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Synthesis of Cellulose Acetate-Polystyrene Membrane Composites from Pineapple Peel Wastes for Methylene Blue Removal

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***Abstract**: The cellulose acetate-polystyrene or CA-PS membrane composite from pineapple peel waste for methylene blue removal has been conducted. The steps were nata de pina preparation, cellulose acetylation process, preparation, and characterization of CA-PS membrane composites. The CA-PS membrane composite was characterized using Fourier Transform Infrared (FT-IR), Scanning Electron Microscopy (SEM), tensile and strain examination, respectively. The as-synthesized CA-PS membrane composite has the characteristic of rejection ability was about 29.96% with the pore size, membrane modulus, stress and strain were 1.9 μm, 12.48 MPa, 31.91 MPa, and 2.55, respectively. In this research, CA-PS membrane composite from pineapple peel waste was successfully removed the methylene blue dye even needs improvement to enhance its capability in rejection efficiency as same as membrane characteristics.*

Keywords: *cellulose acetate-polystyrene (CA-PS), dye removal, membrane composite, methylene blue, pineapple peel waste*

1. INTRODUCTION

In the textile industry, methylene blue is one of the thiazine-dyes which was always used because of its low-price and easy to use. Methylene blue dye is a green hydrated crystal that can absorb into plant roots easily than the other dyes such as Orange-1. It could be a source of organic-pollutants which should be removed from the water (Akarsu et al., 2006). The irresponsible use of methylene blue dyes caused cyanosis and skin irritation when contacting with the dye or water containing dyes (Hamdaoui & Chiha, 2007), increased heart rate, vomiting, shock, Heinz body formation, jaundice, quadriplegia and tissue necrosis in human (Hameed, Ahmad, & Latiff, 2007; Vadivelan & Kumar, 2005).

To decrease the environment pollutant in water containing dyes, there are some attempt such as electro-decolorization process (Amal, Febiyanto, Soleh, & Afif, 2016; Khosravi, Fazlzadehdavil, Barikbin, & Hossini, 2015), photocatalysis reaction (Febiyanto, Eliani, Riapanitra, & Sulaeman, 2016; Febiyanto et al., 2019), adsorption (Ikhsani, Santosa, & Rusdiarso, 2016; Subramani & Thinakaran, 2017), separation

The method used membrane technology (Baslak, Arslan, Kus, & Cengeloglu, 2016; Lin et al., 2016), etc. Nowadays, because of its process that can be done through

a continuous process, no need for chemical auxiliaries, low-energy consumption, and could not lead material destruction causing the fast development of membrane technology in water pollutant treatment (Mulder, 1996).

In Indonesia, pineapple (*Ananas comosus* L.) fruit was widely known for food and industry needs. Based on the data from Badan Pusat Statistik (BPS) Indonesia (2019), stated that the total production of pineapple fruit increases year by year. In 2018, the total production of pineapple fruit was about 1.805.506 tons higher than in 2017 and 2016 with the total production of 1.795.986 and 1.396.153 tons, respectively. At least, the enhancement of pineapple production would increase the pineapple peel wastes that could also lead the environmental pollution in the future. An effort to minimize the pineapple peel waste is to make *nata de pina* as a raw material source in cellulose acetate-based membrane synthesis. Cellulose acetate-based membrane has characteristics that are hydrophilic, no-smell, non-toxic, tasteless, white-colored solid and promising material such as anti-fouling (Goetz, Jalvo, Rosal, & Mathew, 2016) especially in term of protein and lipid (Soemantri, 2003), well-performance under sodium hypochlorite and sanitation (Scoot & Hughes, 1996), and high salt and flux rejection (He et al., 2009). In addition, the cellulose acetate is one of the environmental membranes than a synthetic membrane, biodegradability, chemical resistant, and thermal stability (Nair & Mathew, 2017). Moreover, the waste of pineapple peel can be converted into *nata de pina* via a fermentation process using *Acetobacter xylinum* bacteria. Nevertheless, *nata de pina* from pineapple peel waste as a source in the synthesis of cellulose acetate membrane was not sufficient reported especially about their performance in the dye removal.

Besides, the membrane modification in membrane synthesis aimed to produce new properties or increases their membrane capabilities (El-Din, El-Gendi, Ismail, Abed, & Ahmed, 2015; Kamal, Abd-Elrahim, & Lotfy, 2014). Meenakshi et al. (2002) and Rosdi, Mohd Kanafi, & Abdul Rahman (2018) reported that cellulose acetate is modified by the polystyrene has mechanical properties, tensile strength, and stability better than unmodified cellulose acetate. Therefore, herein we reported the cellulose acetate-polystyrene or CA-PS membrane composite preparation from pineapple peel waste and their characteristic in methylene blue removal will be presented in this research.

2. RESEARCH METHODOLOGY

2.1. Materials

Materials were pineapple peel waste, *Acetobacter xylinum* bacteria, $(NH_4)_2SO_4$, glacial acetate acid, anhydride acetate, acetate acid 67%, concentrated H_2SO_4, acetone, dichloromethane, phenol 5%, and distilled water. Here, solutions were used without further purification before.

2.2. Method

2.2.1. *Nata de pina* preparation

The pineapple peel was washed, cut into small pieces, and blended into smooth. Then, the sample was filtered and squeezed using mori cloth resulting in a liquid concentrate. The concentrate was filtered using Buchner funnel and heated until the concentrate was boiled up. The concentrate subsequently was added 500 grams of sugar. The solution was allowed and then added 25 grams of $(NH_4)_2SO_4$ and 30 mL of glacial acetate acid. The solution named bacterial media was set at a pH of 4-5 and poured into a 1.5 L of the container, covered with paper and then stored for 24 hours. An appropriate 10% *Acetobacter xylinum* bacteria (%v/v of media volume) was introduced into the above bacterial media and covered again with paper. The bacterial media was incubated at room temperature for 8 days for fermentation processing. Subsequently, the as-resulted *nata de pina* was purified.

2.2.2. Cellulose acetylation process

Cellulose acetylation process followed Masaoka, Ohe, & Sakota, (1993) which an appropriate of 5 grams of bacterial cellulose which has been mashed before (Fig. 1 (b)) is added with 12 mL of glacial acetic acid and mixed for 1 hour. Then, the sample is added by the mixture of 0.088 mL of concentrated H_2SO_4 and 20 mL of glacial acetate acid and then mixed for 45 minutes. The mixture is left cold until the media temperature of 18.3 °C and then added with 13.5 mL of anhydride acetate (15.6 °C). Subsequently, this is added with a mixture of 0.612 mL of concentrated H_2SO_4 and 20 mL of glacial acetate acid and mixed for 20 minutes until the solution is obtained. Afterward, the solution is added slowly droplets of 15 mL of acetate acid 67% for 1 hour at 37.8 °C under mixing treatment. The solution is left for 20 hours and added with distilled water under mixing treatment until the precipitate has resulted. The as-resulted precipitate is stored for 10-15 minutes, washed with distilled water up to neutral condition, and filtered again. The precipitate is dried in the oven at 50-60 °C.

2.2.3. Preparation of cellulose acetate-polystyrene (CA-PS) membrane composite

Firstly, the preparation of the CA-PS-polymer solution. A polymer of CA 15% (%w/v) and PS 10% (%w/v) with the ratio of 9:1 was mixed in the solution mixture of dichloromethane and acetone (1:1). The mixture was mixed using stirrer magnetic until the solution was homogeneous. Then, the polymer solution was poured above the glass plate (18x18 cm) which masking tape on each side to get an appropriate depth, and printed by pressing and pushing that solution until a thin layer of membrane polymer was obtained. Subsequently, the adhered-polymer on the glass plate was left for 15-30 minutes to evaporate the solvent. The thin polymer was soaked in distilled water and labeled as cellulose acetate-polystyrene (CA-PS) membrane composite.

2.2.4. Characterization of cellulose acetate-polystyrene (CA-PS) membrane composite

2.2.4.1. Tensile and strain examination

Both ends of the CA-PS membrane composite were clamped on the autograph tools. One of the membrane ends was held firmly. Then, the other membrane end was pulled up until break and the maximum force was recorded. Membrane area was determined during the tensile test taking place.

2.2.4.2. Functional group and morphology analysis

The as-synthesized CA-PS membrane composite was characterized using Fourier Transform Infrared spectroscopy (FTIR) and Scanning Electron Microscopy (SEM).

2.2.5. Determination of rejection efficiency

2.2.5.1. Determination of calibration standard

Methylene blue standard was carried out by varying the solution concentration of 10, 40, 70, 100, 130, 160, 190, 220, and 250 mg/L of methylene blue solution and then added a solution of phenol 5% and concentrated H_2SO_4 with the ratio of 1:1:5 into 10 mL volumetric-flask. The mixture was diluted up to boundary mask using distilled water and subsequently measured by means UV-Visible spectroscopy.

2.2.5.2. Application in dead-end tools

The CA-PS membrane was cutting off into a circle form with a diameter of about 7 cm. The CA-PS composite membrane was placed below the filtration cells which was placed a filter paper before. An appropriate of 100 mL of methylene blue was introduced into the dead-end tools with a flow pressure of 1-5 kg/cm^2.

2.2.5.3. Determination of sample rejection

The methylene blue concentration, the effluent after filtration by the CA-PS membrane composite was measured their absorbance and concentration using UV-Visible spectroscopy. The resulted-values were plotted into calibration standards and then calculated their rejection efficiency.

3. RESULTS AND DISCUSSION

3.1. *Nata de pina* preparation

Preparation of *nata de pina* includes media preparation, bacteria inoculation, and cellulose sheet purification, respectively. For media preparation, the pineapple peel extract was boiled to restrict the contaminant on the bacterial media. Subsequently, adding by ammonium sulfate 10% and sugar as nitrogen and carbon sources to obtain the extracellular polysaccharide enzyme, respectively. The pH optimum of 4 for *Acetobacter xylinum* growth was set by using glacial acetate acid. Pineapple peel filtrate was poured into the above sterile-container to inhibit the contamination and

bacterial growth. After 24 hours, the *nata de pina* sheet was added by *Acetobacter xylinum* 15% (%v/v of media volume total) so that the completed-fermentation was achieved.

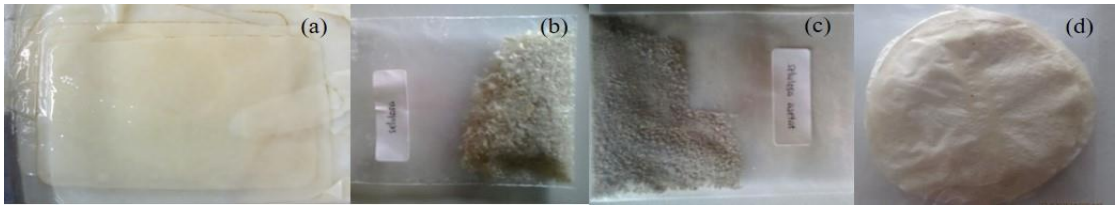

Figure 1. *nata de pina* (a), microbial cellulose powder (b), cellulose acetate (c) and CA-PS membrane composite (d), respectively.

As shown in Fig. 1 (a), the result showed that *nata de pina* can be harvested on the 7th day of incubation. Based on Fig. 1 (a), the as-resulted *nata de pina* sheets have a yellowish and approximately 0.2 cm in thickness. Then, *nata de pina* was washed with flowing water to remove the contaminant (like glucose and protein) along the *nata de pina* surfaces. *Nata de pina* was also purified using NaOH 1% (%w/v) solution for 24 hours for deactivating the microorganism such as the remaining bacteria, neutralized by soaking those sheets in CH_3COOH 1% (%v/v) solution for 24 hours and washed again with distilled water several times. Noted that the addition of NaOH 1% increased swelling and crystallinity of membrane as well as decreased their membrane properties such as low-acetylation capability. However, the addition of CH_3COOH 1% was benefits to neutralize and decreased the crystallinity of the membrane (Syamsu & Kuryani, 2014). Subsequently, *nata de pina* was pressed using "*hand-press*" tools resulted in a *nata de pina* thin layer and dried in air at room temperature of about 27-30 °C. Pressing treatment was not carried out at high temperature so there is no damage to the molecule chain structure in the *nata de pina* samples (Lindu, Puspitasari, & Ismu, 2010). The dried *nata de pina* was named microbial cellulose (Fig. 1 (b)).

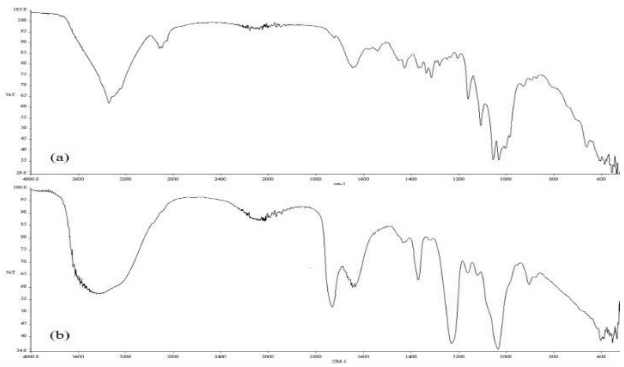

Figure 2. FTIR spectra of cellulose (a) and cellulose acetate (b), respectively.

3.2. Cellulose acetate production

The cellulose acetate synthesis from microbial cellulose contains four steps that are activation, acetylation, hydrolysis, and purification. The activation aims to swell the cellulose structure resulting in the fast and efficient of the acetylation process using glacial acetic acid (Syamsu & Kuryani, 2014). Rachmawati (2007) stated that the acetylation process is required a water-free or less system condition because the esterification is a reversible process. The high-water content in cellulose increases the hydrolysis rate and inhibit acetate cellulose production.

The acetylation process was conducted by adding the anhydride acetate and the mixture of concentrated H_2SO_4 and glacial acetate acid (Fischer et al., 2008). The purpose of the acetylation process was the elimination of a hydroxyl group of cellulose to form an acetyl group (CH-CO) through ester linkage to produce cellulose acetate (Meenakshi et al., 2002; Vaulina, Widyaningsih, Kartika, & Romdoni, 2018). Anhydride acetate reacted with sulfuric acid resulted in protonated-carbonyl of anhydride acetate. Then, the nucleophilic substitution between free-electron pairs of oxygen of cellulose with as-resulted carbocation was taking place immediately. It will be followed by the elimination of H^+ and carboxylate acid.

In the next step, the hydrolysis process was carried out by adding acetate acid 67% into the resulted acetylation solution. According to Pandele et al. (2018), the hydrolysis process is to eliminate part of the acetyl group and dismiss the acetylation process. Fig. 1 (c), the precipitated-acetate cellulose was light brown. The powder of cellulose acetate was then prepared for functional group analysis.

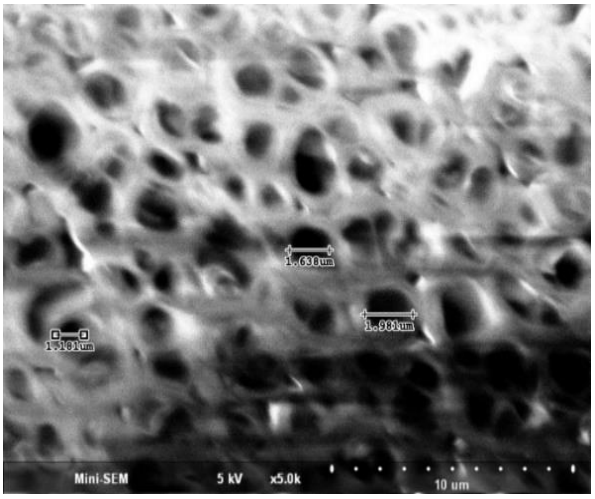

Figure 3. SEM image of cellulose acetate-polystyrene (CA-PS) membrane composite.

3.3. Cellulose acetate-polystyrene (CA-PS) membrane composite preparation

Membrane composite of cellulose acetate-polystyrene or CA-PS was done by the inversion-phase method. The inversion-phase is controlled-process that polymer changes from solution to solid-state (Mulder, 1996). The CA-PS was conducted by mixing the cellulose acetate polymer 15% (%w/v) and polystyrene 10% (%w/v) in the mixture of dichloromethane and acetone with the ratio of 1:1. A thin layer of the CA-PS membrane composite has resulted as shown in Fig. 1 (d).

3.4. Characterization of cellulose acetate-polystyrene (CA-PS) membrane composite

Fig. 2 (a) shows the FTIR spectra of the cellulose sample. As shown in Fig. 2 (a), the adsorption of –OH and –CO group have observed in 3342 and 1108 cm^{-1} attributed to the glycoside and cellulose ring, respectively. Meanwhile, the IR-absorption of cellulose acetate was presented in Fig. 2 (b). Based on Fig. 2 (b), the strong absorption could be identified at 1732 and 1231 cm^{-1} attributed to the stretching of ester carbonyl and C–O bond (Candido, Godoy, & Gonçalves, 2017), whereas the C–H in –O(C=O)–CH_3 has appeared at 1384 cm^{-1} (Daud & Djuned, 2015). Rahmawati, Fadillah, & Mudjijono (2017) stated that the presence of the C–O group indicated the O–H group in the cellulose molecule was successfully substituted by =CO producing the cellulose acetate. It concluded that the cellulose acetate was successfully synthesized.

The morphology of the as-resulted CA-PS composite membrane has been conducted and depicted in Fig. 3. Based on Fig. 3, CA-PS has pore size was approximately 1.9 μm (1.900 nm). However, the formation of the pore size was asymmetry and randomly. This result suggested that the large-pore size was observed in CA-PS composite membrane and has not belonged to the commonly known-membrane that were microfiltration (50-500 nm), ultrafiltration (2-50 nm), nanofiltration (2 nm ≥), and reverse osmosis (0.3-0.6 nm) (Adán, Marugán, Mesones, Casado, & van Grieken, 2017).

3.5. Tensile and rejection efficiency characteristic of cellulose acetate-polystyrene (CA-PS) membrane composite

Fig. 4 shows the tensile characteristic of cellulose acetate-polystyrene (CA-PS) membrane composite. The tensile strength indicates a strong or weak membrane when pulled by a tool. The strongly pulls attributing to the good performance of CA-PS in membrane technology application. Modulus values is a mechanical property that can be seen in this tensile examination. Modulus is a strength of material endurance against the changes in shape. Based on the calculation, resulting in the membrane modulus, stress and strain of the CA-PS membrane were about 12.48 MPa, 31.91 MPa and 2.55, respectively.

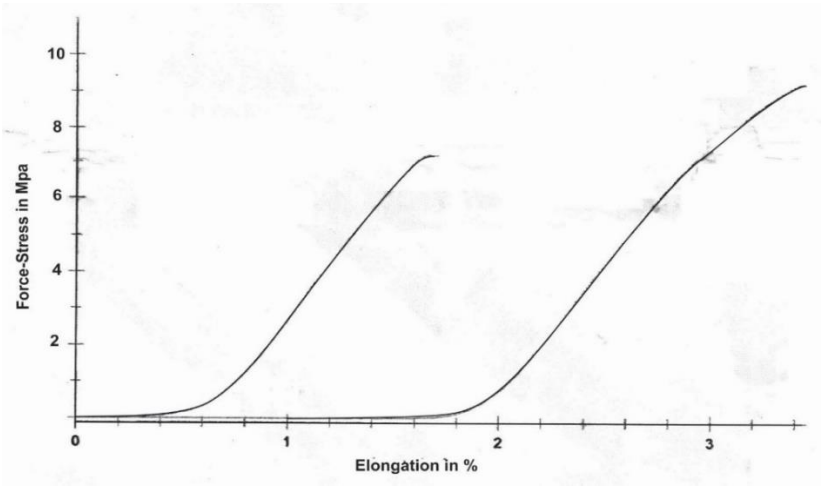

Figure 4. the as-resulted tensile characteristic of cellulose acetate-polystyrene (CA-PS) membrane composite.

Rejection is an ability of the membrane to retain the component so that it cannot flow through the membrane (Juansah, Dahlan, & Huriati, 2009). Rejection value of the sample is performed by the equation as follows: $R(\%) = 1 - (C_A/C_B) \times 100\%$ where R(%) is rejection efficiency, C_A and C_B are the dye concentration after and before filtration using CA-PS membrane composite, respectively. The increase of R(%) increases the selectivity of the membrane in particle separation (Mulder, 1996). The result showed that the maximum absorbance measurement of methylene blue at around 660 nm (λ) (Dhananasekaran, Palanivel, & Pappu, 2016), resulted in the rejection efficiency of about 29.96%. This result shows a low-rejection efficiency even it was higher than the rejection efficiency of the CA-PS membrane from the banana peel (Ayusnika et al., 2014). The low-rejection ability might be caused by the pore size of the CA-PS membrane composite that is too large than the molecular size of MB dye (13-15 Å) (Attia, Girgis, & Fathy, 2008). So, the dyes could easily flow through the membrane sheet and then lowering the rejection ability of the membrane. However, soon, this should be improved by some additional treatments to enhance the rejection efficiency of the CA-PS membrane composite.

4. CONCLUSIONS

The cellulose acetate-polystyrene membrane or CA-PS composite was successfully synthesized and carried out using pineapple peel waste. The sample has pore size, membrane modulus, stress and strain of CA-PS membrane composite were 1.9 μm, 12.48 MPa, 31.91 MPa and 2.55, respectively. The result showed that the rejection ability of the CA-PS composite membrane was about 29.96%. In this research, CA-PS membrane composite from pineapple peel waste was successfully

removed the methylene blue dye even needs additional treatment (such as variation of PS concentration, pore size modification, etc.) to increase the capability and selectivity of CA-PS membrane composite in the near future.

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