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Tutik Murniasih; Galih Olga Rakha Siwi; Bustanussalam; Tri Aryono Hadi

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Utilization of Activated Carbon Composite Adsorbent and Zeolite in Acid Mine Drainage Treatment

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Abstract. Acid Mine Drainage (AMD) is generated from mining activities, one of which is coal mining. The process of AMD formation is caused by the oxidation of sulfide minerals containing sulfur accompanied by the presence of water. The content of heavy metals in AMD such as Fe and Mn in high concentrations can have a serious impact on the environment and the safety of living things. To overcome this impact, treatment is necessary. This treatment aims to optimize the discharge of AMD to the environment by meeting quality standards in accordance with established environmental regulations. The procedure used in this treatment is done by compositing coal activated carbon and zeolite synthesis, then mixing it into 200 ml of artificial AMD at 5 variations of different initial concentrations of Fe and Mn metals with 2 grams of activated carbon and zeolite composite for 90 minutes. In the processing using activated carbon and zeolite composite, the largest concentration reduction was obtained at the initial concentration of Fe metal 9.11 mg/L to 0.17 mg/L with 98.13% adsorption of Fe metal and the initial concentration of Mn metal 8.74 mg/L to 0.0001 mg/L with 99.99% adsorption of Mn metal. This shows that Fe and Mn metals in artificial AMD are adsorbed into the pores of the composite in large quantities, so as to reduce the concentration of Fe and Mn metals. The higher the initial concentration value, the lower the percent adsorption and adsorption ability of activated carbon.

INTRODUCTION

The process of coal mining, which inves the removal of overlying soil and rock, extraction of coal, and subsequent washing of the material, has the potential to generate acid mine drainage (AMD). The presence of water and oxygen facilitates the oxidation of sulfide minerals into sulfuric acid, resulting in the formation of AMD with a pH range of 2 to 8. (Skousen, 2016 ; Muliwa et al., 2018). In addition to possessing a low pH, AMD is characterized by the presence of hazardous heavy metal ions, including but not limited to iron (Fe), manganese (Mn), cadmium (Cd), cobalt (Co), zinc (Zn), and nickel (Ni). These heavy metal ions are introduced into AMD through the interaction between AMD and diverse mineral ores [3]-[5]. The presence of heavy metals, specifically iron (Fe) and manganese (Mn), within acid mine drainage can exert significant ecological consequences on the ecosystem and biota in the vicinity. The Decree of the Minister of Environment 113 of 2003 has regulated the concentration of heavy metals, notably iron (Fe) and manganese (Mn).. According to this decree, the permissible pH range is between 6 and 9. Additionally, the maximum allowable concentrations of Fe and Mn have been established at 7 mg/L and 4 mg/L, respectively. The direct disposal of acid mine drainage into the environment can result in water pollution,

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corrosion of mining equipment, and disruption of the equilibrium in aquatic ecosystems. Therefore, it is imperative to treat AMD prior to its discharge into the environment. [6].

One approach aimed at enhancing the quality of AMD inves the utilization of adsorbents, such as fly ash (Sahoo et al., 2013; Kalombe et al., 2020) and activated carbon [9] to facilitate the adsorption process of heavy metals. Extensive investigations have been conducted to explore the efficacy of activated carbon as an adsorbent for acid mine drainage, specifically as a means of adsorbing sulfate [10] and heavy metal ions [11]. Nevertheless, the adsorption of metal ions onto activated carbon often yields a relatively low absorption value for Mn ions in comparison to other metal ions [9], [12], [13]. Hence, it is imperative to do additional study in order to enhance the assimilation of Mn ions and other ions with relatively poor absorption rates. One potential approach inves the alteration of activated carbon, specifically by the production of composites consisting of activated carbon and zeolite. The utilization of zeolite as an adsorbent exhibits considerable potential in enhancing environmental quality subsequent to mining operations. Zeolite possesses voids that can accommodate free water molecules, rendering it a viable adsorbent for wastewater treatment purposes [14]. Research findings indicate that the utilization of activated carbon-zeolite composites leads to a greater capacity for absorbing metal ions in comparison to unaltered activated carbon and zeolite materials [15]. Extensive investigation has been conducted on the synthesis of composites comprising zeolite and activated carbon, wherein peanut shells have been employed as the main component for activated carbon production [16]. Nevertheless, the limited accessibility of raw materials poses a constraint on the large-scale development of this adsorbent. Conducting this research is of significant importance in order to acquire alternate sources of raw materials for the production of activated carbon generated from low to medium rank coal. Despite the enormous reserves of such coal, its low calorific value renders it less economically viable as an energy source [17]-[20].

METHODS

Materials

The materials used include sodium aluminate, sodium silicate, aquadest, coal, sodium thiosulfate, iodine solution, and the standard solution of Fe dan Mn. The equipment used is an autoclave, drying oven, muffle furnace, desiccator, and the bomb oxygen calorimeter.

Synthesis and Characterization of Activated Carbon and Zeolite Composite

Coal samples was taken form PT Batubara Bukit Asam Tanjung Enim Sumatera Selatan, Indonesia. Activated carbon was produced by utilizing coal samples. Before utilization, the coal underwent a process of size reduction and sieving to achieve a particle size of 60 mesh. Subsequently, it was subjected to a drying period of 1 hour at a temperature of 105○C within a dry oven. The coal samples underwent carbonization at a temperature of 600○C for a duration of 1 hour, with a continuous flow of nitrogen in the muffle furnace. In addition, the carbonized activated carbon is subsequently subjected to a drying process. The process of zeolite synthesis commences with the combination of sodium silicate, sodium aluminate, and distilled water, followed by agitation for a duration of 2 hours utilizing a magnetic stir bar. Subsequently, subject the specimen to autoclaving for a duration of 48 hours at a temperature of 80○C within a desiccated oven. Subsequently, rinse with distilled water and continue filtering until the pH reaches a neutral state. The composite of activated carbon and zeolite was combined in a 1:1 proportion. The composite was thoroughly combined and subjected to continuous stirring for an extended period of time, namely overnight. Subsequently, subject the specimen to thermal treatment by placing it in an oven set at a temperature of 80○C, followed by a subsequent transfer to an autoclave for a duration of 24 hours at a temperature of 170○C.

The characterization of the composite material was conducted utilizing the Fourier Transform-Infrared Spectroscopy (FT-IR) equipment, and the iodine number analysis. The iodine number method (ASTM D4607-94) was used to calculate adsorption capacity. The quantity of iodine that could be adsorb was calculated using activated carbon and zeolite composite that had been roasted in an oven, weighted up to 0.5 g, and placed in an Erlenmeyer. The sample was treated with a 50 mL iodine 0.1 N solution, which was then kept on for 15 minutes while being shaken. The filtrate is then collected, and 10 mL of it is titrated with a 0.1 N $Na_2S_2O_3$ solution. The calculation of the iodine number was performed utilizing Equation (1).

$$Iodine\ number\ (mg/g) = \frac{\{(V_1N_1 - V_2N_2)\times 126.9\times 5\}}{W} \quad (1)$$

Along with the normalcy of the iodine (N1), the weight of the activated carbon (W), The thiosulfate solution (V2) that is necessary, the normality of the sodium thiosulfate (N2), and the iodine solution (V1) that is being examined..

Preparation of Artificial AMD

The preparation of artificial acid mine drainage was carried out by diluting 1000 ppm Fe and Mn standard solutions. The results of the dilution of the Fe and Mn solutions were made into 4 concentration variations for artificial acid mine drainage. Preparation of 4 variations of these concentrations by mixing Fe and Mn standard solutions and dilution with aquades. After that, Atomic absorption The initial concentration of iron (Fe) and manganese (Mn) was determined through the utilization of spectrophotometry (AAS) analysis.. The results of the dilution of the Fe and Mn solutions were made into 4 concentration variations for artificial acid mine drainage. Both solutions were diluted according to the desired concentration using formula in Equation (2).

$$V_1.M_1 = V_2.M_2 \quad (2)$$

where V_1 and M_1 are the initial volume and initial concentration of Fe or Mn of the standard solution, V_2 and M_2 are the final volume and final concentration of Fe and Mn after dilution

Activated Carbon and Zeolite Composite Adsorption Test

A total of 2 gram of activated carbon-zeolite composite with 200 ml of artificial mine acid water into 5 variations of Fe and Mn metal concentrations using a sample bottle. The mixture was then put into the shaking incubator at 150 rpm at room temperature for 90 minutes. Filter and collect the filtrate for testing using AAS a to determine the concentration of Fe and Mn metals.

RESULTS AND DISCUSSION

Adsorbent Characterization

The quality assessment of raw material coal inves doing proximate analysis and determining its calorific value. Furthermore, a study of the proximate analysis and determination of the iodine number of the resulting adsorbent was also conducted. The findings of the analysis conducted on the raw material coal and the subsequent adsorbent are presented in Table 1. The ranking of coal can be determined by analyzing its proximate properties, such as moisture content, ash content, volatile matter, fixed carbon content, total sulfur content, and calorific value. According to the data acquired, the coal being examined is categorized as sub-bituminous coal. Indonesia possesses a substantial abundance of coal reserves of this particular sort. Hence, the application of this particular coal variant as activated carbon exhibits significant potential and may be sustained over an extended period [21].

The estimation of the surface area of activated carbon at ambient temperature is accomplished through the determination of the iodine number. The adsorptive capacity and porosity of an adsorbent can be elucidated by the iodine number of the adsorbent. The high iodine number is indicative of the dimensions of the micropore structure and the extent of the surface area possessed by an adsorbent [22]. The present investigation yielded an adsorbent material exhibiting an iodine number of 728.45 mg/g. This finding aligns with previous research outcomes, which have reported iodine number values ranging from 400 mg/g to 800 mg/g for activated carbon materials [23]–[25]. Nevertheless, the previously mentioned figure falls short of the necessary magnitude, specifically 750 mg/g. The previous number suggests that the adsorption capacity is insufficient to meet the quality standards outlined in the SNI 06-3730-1995 for activated carbon [26].

TABLE 1. Results of analysis of coal and activated carbon-zeolite composite

Analysis Parameter	Coal	Activated Carbon and Zeolite Composite	Unit
Moisture	14,47	9,05	%
Ash	1,16	1,95	%
Volatile Matter	41,72	29,20	%
Fixed Carbon	42,64	59,79	%
Total Sulphur	0,095	-	%
Calorific Value	5392,5	-	Cal/g
Iodine Number	310	728,45	Mg/g

The fundamental theory of Fourier-transform infrared (FT-IR) spectroscopy is based on the phenomenon of molecular adsorption of certain wavelengths of infrared radiation. This adsorption leads to changes in the vibrational and rotational energy levels of the molecules. By utilizing FT-IR spectroscopy, it is possible to acquire molecular structure information through the examination of distinct chemical bonds and functional groups [27]. The observed peaks observed at around 1600 cm−1 in activated carbon can be attributed to the bending vibration of the O-H bond. The bands detected in the wavenumber range of 700 cmℒ1 to 580 cmℒ1 can be ascribed to the symmetric stretching vibrations, which are precisely linked to the structural units of SiO4 or AlO4. The deformation vibration of the Al–O–Si group is responsible for the spectral peak found at approximately 520 cmℒ1, as reported in reference [28].

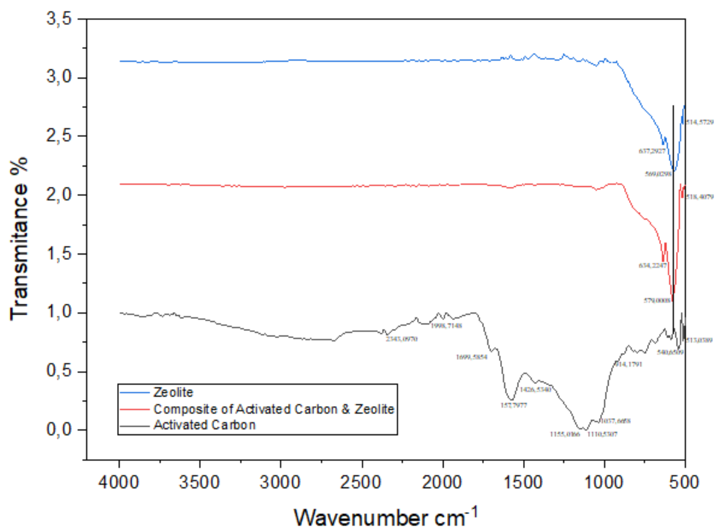

FIGURE 1. FT-IR spectra of activated carbon, zeolite, and composite

Fe and Mn Adsorption Analysis

The simulated acid mine drainage that had been created was subjected to analysis using atomic adsorption spectroscopy (AAS) in order to determine the initial concentrations of iron (Fe) and manganese (Mn) metals. The analysis was performed by employing activated carbon and zeolite composites, both prior to and subsequent to the adsorption treatment.. Based on the provided data, it is possible to calculate and assess the adsorption of each metal, taking into consideration variations in the initial concentrations of Fe and Mn metals. Table 2 displays the results pertaining to the initial concentrations of iron (Fe) and manganese (Mn) metals.

TABLE 2. The concentration of iron (Fe) and manganese (Mn) metals prior to and following treatment.

Concentration of Fe		Concentration of Mn	
Before treatment (mg/L)	**After treatment (mg/L)**	**Before treatment (mg/L)**	**After treatment (mg/L)**
9,11	0,17	8,74	0,0001
18,87	0,18	11,04	0,0026
28,25	0,55	15,22	0,6736
38,11	14,42	18,76	9,2233

Figure 2 displays the adsorption outcomes of Fe and Mn metals at different beginning concentration conditions. The results suggest that the adsorption of iron (Fe) and manganese (Mn) metals exhibits optimal efficiency at low beginning concentrations, but diminishes as the initial concentrations of Fe and Mn metals increase. The observed phenomena can be attributed to the inverse correlation between the initial concentration of metal and the presence of unoccupied binding sites on the adsorbent material. If the initial concentration of metal is low, the adsorbent exhibits a higher abundance of unoccupied binding sites, hence facilitating a more efficient adsorption of metals, ultimately achieving a maximum uptake of 98.13-99.99% [29]. At elevated initial metal concentrations, a significant portion of the adsorption sites get filled by metal molecules, resulting in a considerable quantity of metal remaining in the solution. Consequently, the proportion of metal adsorbed experiences a decrease [30].

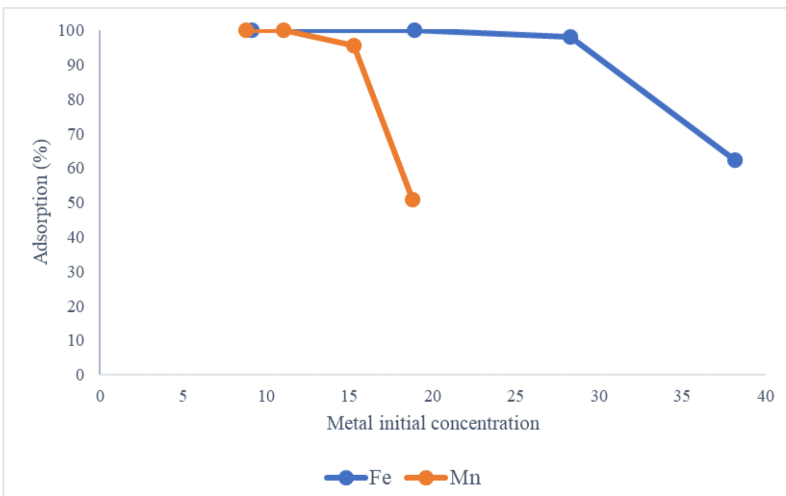

FIGURE 2. Variation of Fe and Mn metal concentration to the percentage pf metal adsorption

CONCLUSION

The findings of the study indicate that the composite material consisting of activated carbon and zeolite exhibits potential as an adsorbent for the treatment of AMD. In experiments using artificial AMD with 5 variations of initial concentrations, the activated carbon-zeolite composite could reduce the concentration of Fe metal with an initial metal concentration of 9.11–38.11 mg/L to 0.17–14.42 mg/L, the highest level of Fe metal adsorption was seen at an initial concentration of 9.11, resulting in an adsorption rate of 98.13%. The activated carbon and zeolite composite can also reduce the concentration of Mn metal from an initial concentration of 8.74–18.76 mg/L to 0.0001–9.223 mg/L, the highest level of Mn metal adsorption is observed at an initial concentration of 8.74 mg/L, resulting in an adsorption rate of 99.99%. The greater the initial concentration of Fe and Mn in AMD, the less adsorption there is. The study's findings indicate that the activated carbon and zeolite composite exhibits significant promise as an adsorbent for AMD waste treatment..

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