

Influence of Cellulose Acetate Concentration on The Performance of Biopolymer Membranes for Naphthol Dye Removal from Textile Wastewater

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ABSTRACT

Membrane technology has continuously advanced as a promising method for treating industrial wastewater, particularly in the textile sector, which generates large volumes of hazardous and toxic effluents. Textile wastewater typically contains high concentrations of complex organic and inorganic pollutants, with dyes being the most persistent and difficult to remove due to their stable aromatic structures. Compared to conventional treatment processes, membrane-based separation offers notable advantages, including lower energy consumption, a smaller footprint, and the ability to efficiently separate dissolved contaminants. In this study, membranes were fabricated via the phase-inversion technique to evaluate their potential for textile wastewater treatment. The membrane preparation involved varying the cellulose acetate (CA) concentration to 13%, 14%, and 15%, while polyethylene glycol (PEG) at 3% was incorporated as an additive to improve pore formation. Acetone was used as the primary solvent during casting. Comprehensive analyses of membrane characteristics—including water uptake, porosity, pure water flux, and dye rejection—were conducted to determine the relationship between polymer concentration and membrane performance. The findings indicate that the membrane with 13% CA exhibited the highest pure water flux, reaching 87.9483 L/m²·h, suggesting greater permeability due to increased pore formation. Conversely, the membrane prepared with 15% CA demonstrated the highest dye rejection, achieving 30.72% for a 20 ppm naphthol dye solution. These results highlight the inherent trade-off between permeability and selectivity: increasing polymer concentration enhances contaminant rejection but reduces membrane flux. The study provides valuable insights for optimizing membrane formulation for textile wastewater treatment applications.

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1. Introduction

The development of the industrial sector has increased wastewater, potentially polluting the environment. The continuous innovations in wastewater treatment. Membrane technology is one of the processing techniques that continues to be developed in treating industrial wastewater, especially in the textile industry. The textile industry generally consumes large amounts of water at every stage of production, especially during the coloring process. The dye solution used to bind to textile fabrics

is only 5% and the remaining 95% will be disposed of as wastewater [1], [2]. Wastewater is toxic and harmful to the environment because it contains high levels of synthetic colorants, high COD, and high metal content [3]–[5]. In the coloring process, many types of coloring materials are used; one of the dyes commonly used in the textile industry is naphthol dye [6].

Naphthol dyes are widely used in textiles, batik, plastics, paper, and other industries, and they require close attention in waste treatment because naphthol-dye-type liquid waste is harmful to the environment and health [6]. There are many methods commonly used in textile wastewater treatment; one of them is membrane technology. Membrane technology involves polymers [7]. This method has been widely used in various industries for the separation and purification [2], [8], [9]. It can serve as an alternative to conventional separation processes [10], [11].

Membrane technology offers several advantages, including: biopolymeric in nature, simple process, very low energy consumption, does not damage the material, no chemicals are involved, and no new waste is generated. Membrane technology is classified as clean technology [12]–[16]. Membranes are divided into three types, namely: microfiltration [17]–[19], nanofiltration [20]–[22], and ultrafiltration [23]–[26].

Many raw materials can be used to make this biopolymer membrane, including cellulose derivatives such as cellulose acetate (CA). The CA is a biopolymer material [25] and is one of the cellulose derivatives of organic acids [27]. The CA biopolymer has high commercial value due to its excellent physical and optical properties, making it suitable for the manufacture of plastics, films, and membranes. It can dissolve in acetone, thermoplastic, and has good stability and durability in the form of plastic layers [25], [28].

The CA material is a macromolecule derived from cellulose, a natural polymer with an organized microfibril structure. It has various advantages, including high transparency, good tensile strength, heat resistance, low water absorption, and biodegradability. These properties are an indication that CA is indispensable for use in industrial fields such as coatings, plastics, films, textile fibers, and textile membranes [29]–[31]. CA is a semi-synthetic polymer that is flexible, renewable, non-toxic, easy to make or produce in the manufacture of biopolymers, raw materials are available from renewable natural sources, and can be created easily [8], [32], [33].

The fabrication and characterization of the CA membrane have been studied previously [14]. The study has focused on the membrane's physical and chemical properties. Although CA membranes have been extensively studied, systematic evaluation of CA concentration (13–15%) on performance toward small naphthol dye molecules remains limited. This study systematically investigates the influence of CA concentration (13–15%) on water uptake, porosity, flux, and rejection performance, specifically for the naphthol dye, a small organic molecule commonly present in textile wastewater.

2. Research Methodology

2.1. Materials

The materials were used in membrane manufacturing, such as CA from Sigma Aldrich, acetone (industrial grade), polyethylene glycol (PEG) 4000, formaldehyde (industrial grade), and distilled water. The rejection analysis was carried out using naphthol blue black (CAS Number: 1064-48-8).

2.2. Procedures

1) Cellulose Acetate Membrane Preparation

The CA membrane was prepared using the phase-inversion method. The composition of the CA membrane includes CA, PEG 4000, distilled water, and acetone. The compositions of the membrane dope are presented in Table 1.

Table 1. Composition Variation on Cellulose Acetate Membrane

CA (%)	PEG 4000 (%)	Acetone (%)	Distilled water (%)	Membrane thickness (μm)
13	3	83	1	200
14	3	82	1	200
15	3	81	1	200

The PEG functions primarily as a pore-forming agent to control membrane structure and performance. It improves porosity, increasing permeability and flux, and its water-soluble nature helps form pores during the phase inversion process. Additionally, PEG enhances a membrane's mechanical strength, hydrophilicity, and anti-fouling properties [34].

The CA membrane fabrication was initiated by preparing a dope solution consisting of CA, PEG 4000, acetone, and distilled water, which was homogenized for 4 hours at 30°C. After the homogenizing process, the solution was left for 12 hours. The dope solution was printed using a printing knife with a 200 µm thickness on a glass surface, then dipped directly into a coagulation bath containing distilled water until the membrane was released. The detached CA membrane was then dried at room temperature.

Before being applied for wastewater treatment, the membrane underwent thermal annealing treatment for 30 seconds at 60°C. The thermal annealing step was performed to stabilize the polymer matrix, reduce surface defects, and control pore size uniformity [35].

2) Water Uptake and Porosity Analysis

The CA membrane sheet was cut into a 4.8 cm diameter circle, weighed, and recorded as the dry weight. The membrane was placed in a closed container containing distilled water for 24 hours. The membrane was weighed and recorded as the wet weight. The water uptake and porosity were calculated using equations 1 and 2:

$$\%WU = \frac{W_{wet} - W_{dry}}{W_{dry}} \times 100\% \quad (1)$$

Where WU is water uptake value (%), W_{wet} is wet membrane weight (g), W_{dry} is dry membrane weight (gram) [36], [37].

The porosity of the CA membrane was calculated using equation 2.

$$\varepsilon = \frac{W_1 - W_2}{A \times l \times \rho_w} \times 100\% \quad (2)$$

Where ε indicates the porosity value (%), W_1 is the membrane weight when wet (g), W_2 is the membrane weight when dry (g), A is the membrane area (cm²), l is the membrane thickness (cm), and ρ_w is the density of water (g/cm³) [25].

3) Flux Analysis

The CA membrane sheet was cut into a 4.8 cm diameter circle. This analysis was conducted using a standard dead-end filtration unit with an effective filtration area of 18.0864 cm². The flow from the filtration device was collected and considered permeate. The flux was calculated using Equation 3.

$$J = \frac{V}{A \times t} \quad (3)$$

Where J is flux analysis value (L/m².h), V is permeate volume (L), A is area of membrane (m²), and t is permeation time (hour) [25], [36]–[38].

4) Rejection Analysis

The rejection test was conducted using a dead-end filtration unit that operated under a vacuum of 0.8 atm. The naphthol dye solution was used as artificial wastewater with concentrations of 20 ppm and 30 ppm.

The naphthol concentrations of permeate were analyzed using a UV-vis spectrophotometer, Genesys 150 UV-Vis Spectrophotometer. The rejections of the CA membrane were calculated using Equation 4.

$$R = \frac{C_u - C_p}{C_u} \times 100\% \quad (4)$$

Where R is rejection value (%), C_p is permeate concentration (ppm), and C_u is feed solution concentration (ppm) [2], [39].

3. Results and Discussion

3.1. Water Uptake and Porosity Analysis

Water uptake analysis was carried out to assess membrane performance in water absorption [8], while porosity analysis was used to quantify the membrane's pore area [40], [41]. Higher water uptake and porosity values indicate more porous surfaces in the membrane, so the amount of permeate that can flow through the membrane at one time will also increase [40], [42]–[45]. The water uptake and porosity analysis are presented in Fig. 1.

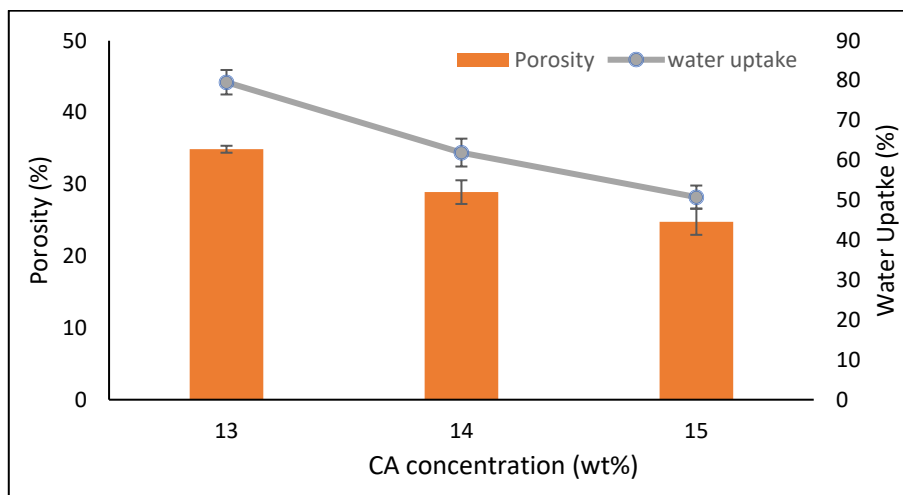


Fig. 1. Example Results of Water Uptake Analysis and Porosity Analysis on Membranes with Concentrations of 13%, 14%, and 15% CA

It is found that the highest water uptake and porosity values are in the membrane with 13% CA. These results show that the membrane's porosity and water uptake are influenced by polymer concentration, with higher concentrations resulting in lower porosity and water uptake. A lower concentration of CA leads to a less dense membrane, making it more porous. This fact is in accordance with the result of previous research [13], [14], [46] where the greater the polymer concentration, the smaller the water uptake and porosity values.

3.2. Pure Water Flux Analysis

Pure water flux analysis is a method used to determine the volume of water passing through the membrane surface per unit time. Pure water flux analysis can be calculated using equation 3. The values of pure water flux for CA membranes are presented in Fig. 2.

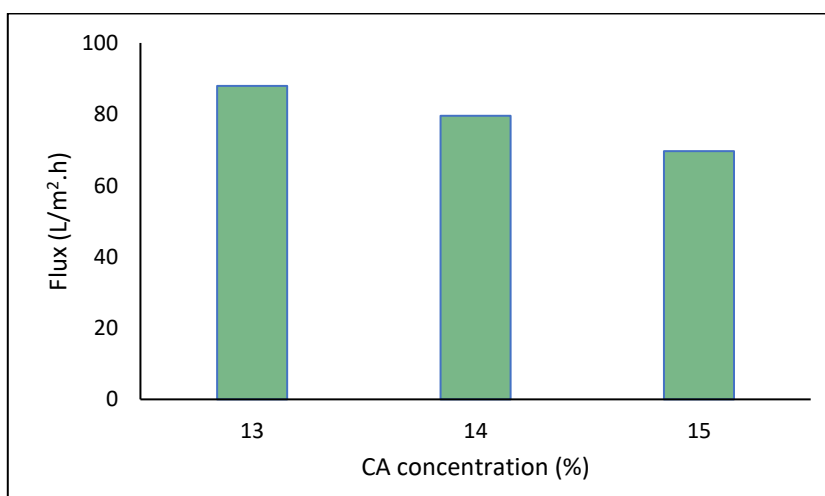


Fig. 2. Example Flux analysis of pure water on membranes with concentrations of 13%, 14%, and 15% CA.

The figure shows that the membrane with 13% CA composition has a higher flux than those with 14% and 15% CA. This is in accordance with previous studies [8], [46], where the higher the concentration of CA in the membrane, the lower the flux value. A higher CA concentration means more polymer chains are present, which will increase the viscosity of the dope solution [47]. It leads to increased entanglement and more resistance to flow, resulting in a less porous membrane structure.

3.3. Rejection Analysis

Rejection analysis is conducted to determine the membrane's performance in filtering pollutants from naphthol dye textile wastewater. The results of the rejection analysis are shown in the Fig.3.

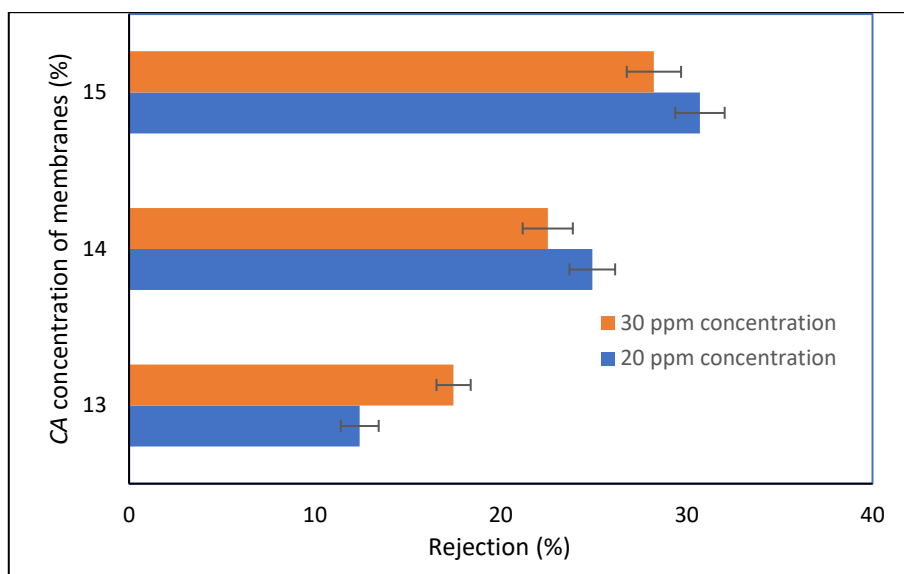


Fig. 3. Rejection of textile wastewater containing naphthol dyes at concentrations of 20 ppm and 30 ppm

Fig. 3 shows the results of the rejection analysis for the naphthol solutions at 20 ppm and 30 ppm as artificial textile wastewater using membranes with CA concentrations of 13%, 14%, and 15%. The results have revealed that the best rejection was achieved at the separation using a 15% CA membrane and 20 ppm naphthol, with a rejection value of 30.72%. The findings of this study are parallel with previous research [13], [48], [49] where a denser membrane structure (higher CA concentration) leads to smaller pores and longer diffusion pathways, enhancing dye rejection through size exclusion. As the polymer concentration in the membrane increases, porosity decreases, leading to higher rejection. The results of SEM analysis in previous research have also supported this fact [14], which showed that the CA 13% membrane had a larger size and more numerous pores, while the CA 15% membrane had a smaller size and fewer pores.

4. Conclusion

The CA membranes have been successfully fabricated. The concentration of CA in the membranes plays a key role in membrane performance. The membrane with 13% CA showed the highest water uptake (79.59%), porosity (34.88%), and flux (87.9483 L/m²·h). The 15% CA membrane provided the highest dye rejection (30.72%). These findings confirm that higher polymer concentration enhances selectivity but compromises permeability. These results demonstrate that higher CA concentration enhances selectivity but reduces permeability, indicating a trade-off between flux and rejection performance. The rejection value is relatively low and should be taken into consideration in subsequent research. Further studies are needed on various CA membrane compositions and their applications in various artificial wastewater before they are used in real wastewater.

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