



Removal of Fe (II) ions from Aqueous solution using Rice-husk Adsorbents in a fixed-bed column

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Abstract

Rice husk has been converted into activated carbon for the adsorbent to remove the heavy metal from the aqueous solution. This study aimed to convert rice husk to activated carbon (AC) for use in the adsorption of Fe ions in a fixed-bed column. Rice husk was first pyrolyzed in an atmosphere of nitrogen gas at 400 °C, then a chemical activation method using sodium hydroxide. The rice husk activated carbon (RH-AC) was characterized using Fourier transform infrared (FTIR) and Scanning electron microscope (SEM) to identify the functional group and microstructure of carbon. The performance of the carbon was tested on the Fe removal from an aqueous solution in a continuous column. The adsorption process was carried out using Fe solution with an initial concentration of 3 mg/L as an artificial sample. The amount of carbon is 25, and 50 g were filled in an adsorber column with a diameter of 5.4 cm and height of 40 cm. SEM images revealed that the activated carbons shown with well-developed pore sizes and pore structure were produced after the chemical activation. The FTIR absorption bands observed in the RH-AC sample confirmed the presence of hydroxyl (-OH), carbonyl, and carboxylic (-COOH) groups of RH-AC adsorbent. The highest Fe removal efficiencies were 91.9% on chemically activated carbon and column mass 50 g at 400 minutes. The overall study revealed the potential value of chemically activated RH-AC as a possible commercial adsorbent in a continuous column wastewater treatment strategy.

Keywords: activated carbon, adsorption, rice-husk, iron, continuous column

1. Introduction

Nowadays, the rapid growth of industry needs special attention because of the impact of environmental pollution. The wastes from industries such as hospitals and mining sectors contain chemicals, such as pesticides, herbicides, chlorine, heavy metals, and other organic (Husin & Rosnelly, 2007; Jakfar et al., 2020; Noor et al., 2017; Othman et al., 2018; Zaki et al., 2019b). Among various pollutants present in surface water, inorganic species of heavy metals and their metalloids are of major concern as they are difficult to remove owing to their smaller ionic size, complex state of existence, and competition with nontoxic inorganic species (Bouazza et al., 2018; Buldurun et al., 2019). Inorganic species, especially Fe are commonly found in the ground water. However, the existence of high doses could cause unfavorable health effects and

damage to many living beings. The exceeds of high dose of Fe ions can cause the water to turn into a brown color, has a bad odor, and the wall of the container which contacted would turn into brown (Chen et al., 2017; Sun et al., 2013; Song et al., 2011).

The maximum tolerance limit of Fe in drinking water specified by the Ministry of Health Regulation, No 492, 2010 is 0.3 mg/L. According to the Ministry of Health Regulation of the Republic of Indonesia No.32/2017 estimate, excess iron concentration in groundwater is 1.0 mg/l (Depkesri, 1990). Therefore, the Fe metal from the water should be taken seriously to protect humans and the environment. Various methods for removing the dissolved heavy metal, including iron, copper, cadmium, Mangan from wastewater have

been used (Glatstein & Francisca, 2015; Jakfar et al., 2021; Xiao et al., 2019). These include filtration, flocculation, coagulation, precipitation, membrane separation, electrochemistry, ion exchange, and adsorption processes (Abu-El-Halawa & Zabin, 2017; Natalea et al., 2020; Pawar et al., 2016). Among the methods, adsorption is easy to operate for the removal of heavy metals from aqueous wastes. This technique is one of the effective and frequent water treatment processes used to remove heavy metals. Adsorption has advantages over other methods because of its simple design, the use of non-toxic, low-cost adsorbent, and being environment-friendly in removing pollutants (Ghafar et al., 2020; Husin & Rosnelly, 2006; Krishnan et al., 2011; Lata et al., 2019).

Many adsorbents have been used for adsorbents and catalysts, such as CaO, NiO, Al₂O₃, activated carbon, and chars (Mahmoud et al., 2015; Okash Toamah & Kadhim Fadhil, 2019; Zaki et al., 2019a). Activated carbon (AC) is one of the most fertile materials use in the adsorption process of removing contaminants such as metallic and non-metallic pollutants from aqueous solutions (Gilmour et al., 2018; Karnib et al., 2014). Many researchers have successfully used an activated carbon from biomass for metal removal from an aqueous solution.

Agriculture waste would be an attractive precursor for developing activated carbon as an adsorbent because of the abundance available. Rice husk is one of the best candidates for activated carbon making in this work. They are abundantly available and non-toxic to the ecosystem, with comparable results presented in the literature. (Masoud et al., 2016). The rice husk contains cellulose fibers as a source of carbon. To the best of our knowledge, the preparation and application of activated carbons from rice husk in the continuing column are still very limited in the literature. The use of rice husk adsorbent was reported mostly in batch system adsorption processes. In a previous publication, there is only a report about the physical activation of activated carbon production at very limited data (Maliki et al., 2019).

This present study explores the effectiveness of using activated carbon from rice husk in the adsorption process for reducing Fe ions in water solution on a continuous column process. The adsorbent was treatment via chemical activation. Activated carbon was

characterized using FITR and SEM to identify the functional group and microstructure of carbon.

This study aimed to convert rice husk to activated carbon (AC) for use in the adsorption of Fe ions in a fixed-bed column. The performance of prepared activated carbon was tested in a column to continue for the removal of Fe ions in an aqueous solution. The effluent samples were then collected at a contact time of 20-400 minutes. The other parameter, such as the breakthrough curve, will be reported in the later publication.

2. Methodology

2.1. Material

The activated carbon used in this work was made from rice husk. The chemicals i.e: NaOH, FeSO₄·7H₂O were purchased from Merck (Germany). All the chemicals used in this work were of analytical grade purity.

2.2. Carbon preparation and chemical activation

Rice husk was collected from a local Rice Thresher near the Syiah Kuala University Campus in Banda Aceh, Indonesia. It was thoroughly washed with tap water until they are free from dirt and dust. The clean samples were then sun-dried for two days, followed by oven-dried at 110 °C for six hours. Approximately 10 g of dried rice husk was introduced in a tube furnace (Human Lab Inc. 300-1500 °C, Korea) for a carbonation process. Then, it was heated at a temperature of 400 °C with a heating rate of 20 °C min⁻¹, under a nitrogen flow of 50 cm³ min⁻¹. The samples were grounded using mortar to reduce their size. The powder was then sieved using an aperture size of laboratory stainless steel sieve with a screen up to a size of 30 to 40 mesh. The obtained carbonized sample was stored in a desiccator and labeled (RH-C).

For the chemical activation step, an amount of carbonized sample was mixed with NaOH and distilled water in a ratio of 1:4:5 (RHC:NaOH:H₂O), w:w:v). In a typical procedure, 2.0 g of RHC was put in a glass beaker with 8.0 g of NaOH and 10 mL of deionized water. The mixture was mix using plate magnetic stirrer bars for 2.0 h at room temperature. The sample was dried at 120 °C for 12.0 h. The obtained material was purified with HCl 0.1 M under magnetic stirring. The rest of the acid was removed by adding distilled water until pH 7.0. Finally, the carbon was dried at 100 °C for 24 h,

cooled, stored, and labeled as rice husk-activated carbon (RH-AC).

2.3. Characterization of rice husk activated carbon

The activated carbon was characterized using FTIR and SEM apparatus. Scanning electron micrographs of the samples were obtained via scanning Electron microscopes (a Philips XL30) with accelerating voltages of 2.0 and 1.0 kV. The dried colloidal RH-AC was spread thinly onto the sample holder and coated with a thin layer of gold in a sputter coating unit. FTIR is a useful technique for group function analysis for activated carbon synthesis. The surface chemistry of the samples was analyzed by identifying the surface functional groups of the samples using Fourier transform infrared spectroscopy (FTIR-2000, Perkin Elmer). The FT-IR spectrum was recorded in the spectral range of 400–4000 cm^{-1} .

2.4. Fixed-bed column reactor studies

The activated carbon was examined for its ability to absorb Fe ions in an aqueous solution. The column experiments were performed using a glass column that had a 5.5 cm inner diameter, a height of 40 cm packed with 25 g, and 50 g of AC-RH.

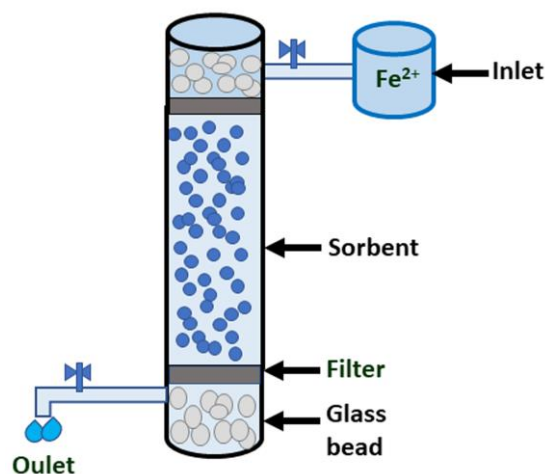


Figure 1. The mechanism of rice husk in the adsorption process to reduce Fe(II) ions in aqueous solution in the continuous column process

The mechanism of rice husk in the adsorption process to reduce Fe(II) ions in aqueous solution in the continuous column process are demonstrated in Figure 1. The feed solution with an initial solution concentration of Fe(II) containing 3 mg/L

and the pH was maintained at 7.0–7.5. The solution was transported using a pump from the top of the column down at a constant flow rate of 1.0 L/min. The effluent samples were then collected for 20–400 minutes. The effects of the contact time were studied under two different RH-AC beds, 25 g, and 50 g. The concentration of the Fe ion in the effluent and the total bulk sorptive concentration of Fe ions were measured using atomic absorption spectroscopy (AAS). The removal percentage of the adsorbed Fe ions concentration was estimated by Equation (1).

$$E = \left(\frac{C_0 - C_t}{C_0} \right) \times 100\% \quad (1)$$

Where E : efficiencies, C_0 : is the initial Fe concentration (ppm), C_t is the Fe concentration at a certain time (ppm).

3. Results and Discussion

3.1. FTIR spectra of RH-C and RH-AC samples

Fourier Transform Infrared (FTIR) analyses of adsorbent (RH-C and RH-AC) were performed to determine the surface functional groups, respectively. From Figure 2, it can be seen that there is a spectrum at wavenumbers 470 cm^{-1} and 1100 cm^{-1} in the RH-C sample, representing the stretching vibration of Si-O functional groups. According to (Chowdhury et al., 2011), the rice husks contain as much as 20% silica.

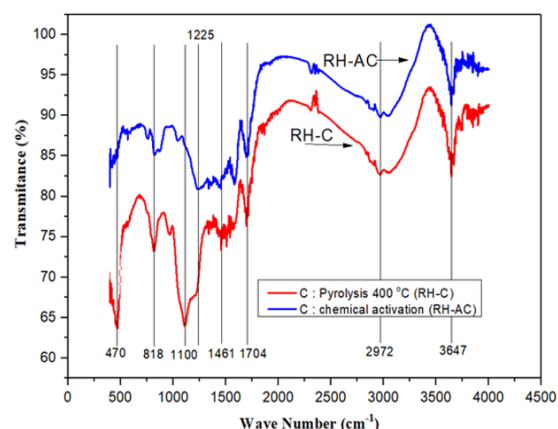


Figure 2. FTIR spectra of RH-C and RH-AC sample by chemical activation

Another research group reported that the band of the Si-O group was seen at waves 1101, 944, 789, and 470 cm^{-1} (Van et al., 2014). The presence of Si-O group in raw rice husk was also reported in the previous

literature (Coates, 2006) and (Gupta & Balomajumder, 2016), namely at the wavelength of $900\text{--}1100\text{ cm}^{-1}$ and $461\text{--}798\text{ cm}^{-1}$. However, the spectra RH-AC at wavenumbers 470 cm^{-1} and 1100 cm^{-1} were not appearing. This fact was because of the NaOH could shed most of the silica content in rice husks during the activation process of RH-AC. The presence of the band at wavelengths 818 cm^{-1} , 1461 cm^{-1} , and 2972 cm^{-1} indicate the presence of aromatic groups (C=C), carbonyl groups (C=O), aliphatic groups (CH), hydroxyl (OH) on RH-AC spectra (Jin et al., 2016).

The NaOH also has an important role in creating the C-O functional group of carbon band, it can break and eliminate C-O-C and C-C bonds from raw materials (Cazetta et al., 2011). The bonds of O-H, C-H, C=O, and C=C are functional groups that are generally found in activated carbon (Pavia, 2009; Prasetyo, 2013). Furthermore, the presence of carbonyl groups on the surface of the adsorbents, denoted by the band at $2972\text{--}818\text{ cm}^{-1}$, has been reported to favor the adsorption of Fe (II) via the formation of -COOH by bonding with neighboring -OH groups.

3.2. Scanning electron micrographs analysis of RH-C and RH-AC

Morphological analysis of the surface of activated carbon was characterized using Scanning Electron Microscope (SEM). Scanning electron microscopy (SEM) images of the RH-C and RH-AC are presented in

Figures 3.a and 3.b. The surface morphology of the activated carbon was different from the precursor as many of the pore size increased and rough surface morphology after activation processes. This fact was because of the chemical degradation effect during the activation processes, which resulted in the release of silica components. The activated carbon prepared contains well-developed pores where there is a good possibility for iron to be adsorbed onto the surface of the pores. The RH-AC developed more pores inside the samples, indicating that additional volatile components were degraded and the porous structure varied during the activation process.

This porous characteristic suggests developing microporous and mesoporous structures in the activated carbon during the activation process. The activated carbon had more microporous structures, as indicated by adsorption isotherms. The development of mesopores was more pronounced and, after chemical degradation of the pore wall, led to the widening of micropores. This fact is in accordance with the FTIR data; there is no silica group visible on carbon with chemical activation.

3.3. Adsorption Efficiency of Fe ions on Carbon Rice Husks

The time dependence study in the continuous column provided information about the rate which removed the Fe pollutant from the solutions and the time required to achieve adsorption equilibrium.

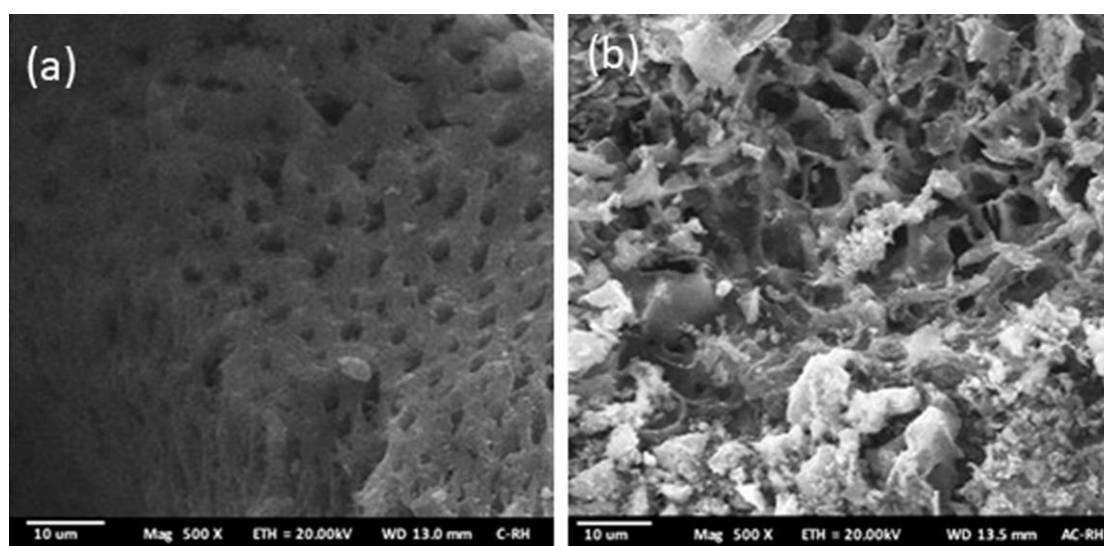


Figure 3. SEM images of (a) RH-C sample; (b) RH-AC sample by chemical activation

These parameters are important for designing an adsorption process. The effects of the contact time in the removal of Fe(II) using RH-C and RH-AC were studied at an initial concentration of 3.0 mg/L with two different adsorbent doses. A series of adsorption experiments were carried out to compare the efficiencies of activated carbon on Fe removal from aqueous solutions. The effect of the column mass on the adsorption of Fe ions on the RH-AC is plotted in Figures 4 and 5.

As can be observed from Figure 4, the relationship plot time versus Fe removal, the absorption efficiency of Fe ions starts 49.8% at 20 minutes, increased slowly to 57.8% at 150 minutes using 25 g carbon mass in the column. Afterward, the Fe reduction efficiency tends to decrease slowly towards saturation even decrease to 56.3% at 400 minutes. The reason for the decrease in efficiency in the column mass 25 grams is because of the carbon has started to saturate for absorbing Fe. Therefore, the carbon mass in the column greatly affects the efficiency and length of time for Fe absorption.

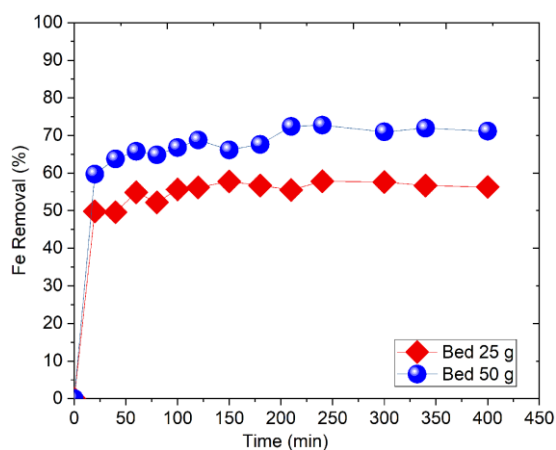


Figure 4. Fe absorption efficiency on rice husk carbon, pyrolysis at 400 °C without activation.

When the column mass increases two times, i.e., 50 g, the Fe absorption efficiency reaches 72.8% at 240 min, as depicted in Figure 4. Therefore, the mass adsorbent in the column was very influential to the adsorption efficiency and duration time of Fe absorption. This result is in accordance with reported by other groups in adsorption systems that the rate of removal is directly correlated with concentration with the adsorbent dose (Rahmani et al., 2018).

The same trend also occurs in chemically activated carbon (RH-AC), as shown in Figure 5. From Figure 5, it can be seen that the percentage removal of heavy metal Fe using rice husk-activated carbon is very significant. It can remove more than 81.3% Fe content in water solution. The percent Fe reduction efficiency is faster at the higher carbon mass. This result is because of an increase in the mass of activated carbon causes a more significant amount of Fe to be absorbed, and the transport time is faster because of an increase in the diffusion coefficient or mass transfer coefficient. It is observed that the carbon with a chemical activation shown continues to increase in Fe removal efficiency until the 400 minutes, the percent Fe removal efficiency reaches 91.9%.

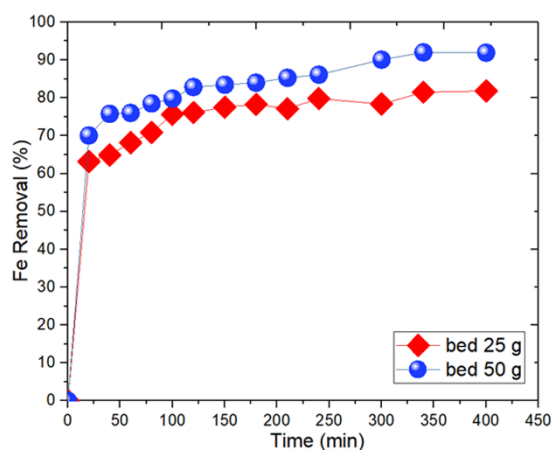


Figure 5. Fe absorption efficiency on rice husk activated carbon, with chemical activation

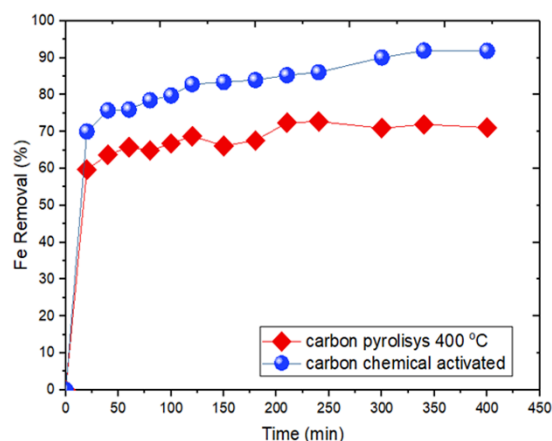


Figure 6. Fe absorption efficiency of husk carbon without and with chemical activation (Co= 3 mg/L), carbon mass of 50 g

Figure 6 shows a comparison of the performance of RH-C and RH-AC for

absorbing Fe aqueous solution 3.0 mg/L at a carbon mass in the column 50 g. As shown in the figure, the Fe removal reached 72.8% at 240 min. Furthermore, the removal of Fe starts to decrease slowly up to 71% at 400 min. The types of carbon have a significant influence on Fe removal. Carbon without further activation shows lower activity because of the smaller pore size; thus the RH-C tends to get saturated faster.

Chemically activated carbon (RH-AC) has a higher performance than carbon from pyrolysis (RH-C). The Fe removal on the RH-AC after 400 minutes reaches 91.9%. This could be because of the improved characteristics of activated carbon following the chemical activation, including enhanced pore volumes, as depicted in SEM analysis (Figure 3). Activated carbon is a porous solid containing 85-95% compounds carbon (Asnawi et al., 2019; Zaki et al., 2020).

4. Conclusion

Activated carbon was synthesized by pyrolysis and chemically activated for the adsorbent. Rice husks were used as precursors to prepare activated carbon with large pore size, sufficient adsorbent, and high Fe absorption efficiency. The activated carbon produce has a better pore size. The extremely fast adsorption rate is observed in the initial 30 min using 50 g RH-AC. A further increase in contact time slowly increases the percentage of Fe metal ion removal. The carbon mass of 50 g in a continuous column has better activity, reduce Fe ions 91.9% from a water solution. The overall excellent absorption performance of activated carbon is because of the enlargement of surface pores which provides a larger contact area. The high capability of RH-AC adsorbent makes it potentially attractive for the removal of Fe(II) ions in wastewater treatment, especially for the groundwater from Fe metal ions.

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