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Preparation of Cellulose Pineapple (*Ananas comosus*) Peel as Adsorbent of Remazol Yellow Dye

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Abstract

The extraction of cellulose from pineapple (Ananas comosus) peel has been carried out through delignification using alkaline and hydrolysis with acid. The extraction of cellulose was carried out by de-waxing, delignification followed by hydrolysis with H_2SO_4 acid. The cellulose obtain was identified by FTIR and SEM-EDS. The results showed that the FTIR spectra of pineapple peel cellulose contain hydroxyl and carboxyl functional groups which useful in the adsorption process. The morphology of cellulose has a more homogeneous and porous shape. The cellulose obtained was used for adsorption of Remazol yellow dye by studying adsorption parameters, namely the effect of pH, contact time and solution concentration. The adsorption process was obtained at pH 2, the optimum contact time was 30 minutes and concentration at 100 mg/L with an adsorption capacity is 19.888 mg/g.

Keywords: *Cellulose; extraction; pineapple peel; adsorption; Remazol Yellow*

Introduction

The textile industry is one of the industrial sectors that has experienced a significant increase every year. Apart from having a positive impact, the increase in industrial activity was also accompanied by the emergence of various environmental problems, namely the increase in the release of synthetic dye waste into water sources. In the dyeing process, some of the dye is absorbed by the textile material and the residue (2-50%) is lost and thrown into the water (Lestari and Laksono, 2020). One of the synthetic dyes used commercially in the textile industry is Remazol yellow. Remazol yellow is known to have been used as a dye in 27.2% of the textile industry (Handayani *et al.*, 2016). Remazol dyes are widely used as reactive dyes because their chromophores produce bright colors and are resistant to testing, but are compounds that are difficult to be degraded naturally by microorganisms (Arianti, Pramitasari and Widodo, 2022). Chemically, the structure of Remazol Yellow (RY) dye consists of an azo compound (R_1-N=N-R_2) which has high chemical stability, making it difficult to decompose directly by nature (Jović *et al.*, 2013). RY is carcinogenic and mutagenic, thus threatening the life of aquatic organisms and humans. The dye waste water treatment process does important action before released into the environment to minimize the various

problems that this compound can cause (Lucinaldo S Silva *et al.*, 2018).

The several methods have been developed for the treatment of dye waste, including precipitation, adsorption, ion exchange, evaporation, and membranes. Precipitation method is the most economical method, but not efficient. Meanwhile, ion exchange and membrane methods generally require relatively high operational costs (Rajasulochana and Preethy, 2016). The adsorption is a potential method because the process is relatively simple and inexpensive (Mondal and Chakraborty, 2020).

Some of the adsorbents that have been used in the adsorption process are activated carbon, silica, rice husk, coconut coir, sawdust, chitin, chitosan, cellulose. Cellulose is a material that is abundant and available in nature. The cellulose found easily in a variety of waste materials. Pineapple peel is an agricultural waste with a fairly high cellulose content which is still not widely utilized, which is around 40.55% (Pardo *et al.*, 2014).

In this study aims to extraction of cellulose from pineapple peel through delignification process with alkaline and acid hydrolysis. The cellulose obtained was used adsorption of RY by studying parameters adsorption like as pH, contact time and concentration of the solution.

Methods

Materials

The equipment used are a set of grinding tool, sieve mesh 80, hot plate, magnetic stirrer, Memmert oven, Ohaus analytical balance, stopwatch, pH universal, thermometer, a set of glass ware, GBC Ultraviolet/Visible Spectrophotometer Cintra 2020, Infrared Spectrometer (Nicolet iS10, Thermo Fisher Scientific, United States), SEM (Supra Pra 55, Zeiss, Germany), and XRD PAN alytical Empyrean DY 2384

The materials used were pineapple peel obtained from street fruit merchant in Jambi City, distilled water, H_2SO_4, NaOH pro analysis, HNO_3 and Remazol yellow Merck.

Preparation Sample

Pineapple peels were washed thoroughly under running water, dried in an oven at 105°C for 2 hours, cooled and ground with a grinder to obtain pineapple skin powder, then sieved through an 80-mesh sieve.

Delignification

As much as 200 grams of pineapple peel powder mixed with 3% NaOH solution (1:10). The mixture was stirred and heated for 2 hours, then washed with distilled water until close to pH 7 and dried in an oven at 105°C for 5 hours. The dried cellulose extract was crushed with a mortar to prevent it from clumping.

Hydrolysis

As much as 50 g of cellulose was hydrolyzed with 600 mL of 3% H_2SO_4 by boiling for 15 minutes, then cooled for 12 hours. The mixture was washed with distilled water until the pH was neutral and dried in an oven at 50-60 °C for 1 hour. The cellulose extract obtained was crushed and stored in a desiccator.

Physiochemical characterization

The physicochemical characterization of cellulose through several measurements with the Fourier Transform Infrared Spectrophotometer (FT-IR), Scanning Electron Microscope (SEM), and X-ray diffraction (XRD. The FT-IR characterization was carried out on standard cellulose and pineapple peel cellulose in the frequency range of 4000-400 cm^{-1} at 25°C with a resolution of 4 cm^{-1} using an Infrared Spectrometer (Nicolet iS10, Thermo Fisher Scientific, United States). The morphology and structure of the samples were determined by SEM (SUPRA 55, ZEISS, Germany). The sample XRD data was detected using a PAN alytical EMPYREAN DY 2384 XRD with Cu Kα radiation.

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The Determination of Wavelength RY

RY dye solution with a concentration of 10 mg/L was scanned at a wavelength of 400-700 nm with the Ultraviolet/Visible GBC Cintra 2020 Spectrophotometer to determine of the maximum wavelength.

Standard Calibration Curve

As much as 20 mL of RY solution with concentrations of 5, 10, 15, 20, and 25 mg/L, the adsorption value was measured at the maximum wavelength. This concentration series is plotted against the adsorption value to determine the standard calibration curve.

Adsorption Process

The adsorption process of RY with pineapple peel cellulose was studied with various parameters, namely pH, contact time, and concentration (Lyu *et al.*, 2017). As much as 20 mL of 10 mg/L RY solution was put into an Erlenmeyer flask at pH 2-7, adding 0.1 g of cellulose each. The flask was then covered and shaken in a water bath at 100 rpm for 30 minutes. Each sample was taken and analyzed. The adsorption capacity of RY on cellulose, Qe (mg/g), is calculated using the following equation:

$$Q_e = V \times (C_0 - C_e)/m \quad \ldots\ldots \quad (1)$$

where C_0 and Ce (mg/L) are the initial and equilibrium concentrations of RY, respectively, V is the total volume of solution (L), and m is the weight of cellulose (g).

All other batch adsorption experiments were carried out by adding the desired mass to 20 mL of RY solution at the specified pH, concentration and contact time. The flask is then closed and shaken in a water bath. Samples were taken and analyzed with a UV-Vis spectrophotometer. The adsorption capacity of RY on cellulose was calculated using equation 1, and the percentage of removal of RY was calculated using equation 2.

$$\text{Efficiency } (\%) = 100 \times (C_0 - C_e)/C_0 \ldots\ldots \quad (2)$$

Result and Discussion

The Cellulose extraction is carried out in several stages: delignification, hydrolysis and bleachin. Delignification process which is a process to break the bonds between cellulose and lignin and hemicellulose. This process is carried out because the presence of lignin can interfere with the adsorption ability of cellulose. Delignification was carried out using 3% NaOH solvent with the chemical reaction shown in Figure 1.

The NaOH solution can break out the lignin structure in the crystalline and amorphous parts and separate some hemicelluloses. OH^- ions from NaOH will break out bonding from the basic structure of lignin, while Na^+ ions binding with lignin to form sodium phenolate (Deivy Andhika Permata *et al.*, 2021). This phenolic salt will dissolve with a black color mark in the solution (Figure 2a

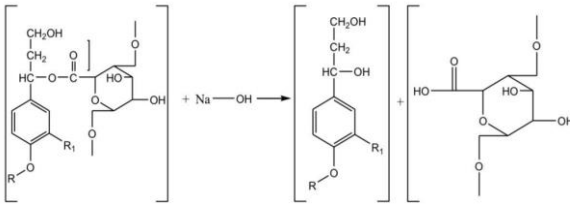


Figure 1. Structure od cellulose.

Figure 2. Delignification (a) hydrolisis proccess (b) and cellulose (c)

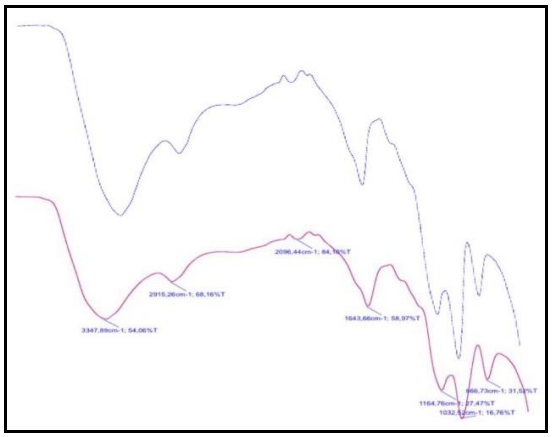
In the process of hydrolysis, sulfate groups are introduced to the surface of cellulose through esterification of the hydroxyl groups of cellulose. This allows anionic stabilization by repulsive forces, leading to the achievement of a stable aqueous dispersion of cellulose (Fig. 2c) (Saravanakumar *et al.*, 2013). Hydrolysis of sulfuric acid can also remove non-cellulose components which act as natural

agglutinatives, so it is expected that the extraction results will produce cellulose with a high crystallinity index, thermal stability and ratio (Silva *et al.*, 2013).

Characterisation of Cellulose

FTIR

The cellulose of pineapple peel and standard cellulose were analyzed by FT-IR and compared (Figure 3). The presence of peaks of 3347.89 and 1643.66 cm^{-1} in both samples indicated that during the extraction process, cellulose was present and not lost (Sun *et al.*, 2005; Sheltami *et al.*, 2012). The process of adding a strong base and acid hydrolysis did not change the structure of cellulose as indicated by the typical peaks of standard cellulose which were identical to those of pineapple skin cellulose. The absence of peaks at 1742, 1514, and 1254 cm^{-1} was due to the significant removal of hemicellulose and lignin through delignification and acid hydrolysis processes. Peak at 1032 cm^{-1} is associated with C-O stretching and C-H vibration of cellulose (Chan *et al.*, 2015).



Fifure 3. The FT-IR spectra of cellulose pineapple peel (a) and standart (b)

Scanning Electron Microscopy-Electron Dispersive X-Ray (SEM-EDX)

The morphology characterization of pineapple peel cellulose using SEM instruments with magnifications of 1000, 5000, 10,000, and 20,000× (Figure 4). The morphology of pineapple peel cellulose at 1000× and 5000× magnification shows a porous fiber structure with unbroken fiber monomers. The cellulose surface has cavities which are pores in cellulose. At magnifications of 10,000× and 20,000×. pineapple peel cellulose morphology shows an unbroken structure. This once again shows that the process of adding a strong base and acid hydrolysis does not trigger the breaking of the cellulose chain.

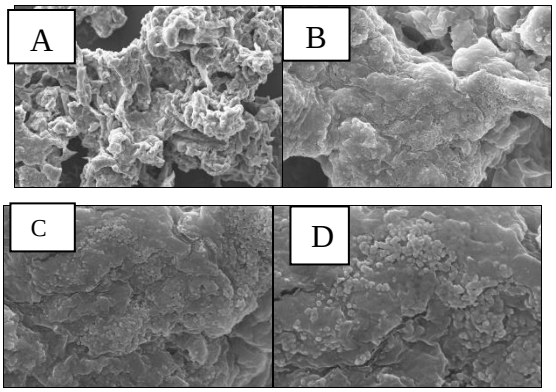


Figure 4. Morphology of pineapple peel cellulose

The elements contained in the analyzed cellulose are displayed on the EDX spectrum (Figure 5). In this spectrum it is known that pineapple peel cellulose contains elements C, O, Na, Al, S, K, and Ti. Based on Figure 4 it can be seen that the elements C and O are the main components making up the structure of cellulose. The presence of Na could come from the NaOH solution during the delignification process. While the other elements, Al, K, and Ti, are possible inorganic elements that are already present in the pineapple peel and are not lost when the cellulose extraction process is carried out. The percentage of constituent elements of pineapple skin cellulose are C (18.76%), O (26.32%), Na (3.31%), Al (0.13%), S (7.69%), K (0.24%), and Ti (3.63%).

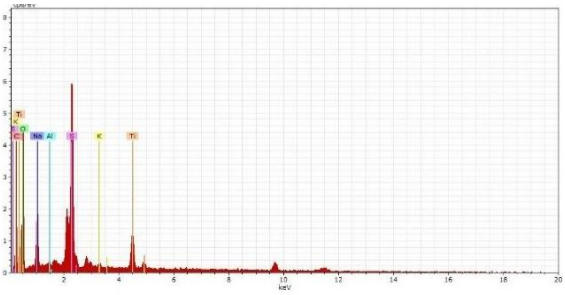


Figure 5. Elemental analysis of cellulose *X-Ray Diffraction (XRD)*

The XRD spectra of cellulose can be seen in Figure 6. This XRD analysis diffractogram will provide information about the structure of pineapple peel cellulose. Based on the data obtained, qualitative observations on the diffractogram show that the cellulose polymer compound is a mixture of cellulose with crystalline and amorphous structures. The XRD diffractogram peaks produce three broad peaks with high intensity with an angle of 2θ, namely 16.99°, 26.97°. and 45.41°. It is possible that the broadening of the peaks came from non-cellulose compounds. This is reinforced by the detection of Na, Al, S, K and Ti elements in the EDX spectrum, where these elements are not constituents of cellulose. The 2θ diffraction peak around 24–26° is characteristic of native cellulose, or cellulose I (Zhang *et al.*, 2019). Cellulose I is a natural form of cellulose which consists of 2 types, namely cellulose Iα and Iβ (Morán *et al.*, 2008).

Standard Calibration Curve

The maximum wavelength (λ_{max}) for RY standard solution is 412 nm. RY standard calibration curve was made in the concentration range of 0-25 mg/L at λ_{max}. The standard calibration curve RY can be seen in Figure 7.

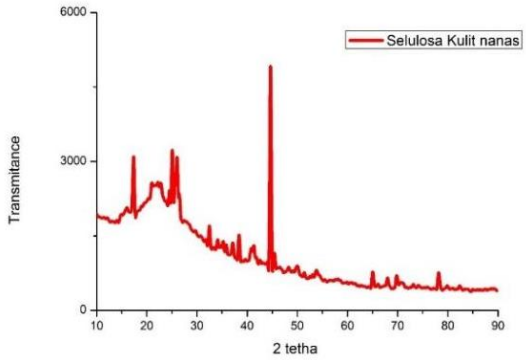


Figure 6. Difractogram XRD pineapple cellulose.

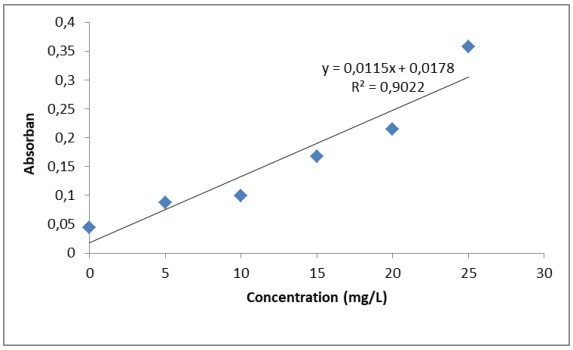


Figure 7. Standard calibration curve of RY.

The Effect of pH on Adsorption

The effect of pH on the adsorption of RY indicated that the adsorption capacity of pineapple peel cellulose was higher at pH 2, which was 0.8119 mg/g (Figure 8).

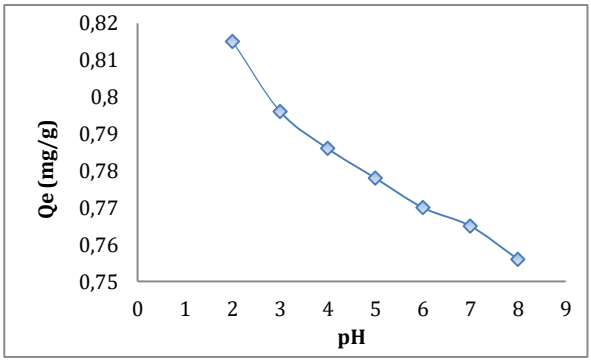


Figure 8. The effect of pH on adsorption RY

The highest adsorption capacity is obtained at pH 2. At low pH there is an increase in the concentration of H^+ in the solution which supports the protonation of the matrix surface, thus increasing the electrostatic attraction between the matrix and the dye (Lucinaldo S Silva *et al.*, 2018). At low pH, the sulfaethylsulfonat (-$SO_2CH_2CH_2OSO_3Na$) group of the dye will change be vinyl sulfonate group (-$SO_2CH=CH_2$). It is group can interact with the cellulose surface to form covalent bonds (Lucinaldo S. Silva *et al.*, 2018). As the pH increases, there will be a decrease in the adsorption capacity of pineapple peel cellulose on remazol yellow. An increase in pH will increase the concentration of OH- in solution so as to reduce the electrostatic attraction between cellulose and dyes as a result of reduced surface protonation of cellulose. This is in accordance with research by Lestari et al where adsorption of RY dye using activated carbon was obtained at pH 2 (Lestari, Prasetyo and Gusti, 2021).

Effect of Contact Time on Adsorption

The contact time is the time required for the interaction between the adsorbent and the adsorbate occur and reached an equilibrium state. Based on the results of the adsorption capacity analysis for time variations (Figure 9), it was found that the highest adsorption capacity was at 30 minutes of contact time, amounting to 0.7816 mg/g. The adsorption capacity at a contact time of more than 30 minutes has decreased, this is due to the fact that not all of the RY dye is electrostatically bound (chemical adsorption) but there is also an assumption that physical adsorption will occur where RY dye will be released back on the surface of the adsorbent when adsorbed has reached equilibrium (Sehaqui *et al.*, 2014).

The Effect of Concentration on Adsorption

The amount of RY adsorbed onto the surface of the adsorbent increased with increasing solution concentration. The solution concentration used to study this effect is 5-100 mg/L (Figure 10). The highest adsorption capacity occurred at a concentration of 100 mg/L with adsorption capacity is 19.88 mg/g. The higher of concentration adsorbate, at a high concentration of solution, the driving force of the adsorbate will be higher towards the active side of functional group in the cellulose surface, thereby increasing the adsorption capacity (Xia *et al.*, 2019)(Mandal and Chakrabarty, 2011).

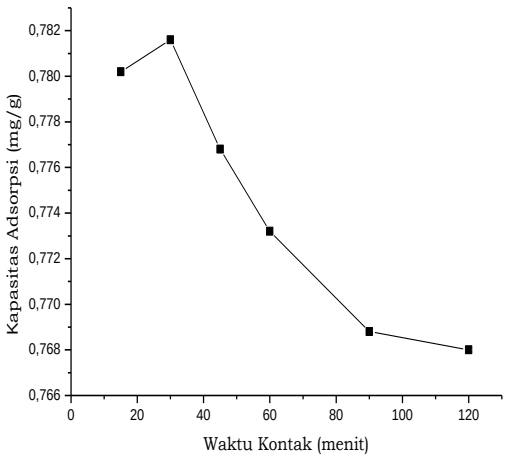


Figure 9. The effect of contact time on adsorption capacity on RY

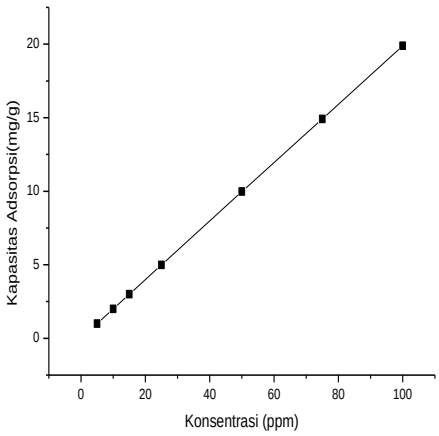


Figure 10. The effect of concentration on adsorption capacity Remazol Y.

Conclusion

The cellulose of pineapple peel have been success extracted through delignification and hydrolysis processes. The cellulose characterization using FTIR and SEM-EDX which confirmed the presence of functional groups and surface morphology of pineapple peel cellulose. The ability of cellulose to adsorption of RY dyes was determine by studied of adsorption parameters such as pH, contact time and solution concentration.

The RY adsorption was obtained at pH 2, contact time of 30 minutes and solution concentration of 100 mg/L with adsorption capacity was 19.88 mg/g. The cellulose of pineapple peel has potential used as a RY dye adsorbent.

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