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Adsorption-Based Peat Water Treatment Using Modified Red Mud

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Abstract

Red mud is an alumina refinery waste residue that can be developed as an effective adsorbent for wastewater treatment, one of which being peat water. Peat water treatment can be carried out through adsorption process employing treated red mud as the adsorbent. This study sought to ascertain the impact of acid types (HCl and H_2SO_4) on the dealumination of red mud and characteristics of the resulting adsorbent evaluated using XRF and BET analyses. This research was conducted in a laboratory scale employing various adsorbent mass (1 g, 3 g, 5 g, and 7 g) and adsorption time (15, 30, 60, and 90 minutes) on the performance of peat water treatment. The results showed an increase in the Si/Al ratio from 1.064% to 1.697% and 1.064% to 1.565% for HCl and H_2SO_4, respectively. Meanwhile, the surface area of the adsorbent also increased by 179.574% and 162.891% when HCl and H_2SO_4 solutions were used as red mud's modifying agents. The results of research on the adsorption process with variations in the mass of the adsorbent 1, 3, 5, and 7 grams obtained the results of peat water pH respectively were 6.6, 7.0, 7.0, and 7.1. In the Fe metal there were removal of 73.39%; 37.09%; 24.19% and -141.93%. Accordingly, the organic matter removal efficiencies were 53.07%, 50%, 44.30% and -17.98%, respectively. In variation of adsorption time for the parameter Fe, there were removals of 29.03%, 27.41%, and 24.19% respectively. Then for organic matter the removal efficiencies were 32.46%, 54.82%, 48.25%, and 44.29%. Based on the experimental results, it can be concluded that the highest performance was achieved when 3 g of adsorbent and 30 minutes adsorption time were applied for peat water treatment process.

Keywords: adsorbent; dealumination; H_2SO_4; HCl; peat water; red mud.

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INTRODUCTION

Bauxite is the major raw material for producing alumina (Al_2O_3). Indonesia possesses considerable bauxite mineral resources with total approximate reserve of about 1.293.838.207 tons (Antam, 2014). Alumina can be obtained from bauxite through various processes, one of which being the Bayer process. In addition to producing alumina, Bayer process also produces bauxite residue or commonly known as red mud. Damayanti and Khareunissa (2017) reported that the mineral content of red mud originating from Tayan, West Kalimantan contains SiO_2 (14,70%), Al_2O_3 (19,22%), Fe_2O_3 (38,50%), K_2O (0,30%), Na_2O (7,54%), CaO (2,61%), MgO (0,37%), TiO_2 (2,52%), LOI (12,36%).

In regard to red mud potential as an adsorbent material, it requires appropriate modification, one of which is by dealumination and calcination processes. Dealumination is basically a process that includes chemical reaction and activation. The interaction of acids with the surface of the zeolite results in the release of alumina species from the zeolite structure. Coordination bonds are formed by the free electrons in the O atom under the effect of the acidic H^+ ion. Al will break free from its bonds because the Al-O group will be more polar and weaker than its original configuration due to the lack of electrons (Norvia et al., 2016). Normally, dealumination of adsorbents is performed by reacting the solid sample with an acid solution. In this research, dealumination was carried out using solutions of hydrochloric acid (HCl) and sulfuric acid (H_2SO_4). According to Ngapa (2017), acid treatment (HCl and H_2SO_4) is intended to dissolve and eliminate the trapped metal oxides that cover the red mud's surface that subsequently enhances its porosity and surface area. This is due to the nature of these acids, which can rearrange the solid particle structure and enlarge the porosity to allow efficient adsorption of adsorbate by the adsorbent (Arung et al., 2014). As calcination process goes by at temperature of 200-700°C for several hours, water and other volatile impurities molecules in the material will vaporize and exit from the material matrix. This phenomenon promotes the opening of red mud pores, thereby increasing the red mud particles capacity (Yi et al., 2013).

Peat water or groundwater is widely found in tidal, swamp, and lowland areas, especially in West Kalimantan. Peat water generally does not meet clean water quality requirements, such as its low turbidity, brown color, high hardness (1,302 mg/L), high iron and organic content, and acidic (pH 4,5) (A'idah et al., 2018). Therefore, prior to its utilization as a source of water for domestic purposes, peat water requires appropriate treatment. The high iron content in peat water results in brownish-red water and metallic odor. The high content of organic substances can be suitable substrates for microorganisms in water that can lead to unpleasant odor upon biological decomposition. In addition, low pH in peat water can cause strong acidity of water (Husada et al., 2016).

Several studies have shown that red mud can be used as an adsorbent to adsorb some environmental pollutants due to its pore size, porosity, surface area and chemical affinity. Red mud with acid treatment had been proven to possess highest phosphorus compound removal of 63.16%, while heat treated red mud has successfully removed phosphorus compound at a slightly lower capacity, which was 61.68%. As expected, the untreated red mud displayed an even lower phosphorus compound removal that was 25.89% (Salim, 2018). In the application of modified red mud as an adsorbent reported by Wang, et al. (2019), the modified red mud with a surface area of 40-70 m^2/g with a pore size of 2.98-3.82 nm was successful in removing various pollutants from aqueous solution and soil, such as metal and non-metal ions, phenolic compounds, and dyes with removal efficiencies above 90%.

Based on the aforementioned literature survey, this research offers a novelty by modifying red mud using two types of acid in the dealumination process and examine its effect on the characteristics of modified red mud as an adsorbent. The modified red mud was analyzed for its surface area and adsorbent content using Gas Sorption Analyzer (GSA) and X-ray Fluorescence (XRF) instruments, respectively. Finally, the modified red mud was subjected to peat water treatment application with variations in adsorbent weight and contact time to find out the best operating condition.

METHODOLOGY

Material

The materials were peat water, aquadest, hydrochloric acid with 37% purity, sulfuric acid of 97% purity, bentonite, and red mud from PT. Indonesia Chemical Alumina (ICA) Tayan. The equipment used in this research were a 100-mesh sieve, electric oven, furnace, Gas Sorption Analyzer (GSA), and X-ray Fluorescence (XRF), and Atomic Absorption Spectrophotometry (AAS).

Red Mud Modification

Firstly, red mud samples were dried under the sun and further oven dried at 100°C for 2 hours. In the next stage, the red mud activation process was carried out via calcination and dealumination through acidification with 1M hydrochloric acid and 1M sulfuric acid solutions. For dealumination purpose, red mud was soaked in an acid solution at room temperature for 4 hours each with liquid/solid ratio of 20 ml/g. After acid processed, red mud was washed with aquadest several times until neutral. Then, calcination was conducted at 700°C for 3 hours in a furnace. The untreated and modified red mud were then subjected to characterization using Gas Sorption Analyzer (GSA) and X-ray Fluorescence (XRF). The treated red mud was mixed with bentonite with a mass ratio of 4: 1. Then enough aquadest was added to form red mud-bentonite suspension. Next, the mixture of red mud and bentonite is dried in the oven at 100°C for 1 hour and calcined at 700°C for 3 hours. After that, a mixture of red mud and bentonite is crushed and sieved using 100 mesh sieve to obtain adsorbent.

Peat Water Treatment Process

Peat water was introduced into a 250 mL beaker. Then, a carefully weighed red mud (1 g, 3 g, 5g, and 7g) as an absorbent was introduced into a 250 mL beaker containing peat water and stirred gently (150 rpm). After a given adsorption duration (15, 30, 60 and 90 minutes), the red mud was separated using filter paper to determine the changes in peat water. Finally, the treated peat water was tested for its total organic matter (TOM), ferrous metal (Fe) content, and pH of the.

RESULTS AND DISCUSSION

The Influence of Acid Type on The Dealumination of Red Mud

Upon evaluation using XRF and GSA apparatus, the results of Si/Al ratio of the adsorbent samples are presented in Table 1.

Tabel 1. Ratio Si/Al of Red Mud

Types of Red Mud	Si/Al Ratio
Raw Red Mud	1.0638
Red Mud-HCl	1.6976
Red Mud-H_2SO_4	1.5657

Table 1 displays a remarkable change of Si/Al ratio of the red mud before and after dealumination. The Si/Al ratio of red mud before dealumination was 1.064, while the Si/Al ratio of the dealuminated red mud using HCl and H_2SO_4 were respectively 1.698 and 1.566.

The increase in the Si/Al ratio is the results of reaction between acid solution with the alumina contained in the red mud that reduce the Al content of the red mud and subsequently improve porosity and the surface area of the dealuminated red mud (Sulistyowati et al., 2018). Furthermore, Table 1 also exhibits higher Si/Al ratio of the HCl dealuminated red mud than the H_2SO_4 red mud. Because dissociation energy value of the Al-O bond (116 kcal/mol) is very much weaker than that of Si-O bond (190 kcal/mol) (Sulistyowati, et al., 2018), the former bond is more easily broken than the latter. This situation is coherent with alumina separation using HCl and H_2SO_4 (Side et al., 2023). Another factor that can result in an enhanced Si/Al ratio is electronegativity. According to Sriyanti (2018), a higher electronegativity value will cause a stronger bonding. Al is more electropositive than Si, where the electronegativity value of Al is 1.5 while Si is 1.8 thus Al binds more easily to Cl^- or SO_4^{2-}. Because Si tends to be neutral, it is more difficult to be attacked by anions. Thus, the increase in the Si/Al ratio in this study was said to be successful because of the decrease in Al in red mud, which was 51.61% for activation using HCl and 55.53% for activation using H_2SO_4. This is as expected because the main purpose of the dealumination process is to break the Al bond of the red mud matrix.

Table 2 summarized the pore characteristics and surface area of red mud adsorbents obtained from GSA analysis.

Tabel 2. Characteristics of Red Mud Pores

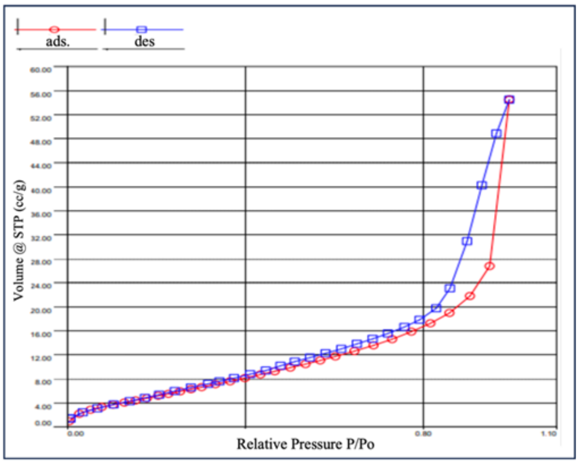
Pore Character	Raw Red Mud	Red Mud-HCl	Red Mud-H_2SO_4
Surface Area (m^2/g)	23.99	67.08	63.08
Total Pore Volume (cm^3/g)	0.08	0.16	0.156
Pore radius (Å)	70.25	48.42	49.65

The results of the GSA analysis test showed that there was an increase in total pore volume and surface area. The surface area of adsorbents obtained from dealumination using HCl increased from 28.994 m^2/g to 67.081 m^2/g, while that resulted from dealumination using H_2SO_4 increased from 23.994 m^2/g to 63.078 m^2/g. The surface area of the adsorbent increases due to activation using acid and calcination, which can dissolve both organic and inorganic impurities that obscuring the pore, so that the pores of the adsorbent are opened and promotes surface area increase. Because surface area is an indicator for active side of the adsorbent, a larger the surface area will promote adsorbate adsorption by the adsorbent. According to (Maleiva et al., 2015) the greater the total pore volume of an adsorbent will result in a higher adsorption capacity. Accordingly, the smaller the pore diameter will provide larger the surface area. Based on the value of the Si/Al ratio and the increase in the surface area of the adsorbent mentioned above, it can be considered that HCl solution is the better dealumination agent. For that reason, the adsorbent used in the subsequent adsorption experiment is the adsorbent that was activated using HCl solution.

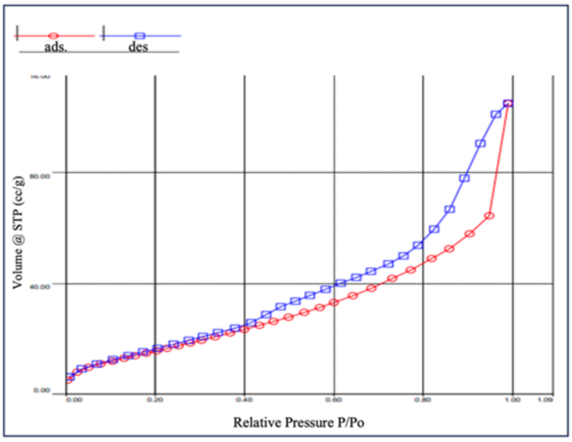
Adsorption Isotherms

Adsorption isotherms that occur when viewed from the isotherm graph based on the pore characteristics of red mud using GSA analysis can be seen in **Figure 1**. **Figure 1** depicts the relationship between the volume of adsorbed nitrogen gas (cc/g) and the relative pressure (P/Po). At relatively low pressures, adsorbate is adsorbed slowly and less significantly. However, at higher relative pressures the adsorbed adsorbate increases quite dramatically until a sharp increase is observed in the graph. When viewed from the adsorption-desorption isotherm graph displayed in Figure 1, the three isotherm graphs follow type II isotherms. This type is an isothermic form in non-porous or microporous adsorbents.

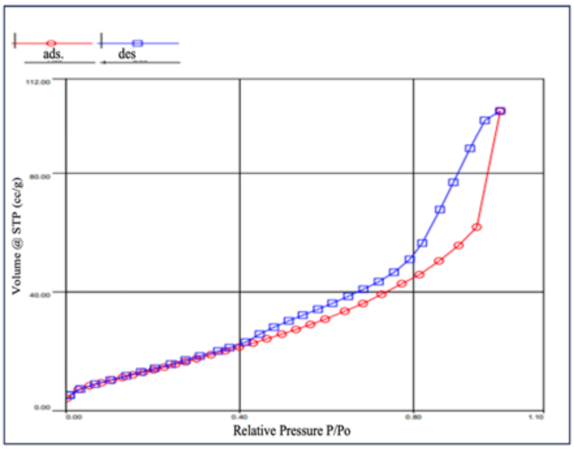
The adsorption process on microporous materials such as red mud when viewed based on the R^2 value tends to follow the BET isotherms where the R^2 value is close to 1, each of which is 0.9995 for pure red mud, 0.9999 for HCl treated red mud, and 0.9999 for H_2SO_4 treated red mud.



(a)



(b)



(c)

Figure 1. (a) Raw red mud desorption adsorption isotherm graph, (b) HCl treated red mud desorption adsorption isotherm graph and (c) H_2SO_4 treated Mud desorption adsorption isotherm graph

Red mud follows the BET isotherm model, which means multilayer adsorption occurs on the adsorbent surface. According to Yustinah et al.(2019), BET isotherms will be more applicable for physical adsorption and used for multilayer adsorption.

Effect of Red Mud Adsorbent Mass Loading Variation on Peat Water Treatment

The influence of adsorbent mass loading variation is a crucial parameter since it may be used to calculate an adsorbent's absorption capability at the initial concentration of adsorbate (Anjani and Koestiari, 2014). Red mud used as an adsorbent is insoluble in peat water, which was proven by the fact that the red mud settled at the base of the container after stirring during adsorption experiment. The experimental results of peat water treatment results with pH, Total Organic Matter (TOM), and Fe parameters can be seen in Table 3.

Tabel 3. Peat Water Treatment Test Results at Various Adsorbent Mass Loading

No.	Adsorbent Loading (grams)	Adsorption Parameters		
		pH	TOM (mg/L)	Fe (mg/L)
1	0	4.4	2.28	1.24
2	1	6.6	1.07	0.33
3	3	7	1.14	0.78
4	5	7	1.27	0.94
5	7	7.1	2.69	3

The data presented in Table 3 reveals that a higher adsorbent loading would result in greater impurities decline. Obviously, a higher adsorbent loading increases pH of the peat water and reduces TOM and Fe concentrations. An increase in pH can be associated with the reduction of humic acid that is represented by TOM value. Humic acid in the peat water that induces lower pH was adsorbed onto red mud adsorbent. During the adsorption process, there is strong interactions between the negative group on the $COOH^-$ (humic acid) and the positive group of the red mud adsorbent (Fe^{3+}) (Morti et al., 2018). The best pH in this test result was found in an adsorbent mass loading of 3 grams with a pH value of 7.0. The acceptable pH values of peat water range between 6 to 9.

As expected, loading of red mud as an adsorbent would reduce Fe^{2+} ion levels. The decrease in Fe^{2+} ion levels can be due to the binding of metals in the peat water by oxygen on the surface of adsorbent. Therefore, the activation of red mud in this study was carried out by dealumination using HCl so that oxygen becomes unbound due to the detachment of Al in Al_2O_3 in red mud (Sriyanti, et al., 2018). On the surface of the adsorbent there is an active group in the form of oxygen which then binds to Fe metal in peat water. The negatively charged red mud adsorbent (O^{2-}) is able to adsorb Fe metal found in peat water. Oxygen can bind to Fe due to van der waals force. This interaction force occurs due to differences in dipole moments due to differences in electronegativity (Rahayu et al., 2015). However, a higher red mud loading and exceeding the optimum limit can cause enrichment of Fe content in peat water. This because red mud is a material whose dominant content is Fe. In addition, the deforested red mud also experienced an increase in Fe content as can be seen in Table 3, so that when the adsorption process runs, the Fe in the red mud dissociates into peat water and increases its content. Thus, the best red mud adsorbent loading for reducing Fe content in this peat water treatment test was 1 gram with a decrease in Fe content from 4.4 mg/L to 0.33 mg/L.

Similar to the Fe level, the total organic matter (TOM) also decreases when the peat water was contacted with red mud adsorbent. This can be caused because organic compounds in the form of humic acids are adsorbed to the adsorbent. Negatively charged humic acids will interact with the active side of the positively charged adsorbent because in red mud Fe is present in the form of Fe_2O_3, so that the active side of the adsorbent that interacts or binds to ions in peat water is Fe^{3+} ions. This causes an electrostatic interaction where the oxygen present in the humic acid is negatively charged and the Fe^{3+} ion present in the red mud is positively charged. The negative charge on humic acids occurs due to the presence of aliphatic groups so that when this group reacts with water it will be ionized. The -COOH group will change to COO^- through removal of H atoms, which is generally influenced by electrokinetic charge. Electrokinetic charge is a phenomenon in which particle charge arises due to ionization in a polar solvent. Under this condition, attractions exist between the negative charge of the humic acid with the positive charge of red mud (Junisu, 2017).

The adsorbent mass loading into adsorbate solution determines the quantity of adsorbate adsorbed from the testing solution. In this study, the best red mud adsorbent mass loading for TOM reduction was 1 gram. However, the results of this study show a difference between theoretical data and factual data because the greater the mass of adsorbents used, the smaller the decrease in TOM. This can be due to the presence of dissolved Fe ions derived from red mud which interfere with the adsorption process of surface pores to bind organic compounds in peat water. This can be seen from the data for Fe level parameters. The higher adsorbent loading, the higher the increase in Fe level. Where it is known that red mud testing, the results of red mud adsorbent adsorption isotherm follow the BET isotherm model which means multilayer adsorption occurs on the adsorbent surface. Absorption of surface active sites has greater potential for desorption than active sites in the pore (Alimano and Syafila, 2014), especially in physical adsorption processes. Based on this, it can be caused by the saturation of the adsorbent so that it can release adsorbate which causes an increase in organic levels as the adsorbent loadings were beyond 1 gram.

Effect of Red Mud Adsorption Time Variation on Peat Water Treatment Results

Adsorption time can be expected to significantly change the adsorption process. The absorption capacity of the adsorbent increases with adsorption duration until the achievement of equilibrium condition (Atikah, 2017). The results of the analysis of the effect of contact time variations on peat water treatment results with pH, TOM, and Fe parameters can be seen in Table 4.

The variation in contact time has a substantial effect on peat water treatment. Theoretically, a longer the adsorption duration will result in a higher adsorption of the adsorbate by the adsorbent. However, if the adsorption process is too long, it can actually reduce the amount of adsorbate because the adsorbent will experience saturation on its surface after reaching an equilibrium state.

Tabel 4. Peat Water Treatment Test Results at Various Adsorption Time

No.	Adsorption Time (minutes)	Adsorption Parameters		
		pH	TOM (mg/L)	Fe Metal (mg/L)
1	0	4.4	4.2	1.24
2	15	6.8	1.54	1.6
3	30	6.9	1.03	0.88
4	60	7.1	1.18	0.9
5	90	6.9	1.27	0.94

From pH data tabulated in table 4, it can be seen that there is a significant increase in pH value at 15-minute adsorption. However, a prolong peat water treatment through adsorption beyond 15 minutes did not significantly alter the pH. The optimum adsorption duration in this study was 30 minutes that the pH of peat water was 6.9 because this value is very close to neutral condition (pH 7.0). According to Morti, et al. (2018) iron in peat water is in the form of Fe^{2+} ions. Hence, interactions exist between Fe^{2+} ions and adsorbents that have a negative charge (O^{2-}) binding to Fe^{2+} metal cations. With the reduction of metal ions, there are more OH- ions in peat water which causes the pH of peat water to increase.

As for the Fe level evaluation, the data presented in Table 4 also suggested that using HCl treated red mud adsorbent for 15 minutes caused an increase in Fe metal content. This should not happen because at the beginning of the adsorption the adsorbent surface is still free of adsorbate particles, so the adsorbate can enter easily into the adsorbent surface. However, in this study there was an increase in Fe levels in peat water because the Fe contained in red mud dissolved in peat water (Gynra & Verghese, 1978). The highest adsorption of Fe occurred in the 30th minute adsorption as indicated by the lowest Fe level in the peat water, which was 0.88 mg/L. The decrease in Fe^{2+} ions in peat water occurred because oxygen in red mud binds Fe^{2+} ions in peat water (Sriyanti, et al., 2018). As the adsorption process was extended to beyond 30 minutes, there is a decrease in Fe adsorption efficiency, most probably due to saturation of the adsorbent and some adsorbate desorbed back to the peat water. Therefore, the optimum adsorption time for Fe metal removal was 30 minutes. At the beginning of the adsorption process, the red mud adsorbent pores are still free from adsorbate particles so that the opportunity for adsorbate to enter red mud adsorbent pores and adhere on their active sites is still very large.

Based on the TOM parameter data summarized in Table 4, the optimum adsorption time for adsorbent particles for the removal of organic substances in peat water was 30 minutes. Under this condition, the TOM value dropped to its lowest value, which was 1.03 mg/L. But at adsorption time beyond 30 minutes, a steady increase in TOM level in the peat water was observed. This finding suggested that organic molecules did not bind permanently to the active side of the adsorbent due to saturation of the adsorbent and some desorbed back (released) to the peat water (Dini et al., 2023).

CONCLUSION

Based on the experimental results, some conclusion can be drawn. Dealumination of red mud using HCl solution could result in higher Al content reduction and surface area than using H_2SO_4 solution. An excessive loading of adsorbent mass caused an increase in Fe ions dissolution from red mud, and subsequently interfered the adsorption process. The best adsorption condition for peat water treatment using HCl- dealuminated red mud was using 1 gram adsorbent and 30 minutes adsorption time. A prolong adsorption process will promote adsorbent surface saturation as indicated by the achievement of an equilibrium state.

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