



The Synthesis and Characterization of Pahae Natural Zeolite–Coal Bottom Ash Adsorbent for Fe and Mn Purifier in Well Water

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Abstract

Zeolite is a porous crystalline mineral alumina silica tetrahydrate that has an open three-dimensional framework structure that has channels and cavities filled with metal ions, while Bottom ash is also known to have high porosity and surface area. Thus, both zeolite and bottom ash can be used as adsorbents. This study aims to make an adsorbent based on Pahae natural zeolite with a mixture of bottom ash from coal as an adsorbent for iron (Fe) and manganese (Mn) metals in well water. Adsorbent made from Natural Zeolite Pahae–Coal Bottom Ash with a size of 200 mesh (74 μm) which has been chemically activated with 1 M NaOH solution, was prepared with various compositions of (100:0)%, (95:5)%, (90:10)%, (85:15)%, (80:20)%, (75:25)% respectively, then mold using a hydraulic press with a load of 6 tons of weight for 10 minutes. The mold samples were then physically activated with temperature variations of 600°C, 700°C, and 800°C for 4 hours, respectively. The addition of coal bottom ash filler is known to increase the porosity of the adsorbent with the composition of (75:25)% with an activated temperature of 800°C by 10%. The results of the AAS test showed that the adsorbent made from Natural Zeolite Pahae–Coal Bottom Ash reduced the content of Fe and Mn metals by 92.37% and 53.49% in well water, respectively.

1. Introduction

Water has an important role in life because it is one of the basic needs for every living thing, including humans. The water used daily on earth by people comes from various sources such as rivers, dug wells, or drilled wells. The water obtained from wells comes from groundwater, which has different water quality in each area. Water from these sources usually exceeds the content of iron (Fe) and manganese (Mn), which is unsafe for consumption [1].

Drinking water quality regulation of the Health Minister of the Republic of Indonesia states that water that is safe for consumption must have a threshold limit value of Fe and Mn content of not more than 0.3 mg/L (No.492/MENKES/PER/IV/2010) [2] and 0.05 mg/L (No.416/Menkes/Per/IX/1990) [3] respectively. In addition to ensuring water safety for drinking, the high content of Fe and Mn in well water can result in an

unpleasant taste and odor, rendering it unsuitable for daily activities such as bathing and washing [4]. Thus, further treatment for well water must be done to meet the requirements to be declared safe for consumption and use in daily human activities. Further treatment in the form of reducing the levels of Fe and Mn contained in well water can be carried out through adsorption processes or water filtration [5].

Natural zeolite is one of the many natural materials with great attention as an adsorption material. This zeolite is widely used as an adsorbent to reduce water pollution caused by impurity compounds [6]. Natural zeolite is a mineral material that is quite common in Indonesia. Zeolite is a porous crystalline mineral alumina silica tetrahydrate, which has a three-dimensional framework structure, formed by tetrahedral $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ interconnected by oxygen atoms, forming an open three-dimensional framework that has channels

and cavities filled with metal ions (usually alkali metals or alkaline earth metals) and water molecules that can move freely [7]. Zeolites can also be used as ion exchangers in brackish water with the help of resins in removing iron, chloride, and Total Dissolved Solid (TDS) levels [8].

In previous studies, using zeolite as a groundwater adsorbent was reported to be able to adsorb Fe and Mn metals up to 98% and 97%, respectively [5]. Other studies also reported that mixing clay, zeolite, and coconut shell-based activated carbon adsorbent can adsorb up to 99.6% of the Fe from mine water [9]. However, natural zeolite has several weaknesses, such as containing many impurities such as Na^+ , K^+ , Ca^{2+} , Mg^{2+} , and Fe^{3+} ; thus, it will reduce the activity of natural zeolite [10]. To improve the properties of natural zeolites used as adsorbents, catalysts, or other applications, initially activating or modifying them may increase their activities [11].

This study used bottom ash derived from coal combustion to optimize zeolite as an adsorbent material. Bottom ash has high porosity and surface area characteristics, making it a good choice for application as an adsorbent [12]. In addition, bottom ash has a sizable content of Si and Al, conferring properties akin to those found in zeolites and other adsorbent materials [13]. Until now, the utilization of bottom ash, a byproduct of the coal industry, has been relatively minimal. Thus, the potential for using bottom ash for various applications, including adsorbent materials, is still very large [14]. In this study, bottom ash will be used as a raw material for synthetic zeolites used in the adsorption and filtration of heavy metals. Previous studies reported that using adsorbents from the sintering process of coal bottom ash reduced the Cd and Pb by 98.16% and 98.41% in soil, respectively [12].

The novel Pahae natural zeolite-Coal Bottom Ash adsorbent was synthesized and characterized to determine its potential as an adsorbent for Fe and Mn metals in well water. As a result, it was found that the novel Pahae Natural Zeolite-Coal Bottom Ash adsorbent can effectively reduce the Fe and Mn composition in the well water.

2. Experimental

2.1. Materials

The natural zeolite of Pahae was obtained from Tapanuli Utara, North Sumatra, Indonesia. Bottom ash was obtained from the combustion of PLTU Pangkalan Susu, North Sumatra. Sulfuric acid 96% was purchased from Mallinckrodt Baker, Paris, KY. Bioethanol with a concentration of 96% was purchased from Merck, Darmstadt, Germany.

2.2. Pahae Natural Zeolite Treatment

The zeolite in lumps was first crushed using a mortar, then sieved with a 200 mesh (74 μm) sieve. The obtained zeolite particles were washed with distilled water three times to remove impurities and dried at 100°C in an oven for 1 hour. The zeolite was then chemically activated in 1 M NaOH solution while stirring using a magnetic stirrer with a rotation speed of 120 rpm at 60°C for 1 hour. The activated zeolite was then filtered, washed

with distilled water until the pH was neutral, and dried at 100°C in an oven for 1 hour [15].

2.3. Coal Bottom Ash Activation

Coal Bottom Ash was dried under the sunlight for a day. Furthermore, the coal bottom ash was sieved using a 200 mesh (74 μm) sieve. Then, the coal bottom ash was activated with 1 M NaOH solution at 85°C for 2 hours while stirring using a magnetic stirrer with a rotational speed of 120 rpm. The coal bottom ash was then filtered using filter paper, washed with distilled water until the pH became neutral, and dried at 100°C using an oven for 1 hour [16].

2.4. Manufacture of Pahae Natural Zeolite-Coal Bottom Ash Adsorbent

A 10 g Pahae Natural Zeolite-Coal Bottom Ash with various compositions of (100:0)%, (95:5)%, (90:10)%, (85:15)%, (80:20)%, (75: 25)%, respectively, were each added into a mold with size (3 x 3 x 1) cm. The printing process was then carried out using a hydraulic press machine for 10 minutes with a given mass of 6 tons. The molded Pahae Natural Zeolite-Coal Bottom Ash samples were left in an open space for a week to prevent cracking when the solid was heated. The samples were then activated with temperature variations of 600°C, 700°C, and 800°C for 4 hours. Furthermore, the Pahae Natural Zeolite-Coal Bottom Ash was stored until further use [15].

2.5. Effectiveness Test of Pahae Natural Zeolite-Coal Bottom Ash in Well Water

Absorber (Pahae Natural Zeolite-Coal Bottom Ash with a composition of (75:25)% activated at 600°C) was first placed into a 250 mL beaker glass and then filled with 100 mL well water containing Fe and Mn metals. The Fe and Mn levels of the well water before and after being treated with adsorbent were then analyzed with Atomic Absorption Spectroscopy (AAS).

2.6. Characterizations

The porosity of the material was tested per ASTM C 642-90. Equation (1) was used to calculate the material porosity. The water absorption capacity of the material was then tested, referring to Indonesian National Standard (SNI) No. 03-0349, by soaking the sample for 24 hours to analyze the ability of the sample to adsorb water. The results obtained were then calculated using Equation (2). Hardness Tester Matsuzawa Seiki (Japan) was used to measure the hardness value of the sample. The test was carried out by giving the sample a mass of 1 kg and a holding period for 30 seconds. The sample hardness value was then calculated using Equation (3).

A Philips PW 1050 X-ray diffractometer (XRD) was used to measure the phase crystallinity of the sample. The test was conducted with a 2 θ scan process from 7° to 70°. Scanning electron microscopy- Energy dispersive X-ray (SEM-EDX), JEOL JSM6390, Oxford Instruments, was used to observe the morphology and elemental composition of the adsorbents. XRF Spectrometer XGT-5200 was used to determine the chemical composition and $[\text{SiO}_2]/[\text{Al}_2\text{O}_3]$ ratio of the samples. THERMO Scientific iCE™ 3500 Atomic Absorption Spectrometer (AAS) was used to measure the content of

Fe and Mn in the well water before and after the adsorption process with Pahae Natural Zeolite-Coal Bottom Ash. The percentage of results of reduced levels of Fe and Mn was then calculated using Equation (4).

$$\text{Porosity (\%)} = \frac{(\text{final mass} - \text{initial mass})}{\text{density of water} \times \text{water volume}} \times 100 \quad (1)$$

$$\text{Water absorption (\%)} = \frac{(\text{final mass} - \text{initial mass})}{(\text{initial mass})} \times 100 \quad (2)$$

$$Hv = 1.8544 \frac{F}{d^2} \quad (3)$$

$$\text{Fe/Mn (\%)} = \frac{(\text{initial concentration} - \text{final concentration})}{(\text{final concentration})} \times 100 \quad (4)$$

3. Results and Discussion

3.1. Porosity Test Analysis

One of the physical properties that significantly affect the performance of the adsorbent is porosity [17]. In this case, the graph of the test results for the porosity of the Pahae Natural Zeolite-Coal Bottom Ash adsorbent material is shown in Figure 1.

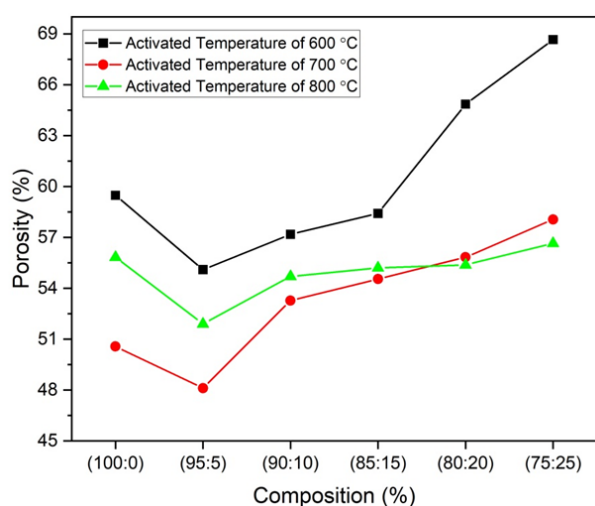


Figure 1. Graph of porosity test results of the adsorbents

Figure 1 shows the highest adsorbent porosity value of 68.6% in the Pahae Natural Zeolite-Coal Bottom Ash adsorbent with a (75:25)% composition activated at 600°C. The adsorbent with the smallest percentage of porosity was found in a Pahae Natural Zeolite-Coal Bottom Ash adsorbent with a composition of (95:5)%, which was activated at 700°C and only had a porosity value of 48.11%. This result indicates that the physical activation temperature and composition of Pahae Natural Zeolite-Coal Bottom Ash adsorbent in the sample affect the adsorbent’s porosity value. Figure 1 shows that the samples activated at 600°C had a higher porosity value than those activated at 700°C and 800°C. This is due to the different sintering temperatures of the two adsorbents, so the increase in temperature cannot be interpreted as an increase in the porosity of the adsorbents [18]. As known, zeolite possesses a sintering temperature of around 200–320°C, while coal bottom ash has several temperature variations in the range of 1000–1200°C [15, 16].

3.2. Water Absorption Test Analysis

The graph of the water adsorption test result of the sample is shown in Figure 2. Pahae Natural Zeolite-Coal Bottom Ash adsorbent with a composition of (75:25)%, which activated at a temperature of 600°C, was found to possess the highest water absorption properties of 42.52%, while the samples with a composition of (95:5)% appear to have the lowest water absorption of 30.99% at an activation temperature of 800°C. This study observed that the adsorbent’s ability to absorb water is strongly affected by the temperature in the activation process [16]. This is consistent with the heating procedure (physical activation) that decreased the number of pores on the surface of the adsorbent [18]. This pore formation allows water to be entangled in the adsorbent; thus, the highest water absorption value is obtained in the adsorbent activated at 600°C.

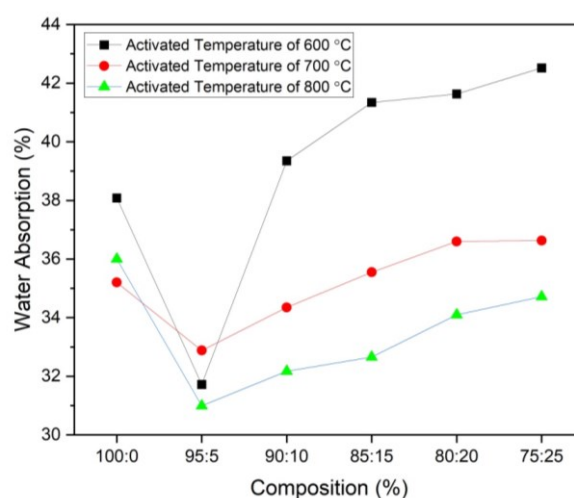


Figure 2. Graph of water absorption test results of the adsorbents

3.3. Hardness Test Analysis

The graph of the hardness test results on the Pahae Natural Zeolite-Coal Bottom Ash adsorbent is shown in Figure 3. In Figure 3, it can be seen that the Pahae Natural Zeolite-Coal Bottom Ash adsorbent samples with a composition of (75:25)%, which were activated at 800°C, 700°C, and 600°C had a hardness value of 150.1734 MPa, 138.693 MPa, and 125.938 MPa, respectively. This data shows that the adsorbent with the highest hardness value is the sample with a composition of (75:25)% activated at 800°C. The decreasing hardness value in the adsorbent activated at a temperature of 600°C is due to the larger pores found in the adsorbent, which implies the reduced adsorbent hardness value [15]. In addition, the factors affecting the nonlinearity of the hardness values for all sample compositions are the sintering temperatures of zeolite and coal bottom ash, which are quite different.

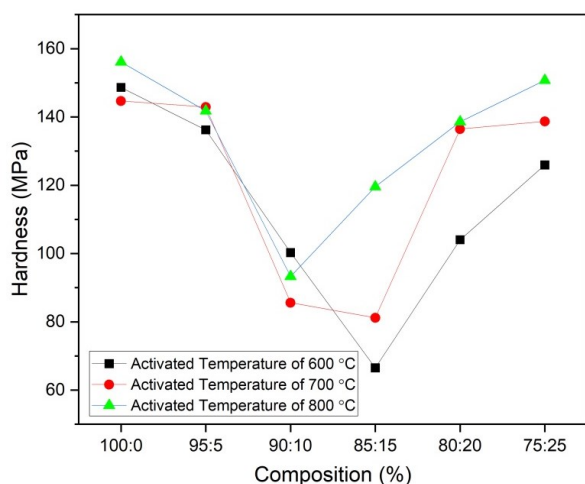


Figure 3. Graph of adsorbent hardness testing results

3.4. Crystal Structure Analysis

X-ray diffraction (XRD) test was carried out to determine the crystal structure formed on the Pahae Natural Zeolite-Coal Bottom Ash adsorbent, and the result is displayed in Figure 4. In this study, the XRD test was only applied to the Pahae Natural Zeolite-Coal Bottom Ash adsorbent with a variation composition of (75:25)%, which activated at a temperature of 600°C due to the highest porosity and water absorption value of this sample.

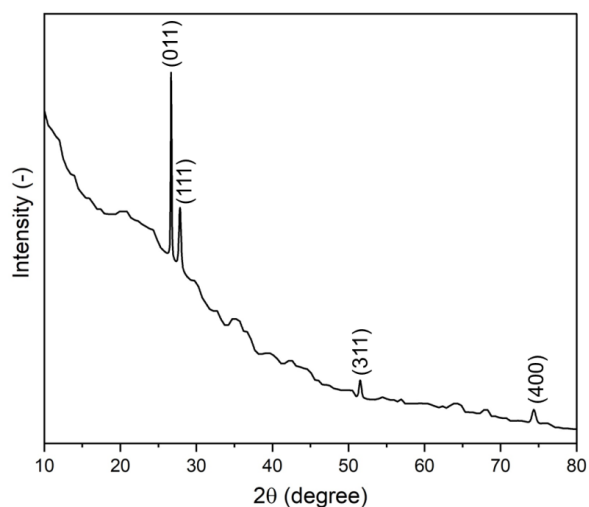


Figure 4. Graph of XRD test results of the adsorbent with a composition of (75:25)%, which activated at 60°C

The highest peak intensity in the sample is indicated by the silica (Si) peak with a hexagonal crystal structure. This hexagonal crystal structure is produced from crystallized trigonal silica (SiO₂) with six equal sides, having two angle values, namely 90° and 120° ($\alpha = \beta = 90^\circ$ dan $\gamma = 120^\circ$), while the edge length is $b \neq c$ [19, 20]. Thus, the presence of silica in the sample significantly affects the optimization of the sample application as an adsorbent.

3.5. Morphological Analysis

Morphological and pore diameter tests on the Pahae Natural Zeolite-Coal Bottom Ash sample with a

composition of (75:25)% at 600°C are shown in Figure 5. The morphological test was only carried out on this sample because this sample is the sample with the most optimal variation based on the results of the porosity, water absorption, and hardness tests. Morphology images show that the mixing of the two materials is homogeneous; this can be seen from particles in the form of still visible lumps. This can happen because the average pore diameter of the Pahae Natural Zeolite-Coal Bottom Ash sample with composition (75:25)% at 600°C is 1.9 μm. However, it can be seen that the pores are not uniformly distributed.

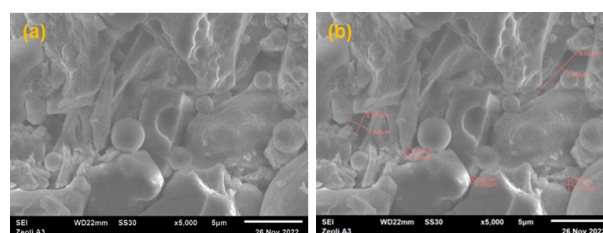


Figure 5. SEM images of (a) Pahae Natural Zeolite-Coal Bottom Ash adsorbent with a composition of (75:25)%, which activated at 600°C, and (b) pore diameter measurements

3.6. Analysis of The Elemental Content of The Adsorbent Using XRF

XRF testing (Table 1) was carried out to determine the elemental content of Pahae Natural Zeolite-Coal Bottom Ash. Based on previous research, it is known that the dominant elements contained in zeolite are silica (Si) and alumina (Al). This is because zeolite comprises silica and alumina crystals bonded by oxygen atoms, creating a porous structure [6]. Due to the pozzolanic nature of coal bottom ash, which refers to materials with silica components, most elements found in coal bottom ash are SiO₂, Al₂O₃, Fe₂O₃, and CaO [21]. As a result, Table 1 shows that the adsorbent contains a Si element of 39.1%. Furthermore, by testing the elemental content, it can be seen that the ratio of Si/Al in the adsorbent is 3.80; thus, it can be categorized as an adsorbent made from natural zeolite with an average Si/Al ratio, which has hydrophilic or water-loving properties [22].

Table 1. XRF test results of Pahae Natural Zeolite-Coal Bottom Ash with a composition of (75:25)%, which activated at 600°C

Element	Weight (%)
Al	10.26
Si	39.08
K	12.10
Ca	13.11
P	2.65
Ti	2.20
Fe	18.11

3.7. Effectiveness analysis of the application of the adsorbent as Fe and Mn Metals Absorbent

The AAS test was carried out to determine the effectiveness of using Pahae Natural Zeolite-Coal Bottom Ash adsorbent with a composition of (75:25)% at 600°C in minimizing the content of the metal elements Fe and Mn

in well water. The results of this test are shown in Table 2. The data in Table 2 shows that the content of Fe and Mn metals in well water has decreased by 92.4% and 53.5%, respectively. This occurs due to the physical adsorption process of well water on adsorbent caused by intermolecular forces on the surface that are not tightly bound [23]. This physical adsorption is based on Van der Waals forces, which allow an adsorbate to travel from one surface to another by attracting or repelling one another between relatively weak adsorbate molecules [24]. This is in accordance with the nature of the zeolite, which acts as an ion exchanger in the adsorbent [25]. Moreover, developing a hierarchical mesopore system in zeolite allows more numerous cations of heavy metal to penetrate, increasing the removal rate and capacity of cations in wastewater [26]. Based on the resulting data, it was observed that the adsorption treatment using Pahae Natural Zeolite-Coal Bottom Ash reduced the content of heavy metals Fe and Mn in well water. The well water treated with Pahae Natural Zeolite-Coal Bottom Ash is then declared safe for consumption since the Fe and Mn compositions in the treated well water are below the threshold set by the Indonesian National Standard (SNI), as shown in Table 2.

Table 2. AAS test results on well water before and after being treated with Pahae Natural Zeolite-Coal Bottom Ash with a composition of (75:25)%, which activated at 600°C

Parameter	Unit	Threshold Limit Value	Before Treatment Value	After Treatment Value	Standard Method
Fe	mg/L	0.3	1.597	0.156	SNI 6989.4:2009
Mn	mg/L	0.05	0.527	0.083	SNI 6989.5:2004

4. Conclusion

Pahae Pahae Natural Zeolite-Coal Bottom Ash adsorbent was successfully synthesized. The addition of coal bottom ash filler mixed with Pahae natural zeolite can increase water absorption and porosity values so that the optimal composition is obtained in the manufacture of Pahae Natural Zeolite-Coal Bottom Ash adsorbent (75:25)% with an activation temperature of 600°C. The percentage of porosity, water absorption, and hardness values in samples with this composition are respectively 68.66%, 42.52%, and 125.94 MPa. Morphological tests showed that this adsorbent has an average pore diameter of 1.9528 μm with a Si/Al content ratio of 3.80 based on the results of the XRF test. The XRD test results showed that the adsorbent has a hexagonal crystal structure. This adsorbent is also proven to reduce the content of Fe and Mn in well water with a reduction percentage of 92.37% and 53.49%, respectively. Thus, it can be concluded that the adsorption treatment using Pahae Natural Zeolite-Coal Bottom Ash adsorbent successfully reduced the composition of heavy metals (Fe and Mn) in well water. As a result, the treated well water became safe for consumption, according to the SNI. However, further studies regarding the porosity of the adsorbent using Brunauer-Emmett-Teller (BET) and analysis using SEM

and FTIR on the adsorbent before and after adsorption in more depth need to be carried out in the future to support this finding.

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