

Morphology and selected properties of cellulose acetate membranes for environmental applications

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Abstract: Microporous membranes were obtained by dry-wet phase inversion from a solution of cellulose acetate (CA) in acetone (13, 14 and 15 wt%). Polyethylene glycol was used as a blowing agent. The structure and mechanical properties were examined. FT-IR spectra show that the addition of polyethylene glycol improves the thermodynamics of the solution and increases the hydrophilicity of the membrane. The SEM method confirmed the microporous structure of membranes with an asymmetric structure and various pore sizes and porosities. Higher CA concentration resulted in better tensile properties.

Keywords: biopolymers, cellulose acetate, membrane, phase inversion.

Struktura i wybrane właściwości membran z octanu celulozy do zastosowań środowiskowych

Streszczenie: Mikroporowate membrany otrzymano metodą inwersji fazy sucho-mokrej z roztworu octanu celulozy (CA) w acetonie (13, 14 i 15% mas.). Jako środek porotwórczy zastosowano glikol polietylenowy. Zbadano strukturę i właściwości mechaniczne. Widma FT-IR pokazują, że dodatek glikolu polietylenowego poprawia termodynamikę roztworu i zwiększa hydrofilowość membrany. Metodą SEM potwierdzono mikroporowatą strukturę membran o asymetrycznej budowie i różnej wielkości porów oraz porowatości. Większe stężenie CA skutkowało lepszymi właściwościami mechanicznymi przy rozciąganiu.

Słowa kluczowe: biopolimery, octan celulozy, membrany, inwersja faz.

According to the United Nations, more than 80% of wastewater in the world is released into the environment with inadequate treatment. This untreated wastewater contributes to water pollution, which affects ecosystems and biodiversity. The discharge of untreated wastewater into rivers, lakes, and oceans can lead to dead zones, where oxygen levels drop to the point that aquatic life can no longer survive. Sustainable Development Goal 6 (SDG 6) of the United Nations' 2030 Agenda for Sustainable Development aims to ensure access to clean water and sanitation for all [1]. Proper wastewater treatment is a critical component of achieving this goal. The economic costs of inadequate wastewater management are substantial. In industrial settings, improper wastewater management can lead to production losses resulting in significant financial burdens for companies.

One of effective and versatile technology for addressing a wide range of wastewater treatment challenges is

by using membranes. Membrane technology has arisen as a promising solution for wastewater treatment, providing various advantages such as high retention rates for contaminants, low energy consumption, and the ability to deal with various types of wastewater [2]. It is used in wastewater treatment processes to remove impurities and pollutants from wastewater, making it safe for disposal or reuse. Membrane-based wastewater treatment processes offer advantages such as high efficiency, compact design, and the capability to produce high-quality effluent [3, 4]. Membranes made from biopolymers are eco-friendly and sustainable alternative to conventional synthetic polymer membranes. Biopolymers are polymers derived from natural sources, often renewable resources like plants, microorganisms, and animals. These materials have gained attention in various fields, including water treatment, due to their biodegradability, low environmental impact, and potential for diverse applications.

The use of biopolymer membranes offers several advantages, including biodegradability, reduced environmental impact, and compatibility with biological systems. Cellulose, derived from plant sources, is one of the most abundant biopolymers on Earth. Cellulose-based

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membranes are used in various applications, such as ultrafiltration and microfiltration for water and wastewater treatment [5]. Cellulose acetate (CA) is one of cellulose derivative materials for membrane production. CA was one of the first polymers used in aqueous membranes, dating back to the invention of the Loeb-Sourirajan asymmetric membrane in the 1950s [6]. Subsequently, CA membranes have been effectively utilized in varied bio-separation systems, encompassing utilizations in ultrafiltration, nanofiltration, and reverse osmosis [7].

The versatility of CA is evident in its application in fabricating gas separation membranes due to its low cost, ease of handling, availability, and mechanical strength [8]. Furthermore, CA is a common and important material used in the fabrication of pressure-driven membranes, demonstrating its widespread utility [9]. The use of CA in membrane technology has also extended to the fabrication of ultrafiltration membranes for separation techniques in environmental applications such as water and wastewater treatment [10]. Additionally, the modification of CA membranes has been explored in order to tailor their surface to have special properties, thus enhancing their potential applications [11]. The synthesis and characterization of CA have been the subjects of extensive research, highlighting its importance as a renewable, environmentally friendly, and easily applicable membrane material [12, 13].

The phase inversion process is a widely used method for preparing asymmetric polymer membranes. During this process, the solubility parameter between the non-solvent and solvent plays a crucial role in affecting non-solvent and solvent exchange [14]. The factors governing the process of membrane formation are affected by thermodynamic principles, incorporating phenomena such as solvent-polymer-nonsolvent phase separation and the solubility interactions among these components [15]. The present study was focused on preparing CA membranes. The effect of concentration of polymer (CA) on the morphological structure of membranes was investigated. A range of characterization instruments has been employed for evaluating the mechanical and morphological properties of the membranes to study their potency for environmental applications, especially in separation techniques.

The aim of the work was to investigate the effect of cellulose acetate on the properties, morphology and performance of membranes meeting requirements such as gas separation or wastewater treatment.

EXPERIMENTAL PART

Materials

In the fabrication of microporous membrane, cellulose acetate (CA) (average molecular weight 30,000 g/mol) served as polymeric support and acetone was used as solvent. Both chemicals were obtained from Sigma-Aldrich

(Merck, Burlington, MA, USA). Polyethylene glycol (PEG) 4000 (Merck, Burlington, MA, USA) was selected as additive, while aquadest (CV General Labora, Yogyakarta, Indonesia) was used as non-solvent.

Preparation of dope solutions and CA flat-sheet membranes

The CA membrane was obtained using dry-wet phase inversion method. The CA polymer and acetone was mixed in a beaker at various concentrations of CA polymer (13, 14 and 15 wt%) to prepare the homogeneous dope solution sample. To prevent oxidation, the beaker was subsequently sealed with aluminum foil. The mixture was then continuously stirred at 150 rpm for 4 hours until a homogeneous mixture solution was achieved. The flat-sheet membranes were obtained by spreading of the dope solution at ambient temperature (26°C) on a glass plate using a casting knife with thickness of 200 µm. The membranes were then immediately immersed in distilled water and left for 24 hours to ensure accurate phase inversion.

Methods

Fourier-transform infrared (FT-IR)

Fourier-transform infrared (FT-IR) characterizations were conducted using Thermo Scientific Nicolet iS10 FT-IR spectrometer (Waltham, MA, USA).

Scanning electron microscope (SEM)

The surface and cross-section of membranes were observed by scanning electron microscope (JEOL JSM-6510LA, Tokyo, Japan). All measurements were performed at 15 kV.

Porosity

The porosity of the membranes was determined using the dry-wet method, which involves the absorption of distilled water by the prepared membranes. Initially, the dry membrane was weighed and immersed in distilled water for 24 hours. The membrane sample was then dried in an oven at 60°C for 48 hours and weighed again. Membrane porosity ε (%) was calculated using Equation 1 [16].

$$\varepsilon = \frac{w_1 - w_2}{A \cdot l \cdot \rho_w} \quad (1)$$

where w_1 and w_2 are the wet membrane weight (g) and the dry membrane weight (g), respectively. A is the area of membrane (cm²), l is the membrane thickness (cm), and ρ_w is density of water (0.998 g/cm³).

Tensile properties

Tensile properties were performed using the HZ-1007A universal testing machine from Dongguan Lixian Instrument Scientific Co. Ltd. (Dongguan, Guangdong, China) in accordance with ASTM D882 standard for thin

plastic sheets with a thickness of less than 1 mm, with a crosshead speed of 10 mm/min and a gauge length of 30 mm. Membranes with dimensions of 10 × 50 mm were used. The average of three measurements was taken as the result.

RESULTS AND DISCUSSIONS

Chemical structure

The effects of the interactions of the cellulose acetate, acetone, PEG 4000, and distilled water were evaluated by FT-IR spectroscopy as shown in Figure 1, while the list of functional groups was presented in Table 1.

The absorption patterns of the CA membranes revealed several essential functional groups. The spectra for CA membranes with three different polymer concentration show the presence of carbonyl (C=O stretching and R-C=O) at wavenumber 1735 cm⁻¹ and 1739 cm⁻¹; O-C-O bending at wavenumber 1215 cm⁻¹ and 1222 cm⁻¹; C-O stretching at wavenumber 1029 cm⁻¹, 1033 cm⁻¹, and 902 cm⁻¹; and C-H bending functional groups at wavenumber 902 cm⁻¹ [13, 17–19]. These functional groups are the characteristic of CA compounds. Furthermore PEG was introduced into the dope solution as an additive to enhance the selectivity of membrane and serve as a pore-forming agent. This compound was identified with the presence of O-H functional groups at wavenum-

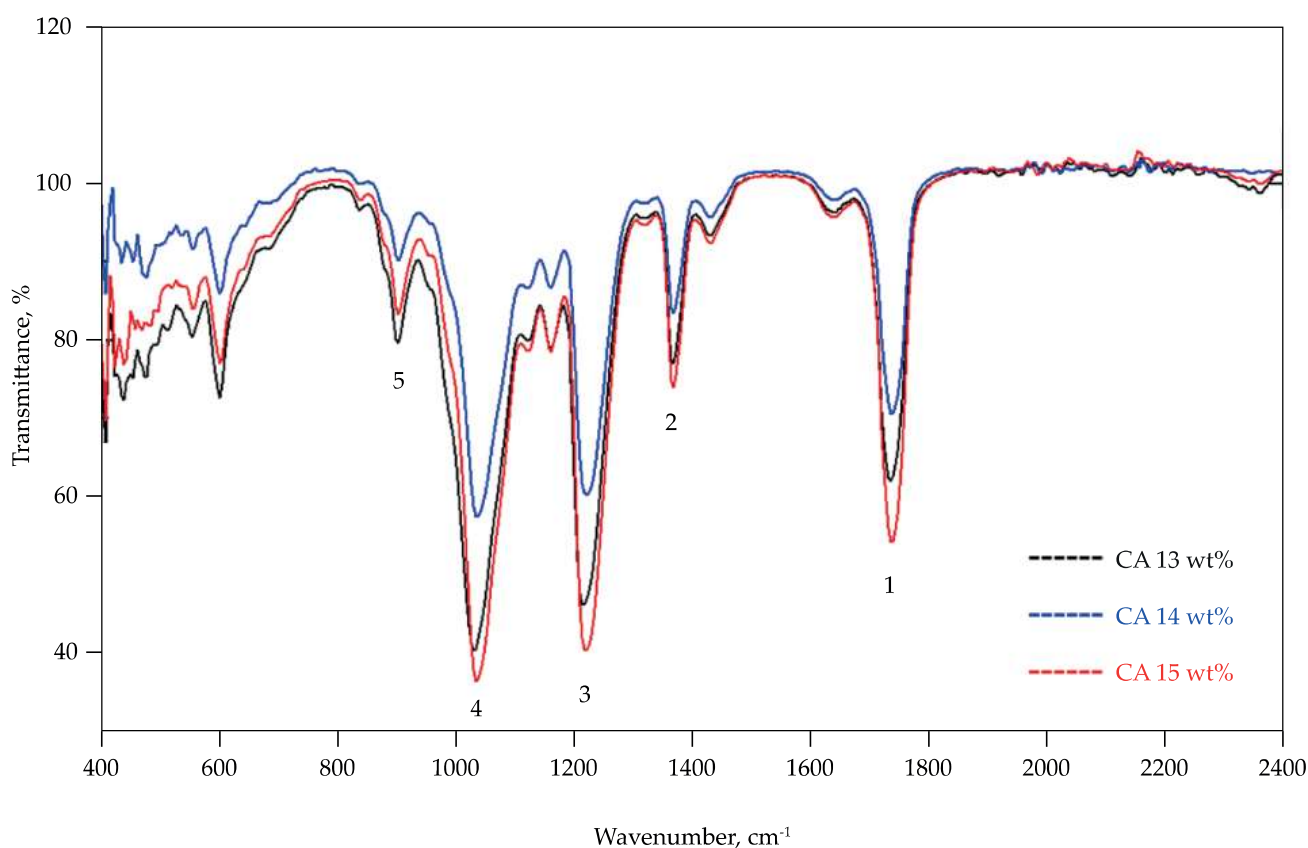


Fig. 1. FT-IR spectra of CA membranes

Table 1. Comparison of IR spectra of CA membranes

Peak Number	Functional Groups	Wavenumber, cm ⁻¹		
		CA 13 wt%	CA 14 wt%	CA 15 wt%
1	C=O stretching	1735	1739	1739
2	R-C=O	1369	1369	1369
3	O-C-O bending; O-H	1215	1222	1222
4	C-O stretching	1029	1033	1033
5	C-O stretching; C-H bending	902	902	902

ber 1215 cm^{-1} and 1222 cm^{-1} . During phase inversion, PEG addition could thermodynamically enhance the dope solution, because the hydrophilic properties of PEG attracts O–H functional groups to diffuse onto the non-solvent to increase membrane hydrophilicity [20].

Morphology

SEM has been widely used to investigate the surface morphology of cellulose acetate membranes, allowing for the visualization of their microstructure and topography [21]. The morphologies of the membrane reflect the thermodynamics and phase separation kinetics between the polymer, solvent, and non-solvent during dry-wet phase inversion. Figure 2 shows the SEM micrographs of the top surface of CA membranes at different concentrations of polymer, while the cross-section micrographs of CA membranes were presented in Figure 3.

Figure 2 shows the top surface of CA membranes depending on the polymer concentration. It is observed that with increase in CA solution concentration, the surface roughness of resulting membrane increases. Membrane with the highest polymer concentration (15 wt%) obtained more textured surface compared to 13 wt%. This phenomena is attributed to the increased viscosity and polymer entanglement in the casting solution that affected the development of the membranes surface roughness [22].

Figure 3 reveals that the average pore size of membrane is in the range of $0.5\text{--}1\ \mu\text{m}$. Based on the result showed, asymmetric membranes characterized by a dense top layer and a sponge-like substructure were obtained in this study. The CA membranes are composed of a thin top layer supported by a porous sublayer containing large void spaces or macrovoids. The top layer is dense, while the pores extend downward. This structural pattern is consistent across all membranes, where the dense top layer and non-homogeneous pores indicate the asymmetry of the membranes. CA membranes in this study were obtained using three different concentrations of polymer, ranging 13, 14, and 15 wt%. Figures 3a–c with 5000x magnification show that increasing polymer concentration decrease the interconnectivity between pores in membrane. The asymmetrical distribution of pores with varying sizes typically diminishes with an increase

in cellulose acetate concentration. It is evident, that with higher polymer concentration, the membrane with the maximum CA content (15 wt%) exhibits a thicker top surface and smaller pore diameter compared to the others.

It is notable that all membranes exhibit a macrovoids structure, the characteristics of which vary in accordance with the polymer concentration. The concentration of polymer during phase inversion affected the diameter of macrovoids formed in the membrane. Figure 3 shows that higher CA concentration leads to smaller size of macrovoids, as the macrovoids in membrane with CA 15 wt% have smaller size compared to 13 wt%. The kinetics and thermodynamics during phase inversion affected the formation of macrovoids which may exhibit different morphologies [23]. This macrovoids structure can be employed as support layers and is suitable for ultra-filtration processes for environmental application such as wastewater treatment. Visual inspection from SEM results indicates that CA membranes with lowest polymer concentration (13 wt%) was more fragile than 14% wt and 15% wt. Thus, higher polymeric solution is recommended for fabrication of CA membranes to obtain higher mechanical strength [24]. Moreover, membrane with a denser pore structure will exhibit higher mass transfer resistance, thereby affecting its applicability in specific applications.

Porosity

The porosity parameters of CA membranes have been measured by using dry-wet method. Figure 4 presents the porosity of the membrane at various polymer concentration.

Membrane porosity was significantly affected by the polymer concentration. As the polymer concentration increased, the resulting membrane has lower porosity, as illustrated in Figure 4. In the fabrication process, the miscibility of acetone in water facilitates diffusion during the immersion step. Consequently, increasing solvent concentration enhances porosity within the nonselective layer of the membrane [25]. The highest porosity value was achieved by the lowest CA concentration, which was 13 wt%. This value supports the SEM images (Fig. 3) which depicts the interconnectivity between pores in membrane decreased with the increasing of polymer con-

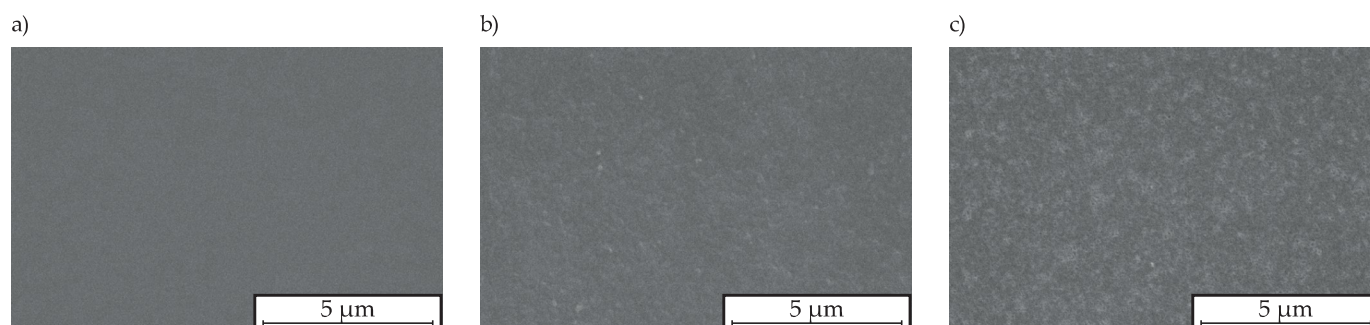
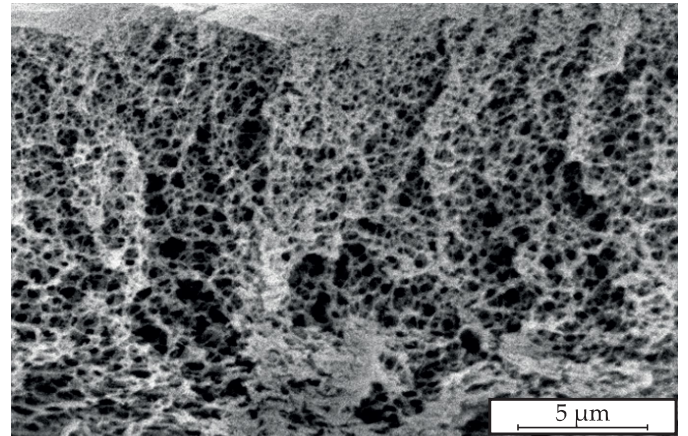
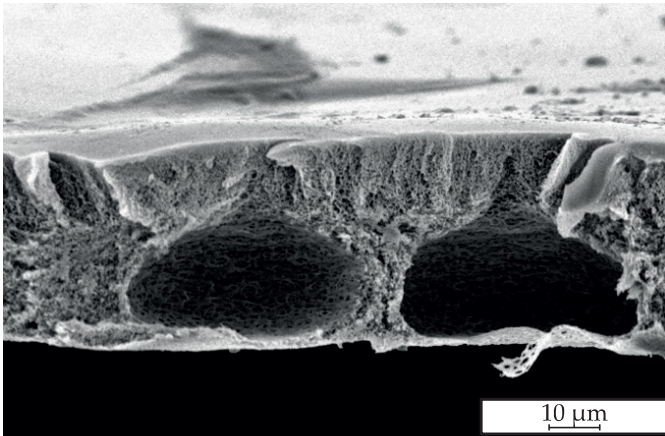
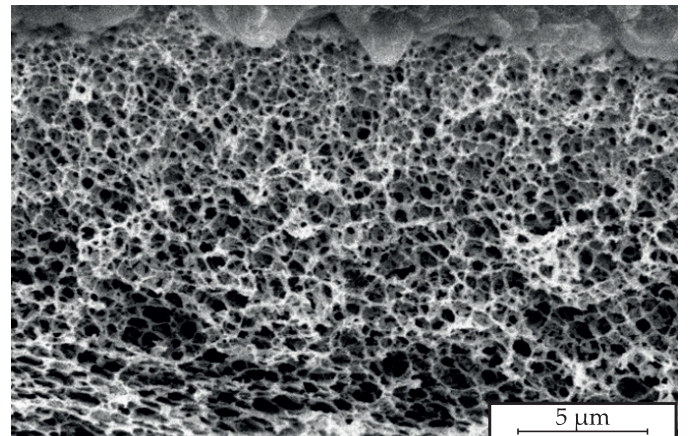
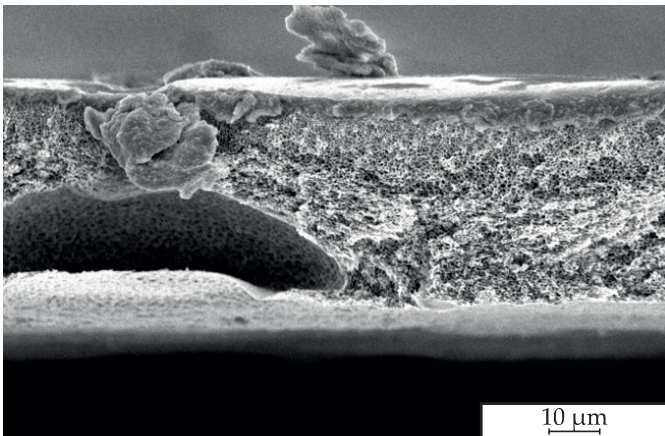


Fig. 2. SEM micrographs of CA membranes surface: a) 13 wt% CA, b) 14 wt% CA, c) 15 wt% CA

a)



b)



c)

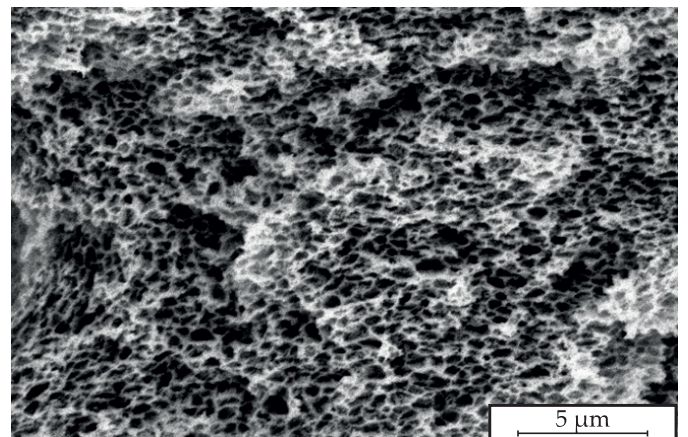
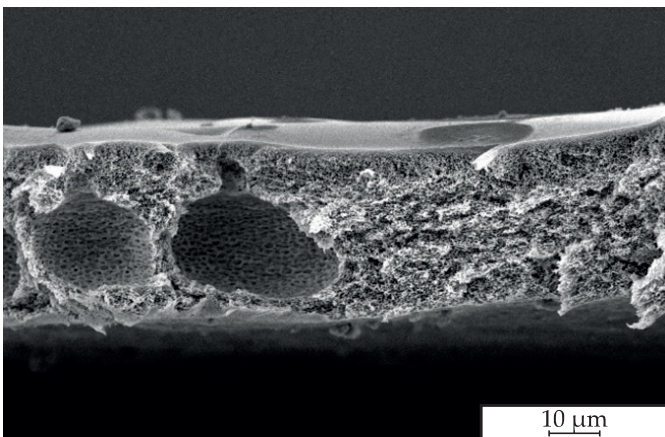


Fig. 3. SEM micrographs of CA membranes cross-section: a) 13 wt% CA, b) 14 wt% CA, c) 15 wt% CA; 1000× and 5000× magnification

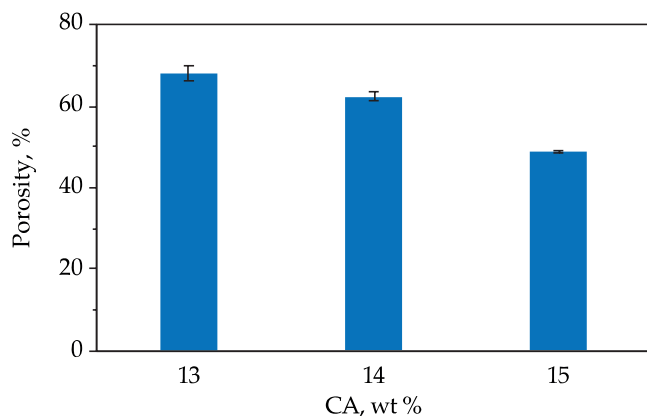


Fig. 4. Porosity of CA membranes

centration. Thus, the membrane porosity value obtained was lower.

Tensile properties

Tensile strength and elongation at break of CA membranes are presented in Fig. 5 and Fig. 6, respectively. Higher CA concentration results in an increase in tensile strength of membrane. This fact can be attributed to formation of macrovoids, as shown in the SEM images (Fig. 3). When the concentration of CA increased, the macrovoids numbers decreased and became smaller in diameter, making the membranes denser. It has improved the membrane structure, like it was described in literature [26–28].

It was also found that the tensile strength of CA-PEG membranes is higher than that of CA membrane without the addition of PEG (7 MPa) [29–31]. The tensile strength of CA-PEG membranes in this study was in the range of 7.6–10.8 MPa. Therefore, the addition of PEG increased the tensile strength of the CA membrane.

The elongation at break is presented in Figure 6. The increase of CA polymer concentration results in higher elongation at break. Elongation at break increases slightly from 11.8% to 12.4% for CA concentration of 13 wt% and 14 wt%. For CA concentration of 15 wt%, the elongation at break increases significantly (15.7%). Better membrane

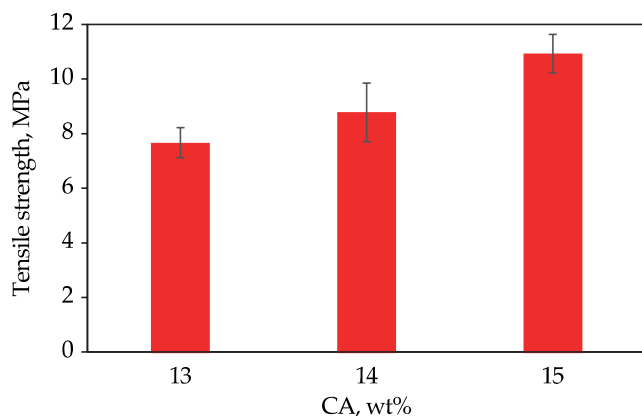


Fig. 5. Tensile strength of CA membranes

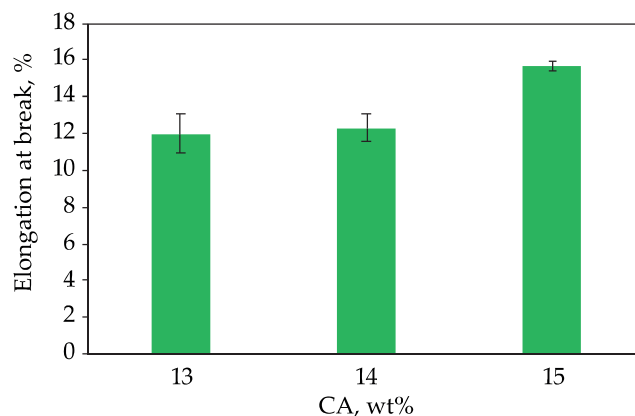


Fig. 6. Elongation at break of CA membranes

structure is attributed to smaller macrovoids and more micropore formed, as shown in the Figure 3. As reported previously [27], increasing polymer concentration from 20 to 25% in fabrication of polysulfone membrane leads to the increase of elongation at break.

CONCLUSIONS

Microporous cellulose acetate membranes were successfully developed. The effect of CA concentration ranging from 13 to 15 wt% was investigated. The addition of CA influenced the kinetic and thermodynamic properties during membrane manufacturing and thus influenced its morphology. Increasing CA concentration decreased the interconnectivity between the membrane pores and decreased the membrane porosity. Increasing the CA concentration improved the tensile properties. The membrane with the highest CA concentration (15% by weight) was characterized by the best tensile strength (10.8 MPa) and elongation at break (15.7%). This study provides useful information for evaluating the performance of CA membranes for environmental applications.

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Authors contribution

M.M. – conceptualization, methodology, supervision, data analysis, writing review; D.C.H. – conceptualization, methodology, supervision, data analysis, writing-review and editing, project administration; A.A. – experimental work, collecting data. All authors have agreed to the published version of paper.

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Authors declare that there are no financial interests of personal issues that influence the results reported in this paper.

Conflict of interest

There is no conflict of interest in the publication of this paper.

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Nanotechnologia, jako nauka zajmująca się tworzeniem struktur na poziomie atomów i cząsteczek, wpisuje się w trend miniaturyzacji, który niewątpliwie jest odpowiedzią na potrzeby dzisiejszego społeczeństwa. Możliwość wytwarzania nanocząsteczek oraz projektowania złożonych nanostruktur, tak aby wykazywały pożądane właściwości fizyczne, chemiczne, czy też biologiczne pokazuje, jak duży potencjał niesie ze sobą ta nauka.

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