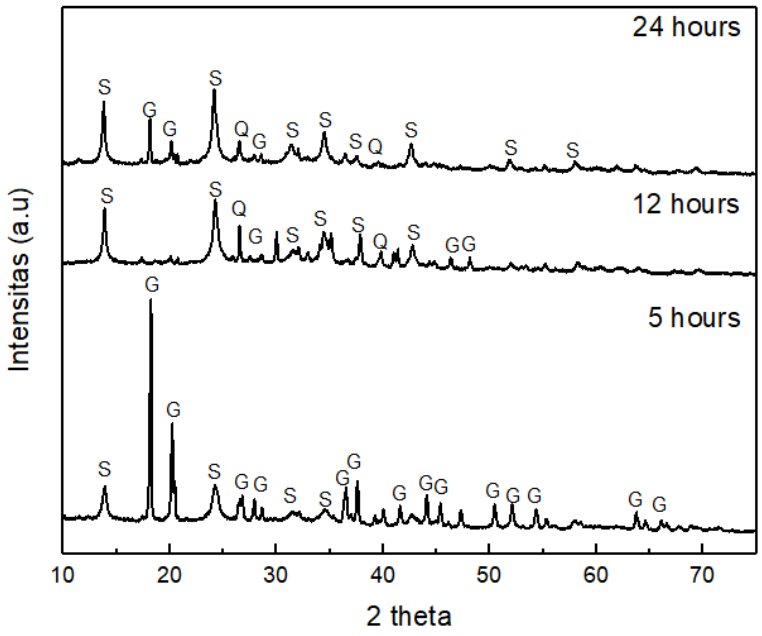

Figure 3. X-ray diffraction patterns of zeolites synthesis; Q = Quartz, S = Sodalite, G = Gibbsite.

Conclusion

Zeolite synthesis was successfully done using the hydrothermal method using raw materials derived from CFA PLTU Tanjung Enim. The results of XRF characterization show that the main components of CFA PLTU Tanjung Enim are SiO_2 and Al_2O_3, so that CFA can be used as raw material for zeolite synthesis. In this study, hydrothermal time affects the formation of zeolite phases. SEM characterization showed that the synthesized zeolite formed aggregates with a crystallite size of about 8–15 µm. XRD data shows that at a hydrothermal time of 5 hours, the dominant phase is the gibbsite phase, but there is a sodalite phase. The 12-hour hydrothermal time shows the formation of quartz, gibbsite, and sodalite phases. The 24-hour hydrothermal time shows that the dominant phase is sodalite, but there are gibbsite and quartz phases. The peak strength of quartz decreased as the hydrothermal time increased, indicating that a long hydrothermal time can increase the conversion of quartz into zeolite crystals.

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