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KINETICS ADSORPTION OF Fe(II) IONS USING CELLULOSE ACETATE FROM NIPAH FRONDS (*Nypa Fruticans*)

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ABSTRAK

Adsorben adalah zat padat yang memiliki kemampuan menyerap komponen tertentu dari fase fluida. Permasalahan pencemaran logam berat seperti besi (Fe(II)) dalam air semakin meningkat akibat aktivitas industri dan domestik, yang dapat menurunkan kualitas lingkungan serta membahayakan kesehatan manusia. Oleh karena itu, diperlukan bahan adsorben yang efektif, ramah lingkungan, dan mudah diperoleh untuk mengatasi masalah tersebut. Salah satu sumber potensial adalah pelepah nipah (*Nypa fruticans*), yang mengandung selulosa dalam jumlah tinggi dan selama ini belum dimanfaatkan secara optimal. Penelitian ini bertujuan untuk mengetahui karakteristik selulosa dan selulosa asetat pelepah nipah serta kemampuan selulosa asetat dalam menyerap ion Fe(II). Tahapan dalam pembuatan selulosa asetat meliputi isolasi selulosa pelepah nipah (*Nypa Fruticans*), sintesis selulosa asetat, penentuan kadar asetil dan derajat substitusi, analisis menggunakan FTIR dan AAS. Hasil yang diperoleh dari selulosa yaitu terdapat gugus O–H, C–H dan C–O sedangkan untuk selulosa asetat dari pelepah nipah adanya gugus C=O, C–O–C dan C–O. Metode adsorpsi digunakan untuk mengikat adsorbat pada massa dan waktu kontak optimum. Kapasitas serapan logam Fe diperoleh pada massa optimum 0,06 g, efisiensi serapan 52,34%. Waktu kontak optimum pada 90 menit dengan efisiensi penyerapan sebesar 43%. Kinetika adsorpsi mengikuti kinetika adsorpsi orde dua dengan konstanta laju sebesar 7,7 x 10^{-3} dan koefisien regresi R^2 = 0, 9998.

Kata kunci: Pelepah nipah, Selulosa asetat, Kinetika adsorpsi, Ion Fe(II)

ABSTRACT

Adsorbents are solid substances that have the ability to adsorb certain components from the fluid phase. The problem of heavy metal pollution, such as iron (Fe(II)), in water is increasing due to industrial and domestic activities, which can reduce environmental quality and endanger human health. Therefore, effective, environmentally friendly, and easily obtainable adsorbents are needed to address this problem. One potential source is nipah palm fronds (*Nypa fruticans*), which contain high amounts of cellulose and have not been optimally utilized. This study aims to determine the characteristics of cellulose and cellulose acetate from nipah palm fronds and the ability of cellulose acetate to adsorb Fe(II) ions. The stages in the production of cellulose acetate include the isolation of cellulose from nipah palm fronds (*Nypa Fructicans*), the synthesis of cellulose acetate, the determination of the acetyl content and degree of substitution, and analysis using FTIR and AAS. The results obtained from cellulose show the presence of O–H, C–H, and C–O groups, while for cellulose acetate from nipah palm fronds, there are C=O, C–O–C, and C–O groups. The adsorption method was used to bind the adsorbate at the optimum mass and contact time. The Fe metal adsorption capacity was obtained at an optimum mass of 0.06 g, with an adsorption efficiency of 52.34%. The optimum contact time was 90 minutes with an adsorption efficiency of 43%. The adsorption kinetics followed second-order adsorption kinetics with a rate constant of 7.7 x 10^{-3} and a regression coefficient of R^2 = 0.9998.

Keywords: Nipah palm fronds, Cellulose acetate, Kinetics adsorption, Fe(II) ions

INTRODUCTION

The nipah tree is a palm plant classified as a mangrove. Nipah palm trees are spread across Indonesia, covering the islands of Sumatra, Kalimantan, Java, Sulawesi, Maluku and Papua. Especially in South Sulawesi. Nipah trees (*Nypa fruticans*) can be found in Lakkang Village, Tallo District, Makassar City. The palm tree has many benefits including brooms, roofing rods, palm sap, and craft materials. One part of the nipah palm that is underutilized by the community is the frond. Nipah palm fronds have a fairly high cellulose

content as a viable source for many applications (Damanik et al., 2024).

Cellulose is one of the most abundant resources in nature and plays an important role as the main constituent of plant cell walls (Kuki et al., 2020). Cellulose is a polysaccharide consisting of 2000–4000 glucose units linked by *β-1,4 glycosidic* bonds. Cellulose is highly abundant, making up about 40–50% of dry weight. around 40% (Mabee and Saddler, 2010), 16-17% in the leaves and 30-34% in the stalks of tobacco (Kulić and Radojičić, 2011) and at 90% at cotton (Huang et al., 2021).

There are several compounds derived from cellulose, one of which is the ester group such as cellulose acetate, cellulose acetate is formed from the substitution of the acetyl group for the hydroxyl group of cellulose. Based on its physical and chemical properties, it has excellent commercial value as a non-toxic, odorless and tasteless white solid. Apart from that, it has the potential to be biodegradable and environmentally friendly. (Fensia Analda Souhoka, 2018).

The use of cellulose acetate in various research fields has been reported, including research by Mayangsari & Setiawan (2016) which has extracted cellulose acetate from paper waste as biodegradable. Apart from that, cellulose acetate contains fiber so it can be used as an adsorbent. Adsorbents can be obtained from various kinds of raw materials such as natural waste (plants or animals). Adsorbents have a function as absorbent or adsorbing substances.

Heavy metal contamination, especially iron, has caused many environmental and health problems, thus requiring the development of efficient and sustainable remediation strategies. Cellulose acetate has emerged as a promising adsorbent for heavy metal removal due to its biodegradability, abundance, and modifiable surface properties (Aldalbahi et al., 2020). Several studies related to heavy metal adsorption use cellulose acetate obtained from several sources. Research reported that cassava peel cellulose can be used as an adsorption agent for iron metal in wastewater with a concentration reduction of 54.66% (Sari, 2020). Cellulose has been successfully synthesized from peanut shell waste resulting in a reduced concentration of Fe metal of 74.67% (Ischak, *et al*., 2021). The effectiveness of cellulose acetate has been proved including modified activated carbon and composite beads, for adsorbing heavy metals such as Cu and phosphate, and investigating its kinetic behavior towards Fe ions is crucial for expanding its application in wastewater treatment (Manik et al., 2025) (Waweru., et al., 2019). The preparation of cellulose acetate/zeolite composites has shown promising results for metal ion adsorption, indicating that modification of cellulose acetate can enhance its capacity and regeneration potential for heavy metal removal (Truong et al., 2023).

This study investigated the kinetic adsorption of Fe ions on cellulose acetate synthesized from nipah leaves, with the aim of elucidating the adsorption mechanisms that govern and optimize removal efficiency. This study further explores the potential of cellulose acetate in heavy metal remediation by examining its performance in the context of iron removal, which contributes to a broader understanding of sustainable adsorbent technologies. An equation to determine the adsorption rate is adsorption kinetics. Adsorption kinetics states that there is a process of absorption of a substance by an adsorbent as a function of time. The characteristics of the adsorbent's absorption ability towards adsorbate can be seen from the adsorption rate. The adsorption rate can be determined from the adsorption rate constant (k) and the reaction order resulting from an adsorption kinetics model.

MATERIALS AND METHODS

Materials and Tools

The materials used in this research were distilled water, acetic anhydride, glacial acetic acid (CH_3COOH) 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 ($Fe(NO_3)_3$), nipah fronds (*Nypa Fruticans*), tissue and waterone.

The tools used in this research were the Atomic Absorption Spectrophotometer (ASA) (AA240FS), Fourier Transform Infrared (FTIR) Thermo Fisher Scientific, desiccator, oven, Ohaus analytical balance, shaker, shield shaker, hotplate, pH meter, thermometer 110°C, porcelain cup, blender, pulp, spatula, clamp, scissors and glassware.

Methods

Isolation Cellulose

A total of 150 grams of dried nipah fronds fiber powder was put into a beaker and 1000 mL of 3.5% HNO_3 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 2% NaOH and 375 mL of 2% Na2SO3 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 17.5% NaOH was added, heated at 80˚C for 30 minutes,

filtered and the residue obt˚ained was washed until the pH was neutral. Then sufficient 10% H_2O_2 was added at a temperature of 60˚C for 15 minutes. The results obtained were cellulose which was then oven at 110˚C for 6 hours (Apriania, *et al*., 2017).

Synthesis of Cellulose Acetate

Acetylation of cellulose to obtain cellulose acetate can be done by putting 10 grams of cellulose into an Erlenmeyer flask then adding 250 mL of CH_3COOH and stirring at a temperature of 38˚C for 1 hour then adding 0.5 mL of concentrated H_2SO_4 and stirring for 45 minutes at room temperature. The activation results were continued with an acetylation process using 132 mL of acetic anhydride and stirring at a temperature of 38˚C for 45 minutes. Next, 25 mL of distilled water and 50 mL of glacial acetic acid were added to stop the acetylation process, then stirred at 50˚C for 30 minutes. The solution obtained was then precipitated and then washed with distilled water until the acetate aroma disappeared. The precipitate obtained was dried in an oven at 55˚C for 12 hours (Apriania, *et al*., 2017).

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

The sample was 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 (Novianty, *et al*., 2024).

Kinetics Adsorption

0.06 grams of synthesized cellulose acetate was added to 10 mL of 1 ppm Fe solution. After that, shaker at a speed of 150 rpm with varying times of 60, 90 and 150 minutes. The mixture was filtered, then the concentration of Cu(II) ions in the filtrate was determined using AAS (Novianty, *et al*., 2024).

RESULT AND DISCUSSION

Synthesis of Cellulose Acetate

Isolation of palm leaf cellulose was carried out to obtain cellulose powder. The nipah palm frond fibers obtained were then dried, then ground and sieved using a Shieve Shaker with a 100 mesh sieve. The nipah palm frond powder obtained was then continued in the delignification process using 3.5% HNO_3 solvent aimed at removing the lignin content in the palm frond powder in the form of nitrolignin.

The cellulose obtained is then continued in the Swelling process using 2% NaOH and 2% Na_2SO_3 solvents with the aim of opening the pores of the cellulose so that impurities will come out. Components such as hemicellulose, minerals and ash content are lost to produce a blackish red color which causes the lignin compounds to dissolve during the heating process (Sheltami, *et al*., 2012).

The process of making cellulose acetate consists of three stages, namely activation resistance, acetylation and hydrolysis. The first stage is the activation process by adding an activator and catalyst using the solvents glacial acetic acid (CH_3COOH) and sulfuric acid (H_2SO_4) Pa which aims to obtain a large surface area of the cellulose fiber so as to facilitate the diffusion of sulfuric acid and anhydrous acetic into the cellulose fiber. The second step involves the acetylation process using anhydrous acetic acid, intended to substitute the hydroxyl groups in cellulose with acetyl groups. In the third step, hydrolysis is carried out to eliminate part of the acetyl groups from the cellulose triacetate and to reduce the presence of sulfate ester bonds. After that, distilled water and glacial acetic acid (CH_3COOH) are added to terminate the acetylation reaction. Next, it is precipitated using distilled water until the acetate aroma disappears, then dried at 55°C for 12 hours with the aim of removing the water content in the cellulose acetate (Adityo Sawong Seto, 2013). The general reaction for the formation of cellulose acetate is shown in Figure 1. The resulting cellulose acetate can be seen in Figure 2.

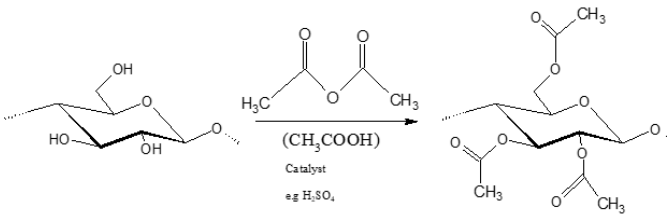

Figure 1. General reaction for the formation of cellulose acetate

Figure 2. Nipah Palm Leaf Cellulose Acetate

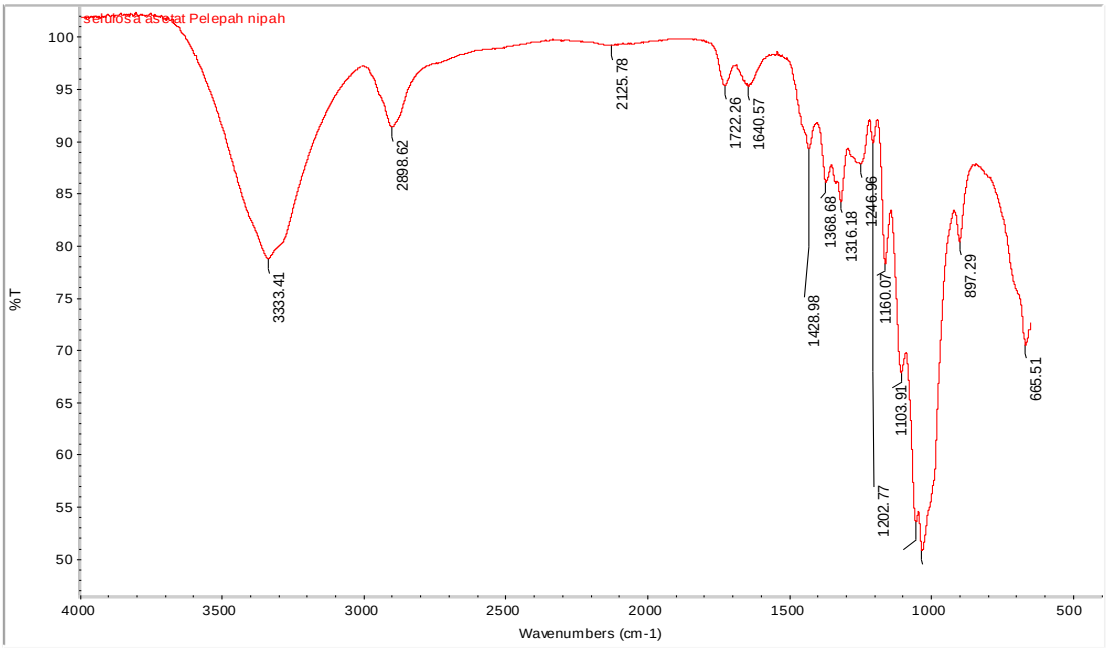

Figure 3. Spectrum FTIR of Cellulose Acetate of Nipah Palm Fronds

Based on the results obtained, characterization of palm leaf cellulose acetate using FTIR shows the presence of the C=O ester group in the absorption area of 1722.26 cm^{-1}, the C–O–C group in the absorption area of 1246.96 cm^{-1} and the C–O group at the wave number 1103.91 cm^{-1}, 1160.07 cm^{-1} and 1202.77 cm^{-1}. The main functional groups of cellulose acetate are the C=O and C–O ester groups which indicates that the cellulose acetylation process has been successful (Amalia, et all., 2024). Theoretically, the formation of the C=O ester group is an indication that some of the hydroxyl groups (–OH) in cellulose have reacted with acetic anhydride, producing an acetyl group (–O–$COCH_3$). These findings are consistent with the results of Masturoh et al. (2025), who reported that acetylated cellulose from corn stalks showed absorption peaks for hydroxyl groups (–OH) at a wavelength of 3333.68 cm^{-1}, carbonyl groups (C=O) at 1724.5 cm^{-1}, and ester groups (C-O) from acetyl groups at 1021.86 cm^{-1}. The FTIR spectrum of cellulose acetate from nipah fronds is shown in Figure 3.

Effect of Contact Time

The process of adsorption Fe(II) ions using an adsorbent in the form of cellulose acetate from palm fronds which was carried out with optimized contact times of 60, 90, and 150 minutes can be seen in Figure 4.

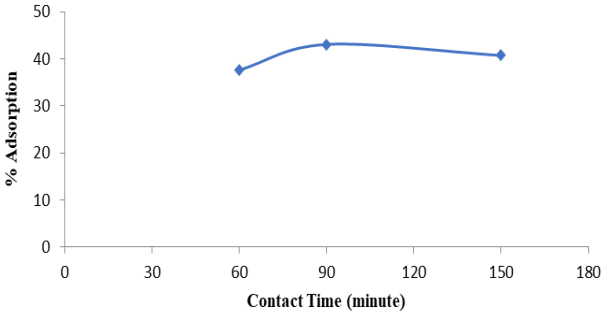

Figure 4. Graph of the Effect of Contact Time on the Adsorption of Fe(II) Ions

Based on the research results in Figure 4, it can be seen that at a mass of 0.06 grams using 150 rpm at optimized contact times of 60, 90 and 150 minutes, it shows that the absorption efficiency of Fe (II) ions in the first 60 minutes is 38%, at 90 minutes the efficiency absorption increased by 43% and at 150 minutes decreased by 40%. An increase in contact time between the adsorbent and Fe metal leads to a higher adsorption capacity of the adsorbent for Fe(II) ions. However, at 150 minutes, a decline in the adsorbent's capacity to adsorb Fe metal was observed. This occurred because the concentration of metal ions in the solution was no longer balanced with the amount of available adsorbent, leading the adsorbent to reach saturation and resulting in reduced adsorption capacity (Irwandi and Silvia, 2015). According to the

research findings, the optimal contact time was 90 minutes, yielding an adsorption capacity of 43%.

Determain Order of Reaction

The adsorption kinetics of Fe (II) ions using cellulose acetate can be determined using a graphical method. Zero order kinetics is obtained from (A0 - A) divided by time (t), from the results of these calculations a curve is created (Figure 5) so that the linearity equation y=-4E-05x + 0.0085 with R^2= 0.9912 is obtained from The value obtained can be seen from the slop and gradient values, namely -4E-05X and the intercept value is 0.0085.

First order kinetics is obtained from $\ln(A_0 - A)$ divided by time (t), from the results of these calculations a curve is created to obtain the linearity equation y = -5E-05x + 0.0108 with R^2 = 0.9982 from the values obtained It can be seen that the slop and gradient values are -5E-05x and the intercept value is 0.0108.

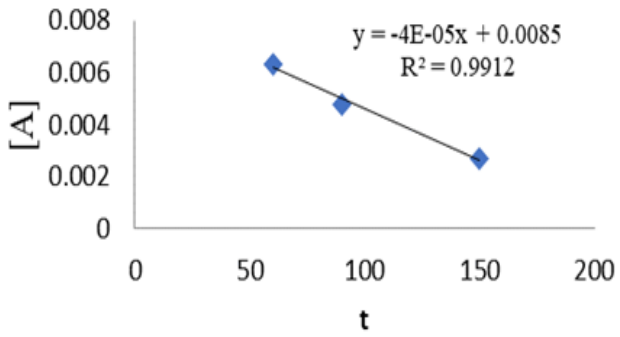

Figure 5. Zero order determination curve

Second order kinetics is obtained from $\frac{1}{[A_0 - A]}$ divided by time (t), from the results of these calculations a curve is created so that the linearity equation y = -6E-05x + 0.014 with R^2 = 0.9998 is obtained. From the value obtained it can be It is known that the slop and gradient values are -6E-05x and the intercept value is 0.014.

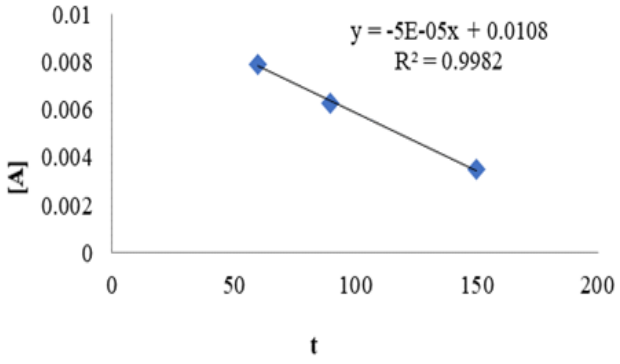

Figure 6. First Order Determination Curve

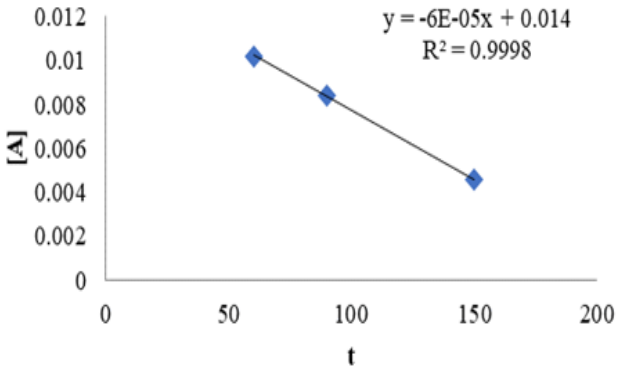

Figure 7. Second Order Determination Curve

Determination of the reaction order is obtained from the relationship between reaction time and residual concentration using a graphic method with equations of order 0, order 1 and order 2. The reaction order is determined based on a linear relationship approach, namely the coefficient value is close to 1. The graph indicates that the adsorption of Fe metal aligns with a second-order kinetic model, as shown by a regression coefficient (R^2) of 0.9998. Second order in the adsorption process shows that adsorption involves a chemical reaction between the adsorbent and adsorbate. The second-order kinetic model assumes that chemisorption, or chemical adsorption, is the rate-limiting step. In this model, the rate of adsorption is influenced by the adsorbent's capacity rather than the concentration of the adsorbate.

Table 1. Kinetics Adsorption of Fe Metal using Cellulose Acetate from Nipah Fronds (*Nypa Fruticans*)

Kinetic Model	Rate Constant (k)	R^2
Order-0	4.6 x 10^{-3}	0.9912
Order-1	5.8 x 10^{-3}	0.9982
Order-1	7.7 x 10^{-3}	0.9998

CONCLUSIONS

From the research conducted, it can be concluded that the optimal contact time for the adsorption of Fe(II) ions using cellulose acetate derived from palm fronds is 90 minutes, achieving an adsorption capacity of 43%. The adsorption process follows a second-order kinetic model, with a rate constant of 7.7 × 10^{-3} and a regression coefficient (R^2) of 0.999

These results indicate that cellulose acetate from nipah palm fronds has potential as an environmentally friendly adsorbent material for the absorption of heavy metal ions such as Fe(II). With abundant and renewable raw materials, this

adsorbent has the potential to be applied in industrial wastewater treatment, particularly in the metal coating, textile, and domestic wastewater treatment sectors, in order to reduce heavy metal pollution in aquatic environments. In addition, the utilization of local biomass waste such as nipah palm fronds also supports the principles of circular economy and sustainable resource management.

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