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Kinetics Adsorption of Fe Metal using Cellulose Acetate from Lontar Palm Fronds (*Borassus Flabellifer*)

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Abstract: *Palm tree fronds (Borassus Flabellifer) contain 54,27% cellulose can be utilized as adsorbent cellulose acetate that can bind to the metal iron. Adsorption method used for binding the adsorbate on the mass and contact time best. This study aims to determine the characteristics of cellulose acetate from stem of papyrus and the ability of the adsorbent cellulose acetate stem of the papyrus in the adsorb metal iron (Fe). The stages of the manufacture of cellulose acetate include cellulose insulation, the synthesis of cellulose acetate, testing the content of acetyl and the value of DS, as well as the use of Fourier transform infrared (FTIR) and atomic absorption spectrophotometer (AAS) to characterize the cellulose acetate. The result obtained is the existence of functional groups O-H, C=O and C-O. The absorption capacity of the Fe metal is obtained on the mass of the optimum of 0,06 g, the efficiency of absorption of 66,39%. Contact time best 90 minutes with the efficiency of absorption of 47,56%. The adsorption kinetics of cellulose acetate on metal iron (Fe) followed the Pseudo Second Order modeling with with values of R2 = 0.9935 and k = 0.0096.*

Keywords: *Palm tree fronds, Cellulose Acetate, kinetics adsorption*

INTRODUCTION

Lontar (*Borassus Flabellifer*) is an abundant plant in Indonesia, especially in South Sulawesi. South Sulawesi is rich in palmyra plants, especially in Wajo Regency, Gilireng District, and Polewalie Village. The lontar palm trees in this area grow along the road, and almost every tree has fallen fronds. Usually, lontar palm fronds are only used as firewood by local people, but because palm fronds are tough, people leave the waste without further action. Palmyra waste has many benefits because it contains 54.27% cellulose (Saduk & Niron, 2018). The high cellulose content found in palm leaf fibre can be processed into an essential ingredient for making adsorbents.

The high cellulose content of palm frond waste can be used as an alternative adsorbent that can bind metals. Cellulose is one of the main components of lignocellulosic materials in the form of homopolysaccharide microfibrils. It comprises *β-D-glucopyranose units* connected by glycosidic bonds (Novia, et al.2015). Cellulose can also be renewed, degraded and modified with other materials to improve the adsorbent produced.

Efforts to increase the use value of cellulose can be modified with glacial acetic acid to become cellulose acetate, which is used as an adsorbent for iron metal (Fe). Iron is one of the heavy metals that is widely used in the industrial world, including electroplating. Apart from that, Fe metal can also be found as a contaminant in the mining industry, clean water processing, residential areas and waste. So, reducing the levels of iron (Fe) in water is necessary because it can cause effects such as dangerous metal poisoning if it exceeds the threshold. This can be avoided by minimizing the metal content through an adsorption process.

The adsorption method is a method that has been widely used because it is effective in water treatment and is used to remove heavy metals (Kan et al., 2013). So, making an environmentally friendly adsorbent from palm leaf fibre is necessary, and it can be used as an absorbent for metals such as iron (Fe).

Iron metal (Fe) can be adsorbed using a cellulose adsorbent, which has been reported by (Ischak et al., 2021) with a peanut shell adsorbent with an optimum mass of 1 gram with an absorption result of 0.7467 mg/L with an efficiency value of 74.67%. Meanwhile, the optimum time is 90 minutes with adsorption of 0.5386 mg/L, and the efficiency value is 53.86%. (Takarani, et al., 2019) Using corn husk acetate adsorbent to absorb Fe obtained an optimum mass of 0.1 gram with an optimum time of 45 minutes. Based on the description above, this study entitled The Use of cellulose acetate from Lontar Palm Fronds (*Borassus Flabellifer*) as an Adsorbent for Ions Fe (II).

RESEARCH METHODS

Materials and Instruments

The materials used in this research were distilled water, acetic anhydride, glacial acetic acid (CH3COOH) 90%, nitric acid (HNO_3) p.a 2 M, sulfuric acid (H_2SO_4) p.a, sodium hydroxide (NaOH), sodium sulfite (Na_2SO_3) p.a 2%, solid iron (III) Nitrate ($Fe(NO_3)_3$), palm fronds (*Borassus Flabellifer*), tissue and waterone.

The instruments used in this research were the Atomic Absorption Spectrophotometer (ASA) (AA240FS) and Fourier Transform Infrared (FTIR) Thermo Fisher Scientific.

Methods

Isolation Cellulose

A sample of palm fronds was collected at Polewalie Village, Gilireng District, Wajo Regency, South Sulawesi. Next, it is dried and then made into powder. The powder obtained was sieved using a sieve shaker of 100 mesh sieve size and dried again for 1 hour in an oven at 110°C.

The dried palm leaf fibre powder was weighed in 150 grams and then put into a beaker, and 1000 mL of HNO_3 3.5% was added, then heated at 90°C for 2 hours while stirring on a hot plate. After heating, the mixture is filtered, and the residue obtained is washed until the pH is neutral. Next, 375 ml of NaOH 2% and 375 mL of Na_2SO_3 2% were added, heated at 50°C for 1 hour while stirring on a hot plate, filtered, and the residue obtained was washed until the pH was neutral. Next, 500 mL of NaOH 17.5% was added, heated at 80°C for 30 minutes, and filtered, and the residue obtained was washed until the pH was neutral (Djuned et al., 2014).

Synthesis of Cellulose Acetate

The synthesis of cellulose acetate is carried out in the following stages: 10 grams of isolated cellulose was put into an Erlenmeyer flask, then 250 mL of glacial acetic acid (CH3COOH) was added and stirred at 38°C for 1 hour, then 0.5 mL of concentrated sulfuric acid (H2SO4) was added and stirred again for 45 minutes at room temperature. The activation results were followed by an acetylation process using 130 mL of acetic anhydride and stirring at 38°C for 45 minutes. Then 25 mL of distilled water and 50 mL of glacial acetic acid were added to stop the acetylation process and stirred at 50°C for 30 minutes.

The solution obtained was then deposited in distilled water and filtered until the acetate aroma disappeared. The obtained precipitate was dried using an oven at 55°C for 12 hours (Apriania et al., 2017).

Characterization of Cellulose and Cellulose Acetate using Fourier Transform Infrared (FTIR)

The samples were analyzed using FTIR-ATR from Thermo Fisher Scientific in the absorption area of 500-4000 cm^{-1}, so that the FTIR-ATR spectrum was obtained which was used to see the absorption peaks of the functional groups in the sample.

Kinetics Adsorpstion

0.06 grams of synthesized cellulose acetate was added to 10 mL of 1 ppm Fe solution. After that, the shaker was at a speed of 150 rpm with varying times of 60, 90 and 150 minutes. The mixture was filtered, and then the concentration of Fe(II) ions in the filtrate was determined using AAS.

RESULTS AND DISCUSSION

Synthesis of Cellulose Acetate

An essential stage in making cellulose acetate is the acetylation process. This acetylation process aims to produce cellulose acetate. The first stage in the synthesis is the addition of glacial acetic acid, which functions as a swelling agent to thicken the cellulose fibres to make them more open so that they can quickly react with anhydrous acetic acid. The next stage, adding anhydrous acetic acid and sulfuric acid, which act as catalysts, aims to replace the hydroxyl groups of cellulose with acetyl groups. The acetylation reaction was stopped by adding dilute acetic acid, then hydrolyzed at 50°C. Anhydrous acetic acid will undergo protonation due to the presence of sulfuric acid, so carbonium ions are formed. This carbonium ion will react with cellulose to form cellulose acetate. In this research, 18.38 grams of cellulose acetate was obtained from palm leaf cellulose, which was yellowish and white. The general esterification reaction of cellulose-to-cellulose acetate is shown in Figure 1 (Gaol et al., 2013).

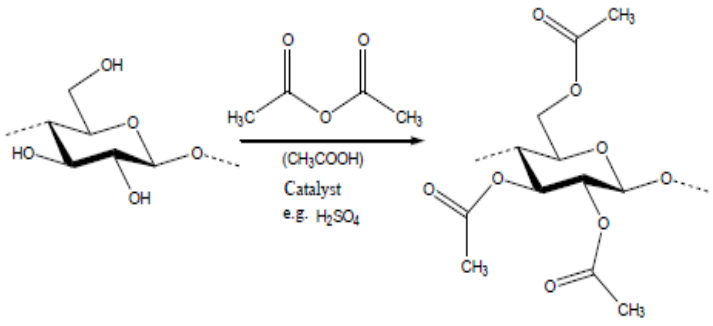

Figure 1. Cellulose Acetylation Reaction

The results of functional group analysis using FTIR show the carbonyl group C=O (1723.73 cm-1) and the ester group C-O (1103.99 cm-1) from the acetyl group which indicates the formation of cellulose acetate with the peak formed in the cellulose acetate

spectrum with wavelength 1723.73 cm-1 and a decrease in the intensity of the O–H group due to being substituted by an acetyl group. The main functional groups of cellulose acetate are the C=O and C–O ester groups (Thaiyibah et al., 2016). The following are the results of functional group analysis using FTIR with the spectrum produced from cellulose and cellulose acetate of palm fronds shown in Table 1.

Table. 1 Spectrum FTIR of Cellulose and Cellulose Acetate of Palm Fronds

Functional groups	**Spectrum area (cm^{-1})**	**Wave numbers range (cm^{-1})**	
		Cellulose	**Cellulose Acetate**
O–H Streching	3200 – 3500	3334.75	3334.91
C–H Streching	2800 – 3000	2897.94	2898.02
C=O	1600 – 1800	1618.11	1723.73
C=C Cincin Aromatik	1500 – 1900	–	–
C – H Bending	1320 – 1480	1428.18	1428.54
C–O–C	1000 – 1300	1200.7	1244.06
C–O	1000 – 1250	1100.52	1103.99

The hydroxyl group (OH) of cellulose acetate in palm leaf fronds is visible at a wavelength of 3334.91 cm-1. The absorption area of palm frond cellulose acetate has a higher and broader absorption area than commercial cellulose acetate, namely 3486.97 cm^{-1}(Nurhayati & Kusumawati, 2014). The C–H functional group is shown at a wavelength of 2898.02 cm-1. The absorption area is 1723.73 cm^{-1}, which indicates the characteristics of the C=O ester group, while the C–O–C group is at a wavelength of 1244.06 cm^{-1}. C–O group at a wavelength of 1244.06 cm-1. The results of functional group analysis using FTIR show the carbonyl group C=O (1723.73 cm^{-1}) and the ester group C–O (1103.99 cm^{-1}) from the acetyl group which indicates the formation of cellulose acetate with the peak formed in the cellulose spectrum acetate with a wavelength of 1723.73 cm^{-1} and a decrease in the intensity of the O–H group due to substitution by the acetyl group. The main functional groups of cellulose acetate are the C=O and C–O ester groups (Thaiyibah et al., 2016). Based on FTIR spectrum data, it can be seen that cellulose acetate from palm fronds was successfully synthesized by inserting acetyl groups into the cellulose.

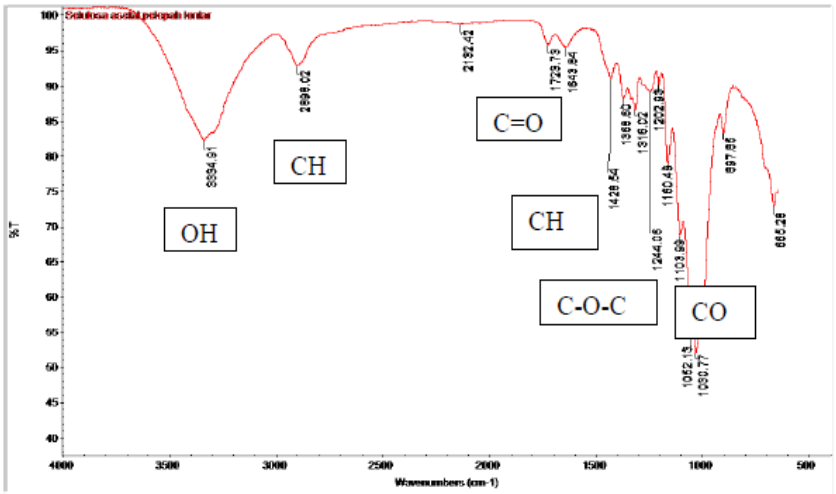

Figure 2. Spectrum FTIR of Cellulose Acetate Lontar Palm Frond

Effect of Contact Time

To determine the optimum contact time for adsorption of Fe(II) ions on cellulose acetate, variations in time were made, namely 60, 90 and 150 minutes. The data presented in Figure 3 shows that at 60 minutes, the absorption value obtained was 44.12%, and an increase occurred in minutes. Ninety minutes, where the absorption efficiency value was obtained at 47.06%, there was a decrease at 150 minutes, with an absorption efficiency value of 43.02%. So, the optimum contact time is obtained at 90 minutes. The adsorbent has absorbed the Fe(II) ions in the solution. This is also influenced by the adsorbent surface, which is already saturated with Fe(II) ions. The size of the adsorbent used also influences the adsorption process. A large surface area will allow interaction between the active site of the adsorbent and metal ions (Khopkar, 1990).

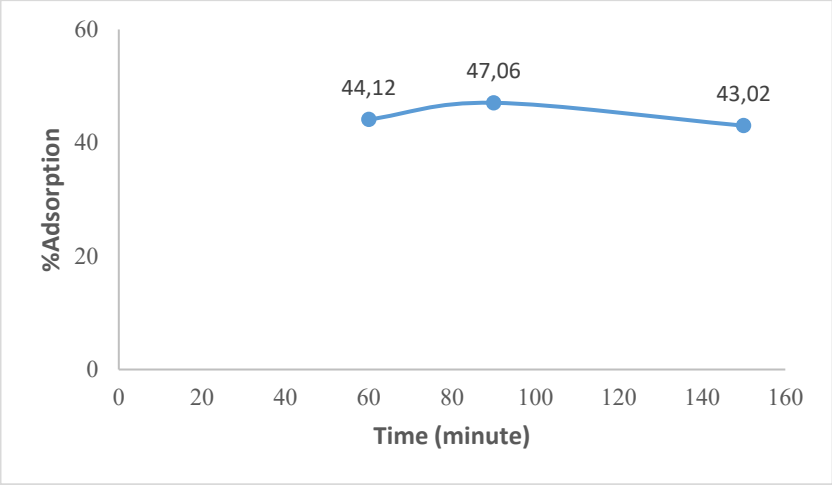

Figure 3. The Effect of Contact Time

Order of Reaction

Determination of the reaction order is obtained from the relationship between reaction time and residual concentration using a graphic method with orde-0, orde-1 and orde-2 equations. The reaction order is determined based on the linear relationship approach, namely the regression coefficient value close to 1. The regression coefficient value is a statistical value in linear regression that describes the strength and direction of the linear relationship between the dependent and independent variables. This value is called standard because the range is -1 to 1. The larger it is (closer to 1), the more accurate the predictions made. The value of the regression coefficient R^2 obtained for zero order is R^2= 0.973, first order R^2 = 0.9872 and second order R^2 = 0.9935. This shows that second-order provides a more linear relationship than zero and first-order, so this research follows second-order reactions.

Table 2. Kinetics Adsorption of Fe Metal using Cellulose Acetate from Lontar Palm Fronds (*Borassus Flabellifer*)

Kinetic Model	Rate Constant (k)	R^2
Orde-0	0,005 minute	0.9730
Orde-1	0,007 minute	0.9872
Orde-2	0,009 minute	0.9935

CONCLUSIONS

The ability to absorb cellulose acetate from palm fronds with an optimum mass of 0.06 grams was 67.4797%. In contrast, the optimum contact time at 90 minutes was 45.4546%, following the second order with a rate constant of 0,009 minutes and regression coefficient R^2 = 0,9935.

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