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Utilization of *Jengkol* Peel (*Pithecellobium jiringa*) as an Adsorbent of Iron Metal

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Abstract

Cellulose, which has the active group OH, is found in Jengkol peel. Jengkol peel has the potential to be utilized as an adsorbent for Iron (Fe) metal because it contains these active groups. The FT-IR spectrophotometer instrument was used to confirm the presence of the -OH group in the Jengkol peel adsorbent, which was observed at the peak of the OH vibration at wave number 3293 cm1. Utilizing NaOH (0.1 M), the active group was activated. With an adsorption percentage of 96.24%, pH 7 was the optimum adsorption pH. With an adsorption percentage of 99.20%, 25 minutes was indeed the optimum contact time. With an adsorption capacity value of 8.581 mg/g, the binding of Fe metal by the adsorbent active group occurs in the complex formation mechanism, it was indicating a relatively high adsorption efficiency.

Keywords: *Adsorbent; Jengkol peel; adsorption; heavy metal; Fe*

Introduction

According to data from the South Sumatra Provincial Central Agency obtained from the farmers in 2018, *Jengkol* yields in South Sumatra Province reached 5,615 tons, putting it one of Indonesia's top-producing provinces (Selatan, 2018).). *Jengkol* remains only consumed as a fruit, and as its value has not been fully realized, the majority of the peel is still wasted. Based on the findings, quite a few *Jengkol* peels continued to be wasted, particularly at the Palembang Jakabaring Main Market. In South Sumatra, *Jengkol* peel has been utilized, as a natural dye for *songket* and *jumputan* textiles (Failisnur, S Sofyan, S Silfia, Salmariza Sy, 2019).

The *Jengkol* plant, well-known as the *Jering* plant, is a species of the Fabaceae (grain tribe) with the Latin name *Pithecellobium jiringa*. It is a typical plant found in Southeast Asia (Hutauruk, 2010). *Jengkol* peel contains chemical compounds in the form of cellulose, hemicellulose, and lignin (Wardani *et al*., 2020). *Jengkol* peel can be utilized as an adsorbent because these compounds contain hydroxyl and carboxyl groups, which can adsorb heavy metals (Idanta, 2017). *Jengkol* peel has been utilized as an adsorbent to absorb the heavy metals Pb and Cu. The adsorption capacities at the optimum pH, namely pH 3, were 1.6667 mg/g and 1.5860 mg/g, respectively. The optimum contact time for Pb(II) metal ions was 30 minutes, with an absorption efficiency of 72.28%, and for Cu(II) metal ions was 90 minutes, with an absorption efficiency of 95.52% *(Hamzah et al*., 2013).

One of the heavy metals that easily enters the environment is the Fe metal ion. Fe metal can be found in water in the form of ferrous ions (Fe^{2+}) and ferric ions (Fe^{3+}) (Aziz *et al*., 2018). Because Fe^{3+} is more stable in neutral or alkaline circumstances than Fe^{2+} and because Fe^{2+} is a strong reducing agent and easily oxidized to Fe^{3+}, Fe will form compounds more readily in the form of Fe^{3+} (Steven Wang & Sugiarso, 2015). Fe metal can be absorbed from the environment through a process called adsorption. Compared to other methods, adsorption is thought to be more effective at removing heavy metals from water because it is simple to prepare and relatively inexpensive (Karim, Juniar, and Ambarsari, 2017).

Changes in pH can affect the presence of Fe metal ions during the adsorption process. The adsorption activity at the adsorbent active site is also impacted by pH changes (Rohmatullaili, 2020). The identification of the optimum adsorption pH was carried out by varying the pH of the metal solution during adsorption, specifically pH 3, 4, 5, 6, 7, and 8.

The amount of time in contact with the adsorbent during adsorption can show how long the adsorbent needs to optimally adsorb before absorbing Fe metal ions. The adsorption capacity under the adsorption circumstances can be calculated using the optimal adsorption time. This data was gathered by altering the contact time with a time range of 15 minutes, ranging from 15 to 145 minutes.

Research Methodology

Tools and Materials

This study used some tools such as a beaker, volumetric flask, blender, oven, desiccator, stirring rod, spatula, Erlenmeyer shaker, measuring cup, glass funnel, dropper pipette, sieve *shaker* with 100 *mesh,* pH meter, analytical balance, stopwatch, FT-IR *Alpha II-Bruker* (*Bruker Alpha),* and Atomic Absorption Spectrometer (AAS) (*Thermo Scientific*). Meanwhile, the materials were *Jengkol* peel waste, NaOH (*Emsure*), HCl (Emsure), and $FeCl_3$ powder (*Emsure*).

Work Procedure

Making and activating the *Jengkol* peel adsorbent

First is cleaning the dirt by washing the *Jengkol* peel in clean water and letting it dry in the sun to a constant weight. After being ground into a powder, dried *Jengkol* peel was then sieved through a 100-mesh.

The prepared *Jengkol* peel powder was put in the shaker after spending 60 minutes soaking in 0.1 M NaOH. The activated *Jengkol* peel powder was then dried in an oven to a consistent weight and chilled before being rinsed with distilled water until having a neutral pH. By using an FT-IR spectrophotometer, dried *Jengkol* peel adsorbents were further characterized both before and after activation.

Adsorption of Fe Metals Procedure

Making Fe Solution

The test solution was $FeCl_3$, made by dissolving 0.05 grams in 1000 ml of distilled water.

pH variation

The adsorbent was weighed up to 0.1 gram before being placed in each container containing 20 mL of 50 ppm $FeCl_3$ solution. The pH of the solution was adjusted by adding 0.1 M HCl or 0.1 M NaOH to achieve pH values of 3, 4, 5, 6, 7, and 8. For 60 minutes, the mixture was shaken with a shaker. After filtering the adsorbent and adsorbate mixture, the filtrate was used for an AAS measurement of the Fe metal.

Contact Time Variation

As much as 0.1 grams of *Jengkol* peel adsorbent was put into each container containing 20 ml of $FeCl_3$ solution. According to the optimum pH determined in the previous procedure, the mixture was shaken using a shaker with variations in contact times of 10, 25, 40, 55, 85, 100, 115, 130, and 145 minutes at room temperature. The mixture of adsorbent and adsorbate was then filtered, and the amount of metal in the filtrate was analyzed by AAS.

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Research Results and Discussion

Jengkol peel contains organic compounds in the form of lignocellulose and hemicellulose. Activation with a strong base will dissolve the lignin (Yuniati, 2015). NaOH as an activator will break down lignocellulose into cellulose and lignin (Wardani et al., 2020). The hydroxyl group bound to cellulose will play a role in the adsorption process of Fe metal on the *Jengkol* peel adsorbent (Yuniati, 2015). Identification of active groups in *Jengkol* peel was carried out using FT-IR instruments. The FT-IR spectra of the identification results are shown in Figure 1.

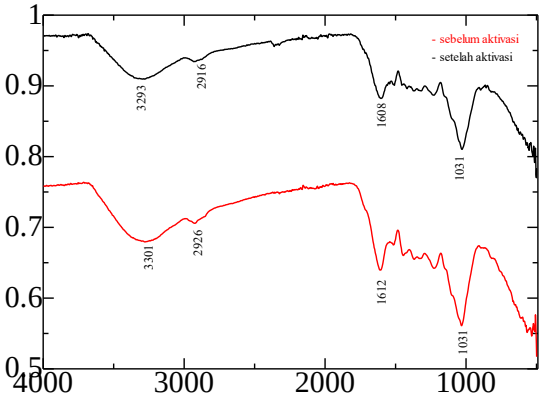

Figure 1. Graph of FT-IR spectra before and after activation

Figure 1 shows the red spectra of the *Jengkol* peel before activation. the characterization result shows absorption at wave number 3301 cm^{-1}. In addition, it shows C-H sp^3 (cellulose) stretching vibration at wave number 2926 and wave number 1608 cm^{-1}. This indicates the presence of C=O groups which means the presence of lignin and hemicellulose (Wardani & Wulandari, 2017).

The black spectrum shows *Jengkol* peel after *activation*. It can be seen that the characterization result shows that there was absorption at wave number 3293 cm^{-1}, which indicates the presence of a vibrating hydroxyl group (OH^-). Then, the wave number 2928 cm^{-1} is the vibration of the alkyl group (CH) from (-CH3). The stretching vibration was at wave number 1608 cm^{-1} which related to the stretching vibrations of the C=O group, thus indicating the presence of lignin and hemicellulose (Wardani & Wulandari, 2017).

The difference in the results of the characterization before and after activation was a shift in the absorption of the hydroxyl group (-OH) from wave number 3301 cm^{-1} to 3293 cm^{-1} (Wardani & Wulandari, 2017). The C=O peaks of lignin did not differ significantly before and after activation. Lignin levels had decreased as indicated by a decrease in intensity before and after activation.

The activation process also changed the color and mass weight of the adsorbent. It happened when the activation was marked by the formation of a black solution, and the weight of the sample after activation was lighter than before, which was originally from 60 gr to 40 gr. The activation process can remove about 35% of the lignin from the initial weight. It is shown in the brown gradation in Figure 2 below:

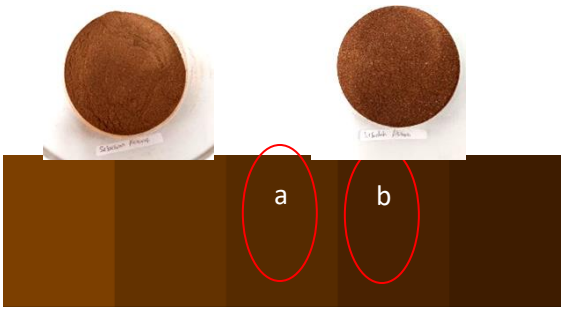

Figure 2. Color gradation of adsorbent samples: (a) before activation and (b) after activation

To determine the optimum conditions of Fe metal being adsorbed by *Jengkol* peel adsorbents was carried out by the pH variations. At different pH levels, the percentage of Fe metal adsorption by *Jengkol* peel adsorbents showed different results. The adsorption results of the Fe solution with *Jengkol* peel adsorbents through pH variations are shown in Figure 3.

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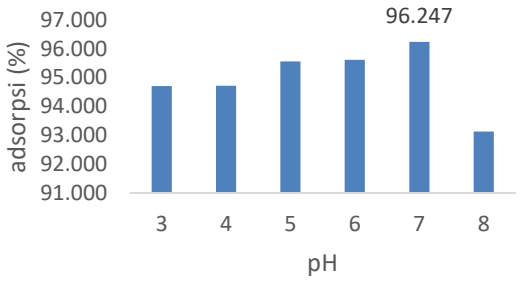

Figure 3. The effect of pH on the adsorption of Fe metal

Figure 3 shows that the optimum adsorption occurred at pH 7. At each pH level, *Jengkol* peel adsorbents absorbed the following amounts of Fe metal ions: 94.70%, 94.71%, 95.6%, 95.62%, 96.24%, and 93.13%. In an aqueous solution, pH 7 indicates a balance between the concentrations of OH^- and H^+, with no excess OH^- binding Fe metal ions and forming $Fe(OH)_3$. To obtain optimal adsorption under these adsorption circumstances, Fe metal ions exclusively interact with the OH group on the *Jengkol* peel adsorbent cellulose. In contrast to the previous studies, the adsorption of Fe metal using fly ash achieved optimum adsorption at pH 5. Fly ash's silanol groups are deprotonated to generate anions at pH 5, which bind Fe metal cations very efficiently (Irawan & Ain, 2018). After testing at pH ranges between 2 and 5, the adsorption of Fe metal with plantain peel biological charcoal showed optimal adsorption at pH 3. pH 3 affected the density of the biological charcoal used, which also had an impact on the adsorption results (Nirmala *et al*., 2015).

Due to the excess H^+ in the solution, the adsorption process at pH 3, 4, 5, and 6 produced lower adsorption percentages. Excess H^+ will protonate the adsorbent's active group (OH) in adsorption conditions below the optimum pH (pH 3 to 6). In contrast to Fe ions, which are charged 3^+, H^+ ions in solution are more readily attracted to the electronegative side of OH- from cellulose. It makes more difficult for Fe metal ions to reach the adsorbent active site (Rohmatullaili, 2020). The presence of H^+ will make it more difficult for the positively charged Fe metal ions to bind the active groups (-OH) on the adsorbent's surface.

Compared to another pH, adsorption at alkaline pH 8 produced the smallest amount of adsorption. Excess OH- in the solution can interact with Fe metal ions, resulting in solid $Fe(OH)_3$ (Amalina *et al*., 2015). The amount of adsorption reduces because solid $Fe(OH)_3$ precipitates and does not take part in the adsorption process. The same thing was reported for the adsorption of Fe metal using Zeolite 4A as an adsorbent, which showed a decrease in adsorption capacity at pH 7 to 8. The higher the pH, the more difficult the metal ions in aqueous solutions dissolve and form hydroxide bonds (Sari *et a*l., 2022).

A variation of contact time was carried out to determine the optimum time needed by the adsorbent to adsorb Fe metal ions**.** The optimum adsorption time is reached when all the active groups have bonded with Fe metal ions. Consequently, the addition of contact time will not affect the resulting adsorption percentage. The results of contact time variations can be seen in Figure 4 below:

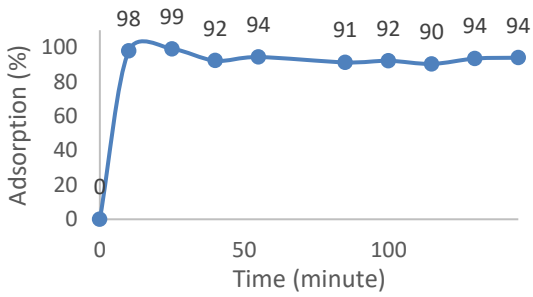

Figure 4. The effect of time on Fe metal adsorption.

Adsorption of Fe metal ions drastically increased from the 10th to the 25th minute. After 25 minutes, the percentage of adsorption slowly decreased until 145 minutes. Optimum adsorption occurred at the 25th minute with an adsorption percentage of 99.20%.

At the contact time of the 10th to the 25th minute, there was an increase in the adsorption efficiency of the *Jengkol* peel adsorbent. This was a result that at the beginning of the adsorption process, the

surface of the *Jengkol* peel adsorbent still did not absorb too many Fe metal ions. As a result, the Fe metal absorption process was more effective because the surface of the *Jengkol* peel adsorbent was not blocked by the Fe metal that had bound to the *Jengkol* peel adsorbent. The adsorption percentage was still being added from the 10th minute, and as much as 97.91% increased by 99.20% in the 25th minute. It was caused by the active groups had not all interacted with Fe metal ions in the 10th minute, but they all interacted with Fe metal ions in the 25th minute.

The absorption of *Jengkol* peel by the adsorbent began to gradually decrease at the contact time of 25 to 145 minutes. This occurred because the *Jengkol* peel adsorbent's surface became increasingly coated with Fe ions. As a result, the active group began to become sterically blocked, which prevented metal ions from binding and stopped the *Jengkol* peel adsorbent from adsorbing more Fe ions.

The optimum adsorption capacity of Fe metal under the adsorption conditions was 8.581 mg/g, which means that 1 gram of *Jengkol* peel adsorbent was able to adsorb 8.581 mg/g of Fe metal. Previous research conducted by Taihuttu *et al*. (2019) reported that the adsorption of Fe metal with sago pulp waste adsorbent had an adsorption capacity of 0.3211 mg/g, which was reached in the 90th minute. These results indicate that the utilization of *Jengkol* peel adsorbent is more effective with an adsorption capacity value reaching 26 times than using sago pulp adsorbent (Taihuttu *et al*., 2019). The mechanism for binding Fe metal to the OH groups of cellulose occurred by forming complexes through covalent bonds (Mandasari & Purnomo, 2016), which is shown in Figure 5.

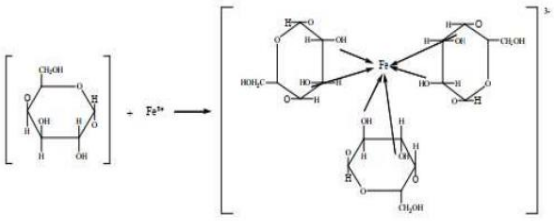

Figure 5. Binding of $Fe3^{+}$ metal to the OH group of cellulose (Taihuttu *et al*., 2019).

Conclusion

Based on the research results, the presence of the -OH active group from cellulose, which can be detected in the FT-IR measurements at wave number 3293 cm^{-1}, is what causes the *Jengkol* peel adsorbent to bind Fe metal ions. The optimal pH was found at pH 7 with an adsorption percentage of 96.24%. The pH variation had an impact on the adsorption process of Fe metal ions using *Jengkol* peel adsorbent. With a 99.20 % adsorption and an 8.581 mg/g adsorption capacity, the optimum contact time for the adsorption of Fe metal using *Jengkol* peel adsorbent was reached in the 25th minute.

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