

Synthesis of Cellulose Acetate from Screw Pine (*Pandanus tectorius*) Leaves for Enhancing Water Permeability and Fouling Resistance of PVDF Membrane

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Abstract: Cellulose derivatives from natural resources continuously develop to find the best hydrophobic polymer-based membrane technology performance. This study was conducted to improve the hydrophilicity, performance, and anti-fouling of polyvinylidene fluoride (PVDF) membrane with cellulose acetate (CA) filler synthesized from screw pine (*Pandanus tectorius*) leaf cellulose. CA was synthesized by the Fischer esterification mechanism and the PVDF membrane was fabricated using the phase inversion method with 0.3% CA concentration. FTIR analysis of CA shows an absorption at 1700 cm^{-1} suggesting that the hydroxyl group of cellulose had been successfully substituted with an ester group (C=O), and CA has a high degree of substitution (DS) value of 3.50. Adding CA improved the hydrophilicity and anti-fouling properties of up to 86.45% of PVDF membranes. Furthermore, CA increased the value of water permeability 2–3 times than pristine PVDF membrane. The presence of CA enhanced the porosity of the PVDF membrane, which promoted the membrane's effectiveness for MB filtering. As a result, CA from screw pine leaf cellulose has promising features as a filler for PVDF membranes and potential dye filtration.

Keywords: anti-fouling; cellulose; cellulose acetate; polyvinylidene fluoride; water permeability

■ INTRODUCTION

Polyvinylidene Fluoride (PVDF), commonly known as poly(1,1-difluoroethylene), has a repeating unit of $(\text{CH}_2-\text{CF}_2)$ [1]. PVDF is a polymer that is frequently utilized as a matrix membrane due to its excellent properties, such as thermal stability, membrane-forming ability, and high mechanical strength [2]. Nonetheless, the hydrophobic nature of PVDF membrane causes fouling, decreasing the membrane performance and shortening the membrane lifetime [3-4]. It is crucial to increase hydrophilicity to reduce PVDF membrane fouling.

Among the numerous materials utilized to promote PVDF membrane hydrophilicity, natural polymer has received attention due to its low cost and ease of availability [5]. Cellulose is one of the most common natural polymers in the world, accounting for 700,000 billion tons of total biomass production per year [6]. Natural cellulose-based fibers have several advantages over synthetic fibers, including low cost, low density, good strength, good

stiffness, environmentally friendly, biodegradable, renewable, and health-safe [7-8]. The cellulose skeleton's hydroxyl groups can be chemically modified to form ester groups that are insoluble in water [8-10].

Cellulose acetate (CA) is a cellulose ester derivative that has been widely applied in membranes. CA is a desirable polymer due to its low cost, biocompatibility, biodegradability, and acceptable physical qualities in industry [11-12]. CA is utilized in food packaging, coatings, sealants, and membrane separation [13]. Wood and cotton are the most important material resources for industrial synthesizing CA, although they have a high lignin content which requires more chemicals to remove cellulose [14]. Screw pine (*Pandanus tectorius*) is a non-wood plant of Pandanaceae family that grows naturally on river banks and swamps in the tropics that are rarely utilized. Isolation of cellulose from non-wood materials can reduce the usage of chemicals [8,15]. The functional groups of screw pine fibers are similar to those of flax,

kenaf, and linen natural fibers [16]. Screw pine leaves have a chemical composition of 37.3% cellulose, 34.4% hemicellulose, 15.7% pentose, 24% lignin, and 2.5% others [17]. As a result, Screw pine leaves can potentially be a source of CA synthesis.

Previous research used commercial CA [18-20] for matrix membranes and generated CA [14,21-23]. CA commercial has been used to improve water flux and fouling resistance in polyethersulfone (PES) [24], polysulfone (PSf) [25], and PVDF membranes [12,26-27]. So far, synthetic CA has not been used as a matrix membrane filler. No previous research has employed CA synthesized from screw pine as a PVDF membrane filler. This study used screw pine (*P. tectorius*) leaves as the starting material to synthesize cellulose acetate. It was used as a filler for PVDF membranes to improve water permeability and anti-fouling qualities. Water permeability, rejection, and anti-fouling from the flux recovery ratio (FRR) test were used to evaluate the performance of the manufactured PVDF/CA membrane.

■ EXPERIMENTAL SECTION

Materials

The materials used in this study were screw pine leaves collected from Yogyakarta coastal area, aquadest, NaOH, HCl, NaOCl, acetic acid anhydride, acetic acid glacial, H₂SO₄, DMAc, and PEG 400. Chemicals were purchased from e-Merck, Germany. PVDF (Solef PVDF 1010 Mw 352 kDa) was obtained from Solvay.

Instrumentation

The instrumentations used in this study were Fourier transform infrared (FTIR) Shimadzu FTIR Prestige 21 Model 8201 PC, scanning electron microscope (SEM) JEOL JSM-6510LA, thermogravimetric analyzer (TGA), and differential scanning calorimetry (DSC) STA Linseis PT 1600.

Procedure

Isolation of cellulose from screw pine leaves

Cellulose from screw pine leaves was isolated [17]. Screw pine leaves were collected from Yogyakarta coastal area. Screw pine leaves are stripped of their thorns and cut into 12 × 3 cm pieces. The leaf pieces were then sun-dried.

Dried screw pine leaves were cut into smaller pieces of around 1 × 1 cm and immersed in water for 3 × 24 h, with the water changed regularly. The screw pine leaves were then sun-dried again and grounded. Screw pine leave powder (100 g) was mixed in 10% NaOH for 2 h at 100 °C, with a screw pine leave to NaOH ratio of 1:20. The mixture was then neutralized to pH 7 and dried for 3 h at 60 °C. The dry powder was mixed with 4% NaOCl pH 4.5 at 100 °C for 2 h with a 1:20 alkalization product to NaOCl ratio. To obtain cellulose from screw pine leaves, the mixture was neutralized to pH 7 and dried at 60 °C for 3 h.

Synthesis of CA

CA was synthesized following [28] with modifications. Screw pine leaf cellulose (1 g) was added with glacial acetic acid (2.4 mL) and agitated for 60 min at 38 °C. The mixture was then stirred for 45 min at 38 °C with glacial acetic acid (4 mL) and sulfuric acid (0.5 mL). The mixture was then chilled to 18 °C. The acetylation procedure was then carried out. Acetic anhydride (10 mL) and sulfuric acid (0.5 mL) were added to the mixture and agitated for 90 min at 35 °C. The acetylation process was stopped by adding distilled water (1 mL) and glacial acetic acid (2 mL) and stirring at room temperature for 1 h. The mixture was neutralized with distilled water. The product was dried at 60 °C for 3 h to obtain cellulose acetate.

PVDF/CA membrane fabrication

Membrane PVDF/CA was fabricated by phase inversion [17]. First, DMAc (9.32 g) and PEG 400 (0.48 g) were stirred 5 min. The mixture was then added with cellulose acetate (0.036 g) and stirred for 10 min. Finally, PVDF (2.16 g) was added to the mixture and agitated at 60 °C for 24 h to prepare the dope solution for the PVDF/CA membrane. Otherwise, the PVDF dope solution was made using the same method, but without the CA filler. The dope solution was then cast in a glass plate and placed in a coagulation bath containing distilled water to produce the membranes.

Characterization of cellulose acetate

The FTIR spectral analysis was used to calculate the degree of substitution (DS) of cellulose acetate.

According to Eq. (1), the absorbance of C=O cellulose acetate was compared to the absorbance of C-O cellulose.

$$DS = \frac{Abs_{C=O}}{Abs_{C-O}} \quad (1)$$

The functional groups of the cellulose and cellulose esters were determined by FTIR spectrophotometer. The dried samples were combined in a 1:100 ratio with potassium bromide (KBr) powder, compressed into a thin transparent pellet, and the transmission was measured at 4000–400 cm^{-1} .

The surface morphologies of cellulose and cellulose acetate were analyzed by SEM. Coated samples with a thin layer of gold were mounted and imaged at 500–5000× magnifications with 10 kV accelerating voltage at a pressure of 70 Pa. The thermal stability of cellulose and cellulose acetate was studied by TGA. The samples were heated up to 600 °C at a heating rate of 10 °C/min under an air atmosphere.

PVDF and PVDF/CA membranes characterization and performance evaluation

The performance of PVDF and PVDF/CA membranes was evaluated [17]. PVDF and PVDF/CA membranes were evaluated by water contact angle, porosity (ϵ), pure water flux (PWF), water flux, rejection, and flux recovery ratio (FRR). Membrane porosity was determined using the gravimetric technique and Eq. (2),

$$\epsilon(\%) = \left(\frac{W_{wet} - W_{dried}}{A \times I_{wet} \times \rho} \right) \quad (2)$$

where W_{wet} is the weight of the membrane (g), I_{wet} is the thickness of the membrane (cm) in wet condition, W_{dried} is the weight of the dry membrane (g) after oven at 60 °C for 24 h, ρ is water density ($g\ cm^{-3}$), and A is the membrane area (m^2).

The membrane performance was evaluated using a dead-end system with continuous measurements beginning with PWF, water flux, rejection, and FRR. The membrane (diameter 5 cm) was placed in a stirred cell and then filled with distilled water to measure PWF (J_1) (Eq. (3)). To assess water flux and rejection (%R) (Eq. (4)), the distilled water was replaced with a 100-ppm methylene blue (MB) solution. Before performing the FRR test (Eq. (5)), the MB solution was partially replaced with distilled water and swirled without pressure to

eliminate any leftover MB on the surface membrane before being completely replaced with new distilled water. All measurements were taken using a two-bar system compaction for 15 min.

$$J_1 = \frac{v}{A \times t} \quad (3)$$

$$\%R = \left(1 - \frac{C_p}{C_r} \right) \times 100 \quad (4)$$

$$FRR = \frac{J_2}{J_1} \times 100\% \quad (5)$$

J_1 denotes PWF (L/m^2h), v permeates volume (L), A membrane surface area (m^2), t time (h), C_p permeate concentration, and C_r concentration J_2 is water flux (L/m^2h), and retentate was determined using a UV-vis spectrophotometer at 664 nm. The thermal stability of PVDF and PVDF/CA membranes was evaluated by TGA. The membranes were heated up to 900 °C at a heating rate of 10 °C/min under an air atmosphere.

RESULTS AND DISCUSSION

Isolation of Cellulose and Synthesis of CA from Screw Pine Leaves

Initially, cellulose was isolated by soaking screw pine leaf powder in water to remove pentose sugar, tannins, and pigments. White powders of cellulose were obtained at 40.24% yield. The fiber does not appear in SEM images of screw pine leaf powder (Fig. 1(a) and 1(b)) because it was bonded by lignin and hemicellulose [7]. An alkalization method was used to remove lignin and hemicellulose. The bleaching process was performed to degrade the remaining lignin to increase cellulose purity [29-30]. SEM images of cellulose and the magnification (Fig. 1(c) and 1(d)) revealed the individual fiber after alkalization and bleaching. Cellulose was successfully isolated from screw pine leaves, as confirmed by SEM. The isolated cellulose was converted into CA. Cellulose morphology changed after the acetylation process. SEM images of CA and the magnification (Fig. 1(e) and 1(f)) showed that CA formed aggregates with produce hole, where no fibers were found in synthesized CA. Morphological changes from cellulose to CA occur due to acetylation substitution towards the -OH group, in which initially

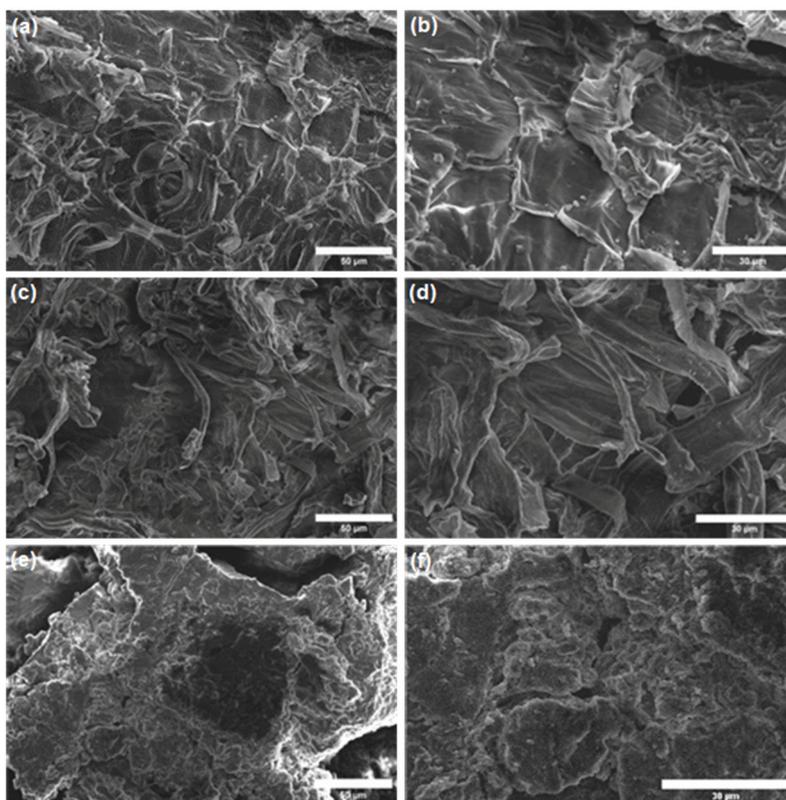


Fig 1. SEM image and the magnification of (a, b) screw pine leaf powder, (c, d) cellulose, and (e, f) CA

intramolecular hydrogen bonds of the $-OH$ functional group were formed [31].

Analysis of the functional group of screw pine leaf powder, cellulose, and cellulose acetate was investigated using FTIR (Fig. 2). The absence of peaks at 1504 and 1235 cm^{-1} for $C=C$ stretching lignin in cellulose spectra [8] indicated that lignin was eliminated, and cellulose was successfully isolated. In the FTIR spectra of CA, bands appeared at 1754 , 1374 , and 1235 cm^{-1} , corresponding to functional groups of $C=O$ stretching for carbonyl ester, $C-H$ in $O-(C=O)-CH_3$, and $C-O$ stretching for acetyl. The decrease in absorption intensity for $-OH$ stretching at 3384 cm^{-1} shows that the $-OH$ group of cellulose was substituted with acetyl groups from acetic anhydride [28,32-33]. The success of CA synthesis was validated by FTIR data, which matched the CA obtained in prior studies [28,32,34]. Furthermore, the DS value supports the successful CA synthesis. The DS of CA was 3.50, confirming the formation of cellulose triacetate.

The Fischer mechanism reaction was used to synthesize CA (Fig. 3). The initial esterification reaction

was carried out by substituting cellulose hydroxyl groups with acetyl groups. CA synthesis begins by enhancing the reactivity of cellulose's acetyl group with glacial acetic acid. The acetylation process requires a large cellulose surface, and the rate of the acetylation reaction is determined by the accessibility of the surface

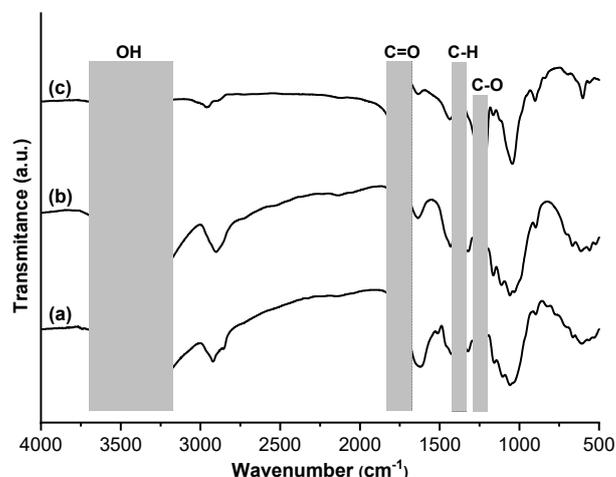


Fig 2. FT-IR spectra of (a) screw pine leave, (b) cellulose, and (c) CA

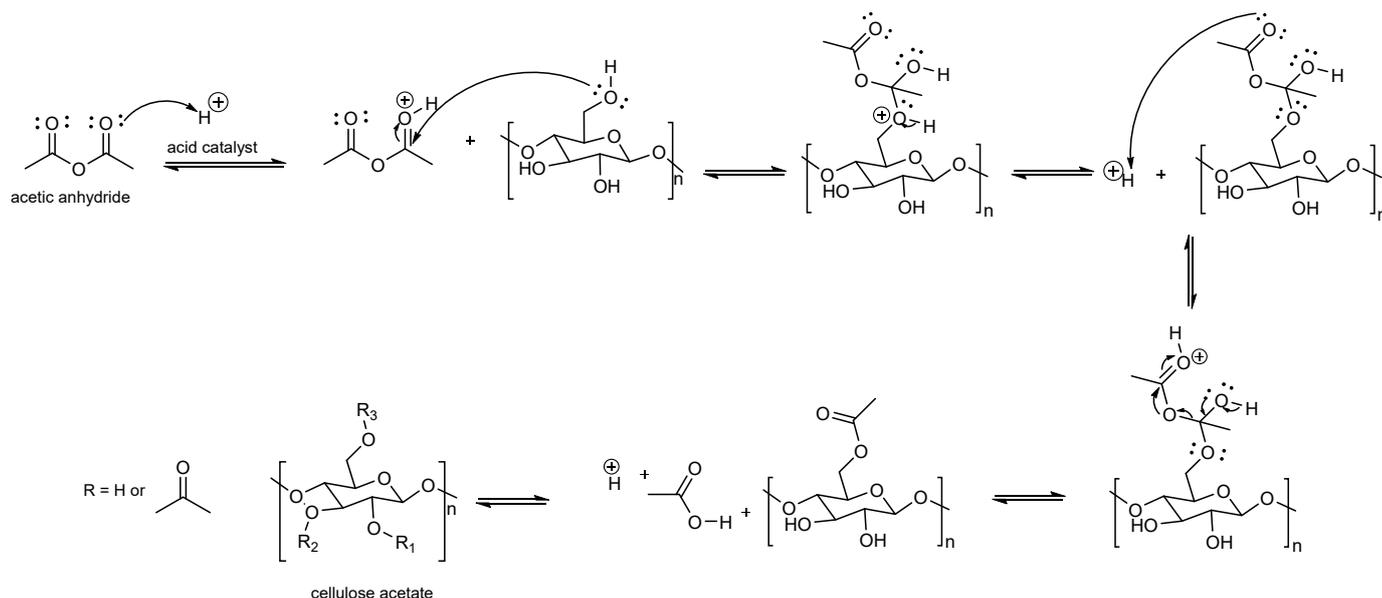


Fig 3. Fischer esterification mechanism reaction

opening ability of the cellulose fiber. The greater the surface area of the cellulose fiber, the easier the subsequent acetylation reactions [35]. Acetylation was accomplished by using acetic anhydride, and sulfuric acid was used as a catalyst. When the -OH group of cellulose is exchanged with the acetyl group of acetic anhydride, the desired CA and acetic acid are produced as a result. Because acetylation is an exothermic reaction, the reaction was carried out at a low temperature to maintain the cellulose is being degraded. The acetylation reaction is terminated through a hydrolysis process with the addition of acetic acid. This method eliminates unreacted acetic anhydride and releases the acetyl group [34]. CA was obtained as a white powder in 89% yield.

The cellulose and CA thermograms revealed three stages of degradation (Fig. 4). The first degradation occurred below 100 °C, exhibiting water evaporation. Because of the acetylation process of cellulose, mass reduction of cellulose was greater than CA in the first degradation (Table 1). Water absorption of cellulose acetate is reduced when hydroxyl groups are replaced with acetate groups. In the second stage, the cellulose and CA polymer chains degraded, with CA having more excellent thermal stability than cellulose [14]. CA polymer chain degradation is identical to CA produced by Chuayplod and Aht-Ong [36], which degraded at 298–375 °C. The

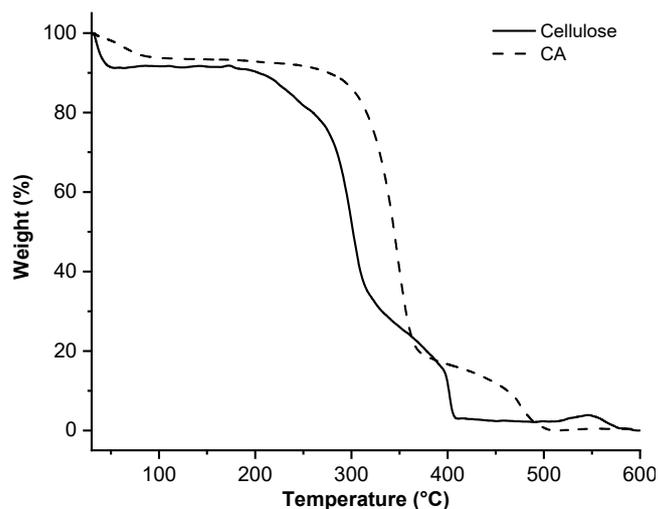


Fig 4. Thermogram of (a) cellulose and (b) CA

Table 1. Thermal stability of cellulose and CA

Samples	Temperature (°C)	Mass reduction (%)
Cellulose	31–54	8.76
	205–407	86.52
	407–600	3.28
CA	31–90	5.99
	270–370	70.59
	390–600	17.16

third stage is cellulose and CA backbone degradation [14]. This finding demonstrated that the acetylation process improved cellulose's thermal stability.

Characterization and Performance Evaluation of PVDF and PVDF/CA Membranes

PVDF and PVDF/CA membranes were characterized by TGA and DTG analysis. Thermograms of PVDF and PVDF/CA membranes (Fig. 5(a)) showed similar thermal stability. Degradation at temperatures under 100 °C appeared in the PVDF membrane due to water evaporation [4,17]. Whereas evaporation of water in the PVDF/CA membrane did not appear. Degradation of PVDF polymer matrix in PVDF and PVDF/CA membrane occurred at a relatively similar temperature at 420 and 490 °C. Therefore, the addition of CA in the PVDF matrix membrane did not drastically affect the thermal stability of the PVDF membrane. DSC thermogram of PVDF and PVDF/CA membrane (Fig. 5(b)) showed the endotherm peak. The addition of CA caused a shift in melting temperature (T_m) from 169 to 171 °C. It showed that the PVDF membrane has two crystalline phases, α and β phases [37]. The addition of CA

did not decrease the characteristic melting point of the PVDF membrane.

Water contact angle, porosity, PWF, water flux, rejection, and FRR were used to evaluate the performance of PVDF and PVDF/CA membranes, as shown in Table 2. The addition of cellulose acetate decreased the water contact angle of the PVDF membrane. The acetylation process decreases the number of cellulose hydroxyl groups [38]. Whereas cellulose hydroxyl groups unsubstituted with acetate groups can ensure the hydrophilic properties of CA. The porosity of the PVDF membrane increases up to 85.47% with CA addition. The difference in hydrophilicity between PVDF and CA causes a repulsive force that increases the porosity of the PVDF membrane [4].

PWF and water flux increased twice in the PVDF/CA membrane. Increasing the porosity of PVDF/CA improves membrane water permeability because water can easily travel through the membrane.

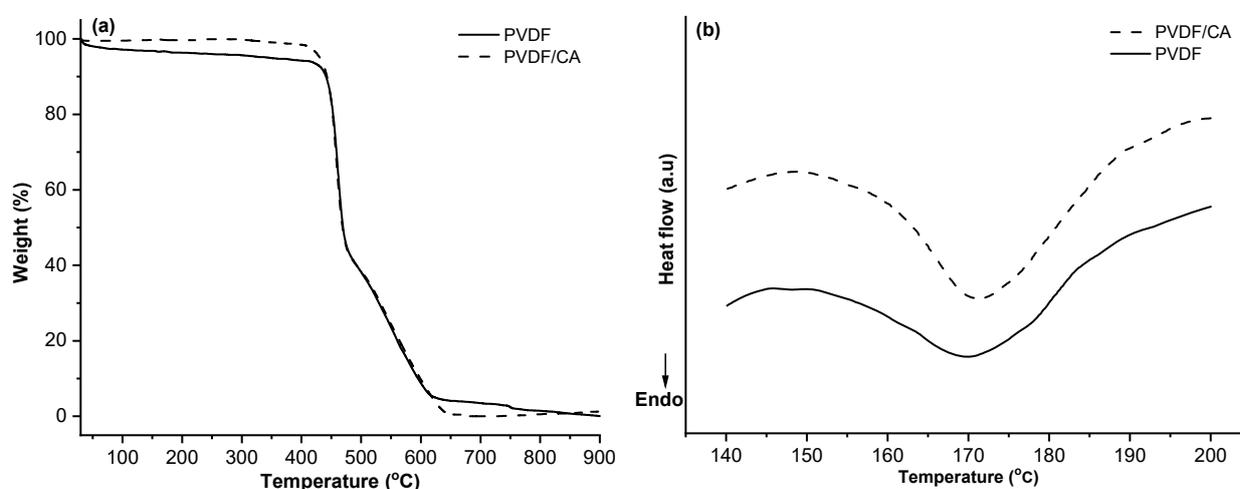


Fig 5. (a) TGA and (b) DSC of PVDF and PVDF/cellulose membranes

Table 2. Performance evaluation of PVDF and PVDF/CA membranes and the comparison with PVDF/CA membrane other studies

Membrane	Feed molecule	Water contact angle (°)	Porosity (%)	PWF (L/m ² h)	Water flux (L/m ² h)	Rejection (%)	FRR (%)	Ref.
PVDF	MB	89.70 ± 2.12	70.70 ± 0.48	17.08 ± 2.02	11.22 ± 0.18	82.89 ± 2.11	80.26 ± 0.68	This work
PVDF/CA	MB	88.13 ± 1.01	85.47 ± 0.88	45.42 ± 3.07	42.15 ± 3.08	77.73 ± 3.57	86.45 ± 3.62	This work
PVDF/CA	PEG	~93.00	-	136.20	-	3.00	94.20	[12]
PVDF/CA	PEG	~61.00	~82.00	522.00	~300.00	~68.00	~76.00	[26]
PVDF/CA	MB	56.43	~84.00	140.00	83.17	90.24	95.77	[27]

The higher the porosity of the PVDF/CA membrane, the lower MB rejection because the MB molecule can pass through the membrane. Lower membrane rejection is achieved by increasing water permeability [17,39]. PVDF membrane anti-fouling properties improved with the addition of CA, which has an excellent anti-fouling performance and minimizes interaction between MB and surface membrane [40]. The washing treatment performed before the FRR test improved the anti-fouling value of the PVDF/CA membrane. PVDF/CA membrane performed well and was comparable to earlier studies that employed commercial CA as a filler for PVDF membrane. Table 2 shows a comparison of membrane performance with previous studies. Our PVDF/CA membrane performances are comparable to other commercial PVDF membranes with the addition of CA. In this work, including CA synthesized from screw pine cellulose resulted in higher porosity, water permeability, and FRR values than the PVDF membrane. As a result, the PVDF/CA membrane developed in this work has the potential to be used in dye wastewater filtration.

■ CONCLUSION

Cellulose was successfully isolated from screw pine (*Pandanus tectorius*) leaves by alkalization and bleaching treatment which was confirmed by FTIR, SEM, and TGA analysis. Cellulose acetate (CA) was produced with high yields of 89% utilizing Fischer esterification processes and acetic anhydride reagents. The cellulose acetates were more thermally stable than cellulose and had a high DS of 3.50. The presence of CA did not affect the thermal stability of the PVDF membrane. The addition of CA improves PVDF membrane performance by increasing water permeability by twice and anti-fouling qualities up to 86.45%. PVDF/CA membrane also has good filtration ability against methylene blue (MB) up to 77.73%. Thus, these membranes have the potential for dye wastewater treatment. This synthesized CA has good properties as a filler to PVDF membrane for filtration MB.

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■ AUTHOR CONTRIBUTIONS

Elyna Wahyu Trisnawati conducted research, analyzed data and wrote an original draft; Indri Sri Cahyani and Diah Safriyani performed the experiments and data collection; Edi Pramono developed methodology and verification data; Venty Suryanti conducted formulation and verification data as well as wrote a manuscript (review and editing). All authors agreed to the final version of this manuscript.

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