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Research Article

Synthesis and Characterization of Cellulose Acetate Derived from Microcrystalline Cellulose (MCC) for Membrane Applications

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Abstract. The clean water crisis in Indonesia is worsening due to limited distribution, pollution of water sources, and rapid population growth and industrial activity. Membrane technology is an effective solution, but Indonesia still relies on imported synthetic polymer-based membranes that are less sustainable and prone to fouling. Microcrystalline cellulose (MCC) from lignocellulosic biomass, such as oil palm empty fruit bunches (OPEFB), offers a more environmentally friendly alternative due to its hydrophilic properties, biodegradability, and abundant availability. However, the limited solubility of MCC in organic solvents requires a derivatization process to cellulose acetate (CA) before use as a membrane material. This study aims to produce CA from MCC through an acetylation process and evaluate the degree of acetylation (DA), degree of substitution (DS), and changes in the chemical structure of the modified product. Acetylation was carried out using glacial acetic acid, acetic anhydride, and sulfuric acid catalysts with varying reaction times of 2–4 hours. The titration results showed DA values of 38.70–39.41% and DS of 2.43–2.50, with an optimum reaction time of 3 hours. These values are within the ideal range for membrane applications, indicating good process efficiency. FTIR analysis showed the disappearance of lignocellulose groups and the appearance of a carbonyl (C=O) peak at ~1740 cm^{-1} as well as an increase in the intensity of the acetate C–O group, confirming the success of acetylation. These results indicate that MCC from OPEFB has great potential as a sustainable and easily processed CA membrane base material for water purification applications.

Keywords: Microcrystalline Cellulose, Cellulose Acetate, Acetylation, Advance Materials

1. Introduction

Indonesia, as an archipelagic nation surrounded by oceans, continues to face a clean water crisis. As of March 2025, approximately 28 million Indonesians still lack access to clean water (Ihsan & Ilfan, 2025; Ridwan et al., 2024). This is further compounded by the fact that another 21 million still lack access to clean, potable water. This crisis is not only a matter of distribution between urban and rural or remote areas, but also a matter of quality, with many people in these areas relying on shallow water sources, open springs, or rivers that are vulnerable to pollution, posing health risks (Sebayang et al., 2025; Ridwan et al., 2024) . The availability of clean water will continue to be a global challenge, exacerbated by population growth, urbanization, and increased industrial and agricultural activity (Ekowati & Lusno, 2025). Natural disasters such as floods, landslides, droughts, and seawater intrusion can also contribute to a clean water crisis (Ekowati & Lusno, 2025; Susilo et al., 2025).

Membrane technology has become an effective and efficient separation technology; the use of this technology continues to grow because it has several advantages compared to conventional technologies such as distillation and extraction (Grzybek et al., 2024; Islam et al., 2023; Wang et al., 2024). Membranes are defined as thin, selective, and semipermeable layers located between two phases, namely the feed phase and the permeate phase. This selective nature allows membranes to be used in the separation process of substances. Another advantage is the simple and environmentally friendly operation process. Applications of membrane technology in industry include clean water treatment,

waste treatment, energy, separation of organic materials, as well as health and medical applications (Grzybek et al., 2024; Wang et al., 2024). However, Indonesia also faces the next problem regarding the availability of membranes themselves. Until now, Indonesia has not been able to produce membranes independently and still relies entirely on imports. Then the majority of commercial membranes are still based on synthetic polymers such as polyethylene (PE), aromatic polyamide (aramid), polyvinylidene chloride (PVDF), polysulfone (PS), polyethersulfone (PES), polyvinyl alcohol (PVA), polytetrafluoroethylene (PTFS), and polycarbonate (PC), which are sourced from petroleum. This has the potential to raise sustainability issues as well as a tendency towards fouling problems due to its hydrophobic nature (Abdullah et al., 2024; Qi et al., 2025).

In response to this issue, recent research has focused on the use of natural polymers as membrane raw materials (Microcrystalline cellulose, MCC) with additives (PEG). Indonesia's natural cellulose resources are abundant and can be derived from waste such as empty oil palm fruit bunches (EFB), rice straw, bagasse, wood waste, and other agricultural waste with cellulose content ranging from 37-65% (Abdullah et al., 2024; Suyambulingam et al., 2025). Besides its availability, cellulose also offers advantages such as biodegradability, crystallinity, and hydrophilic properties that can reduce membrane fouling problems (Islam et al., 2023).

Microcrystalline cellulose (MCC) is a converted form of cellulose, which undergoes partial hydrolysis and depolymerization, which removes its irregular amorphous parts to produce high-purity cellulose up to 95.19%, highly crystalline cellulose between 55%-80% with a reduced degree of polymerization (around 400), a highly porous structure, and has multifunctional polar groups, especially abundant hydroxyl (OH) groups (Alsohaimi et al., 2023; Varma et al., 2025; Zahran et al., 2020). Therefore, MCC is very attractive to be adapted to membrane applications both as a polymer matrix material (such as Cellulose Acetate/CA, or Polyethersulfone/PES) and as a composite filler to produce Mixed-Matrix Membranes (MMMs) (Qi et al., 2025; Varma et al., 2025). In addition, the use of MCC in membrane fabrication faces a major challenge, namely, the solubility of cellulose in conventional organic solvents is very limited, so that commonly used fabrication techniques (phase inversion) that use solvents such as DMF or acetone are difficult to apply directly to MCC without derivatization. Therefore, acetylation of MCC to cellulose acetate (CA) is a widely used solution to obtain cellulose-derived polymers that are soluble in standard membrane solvents and easily processed into membranes, including hollow-fiber configurations that are superior for industrial-scale applications due to their high specific surface area and operational modularity (Sango et al., 2024). The Acetylation process involves the degree of substitution (DS) of the hydroxyl (-OH) groups on the anhydro glucose units with acetate (-OCOCH3) groups. The DS (0–3) determines the average number of substitutions and has a strong influence on the solubility, hydrophilicity, and mechanical properties of CA. A DS around 2.0–2.5 is generally suitable for membrane applications because it provides good solubility in organic solvents and a balance of mechanical/hydrophilic properties (Sango et al., 2024). So that the cellulose acetate (CA) membrane will have a porous and dense structure that can be used as a membrane-making material in the Reverse Osmosis (RO) process, desalination membrane, ultrafiltration (UF) process, and nanofiltration (NF).

This is supported by previous studies, such as the removal of certain dyes using CA/MCC membranes, which are able to increase the rejection of Methylene Blue (MB) up to 99%. PES/MCC membranes show significantly greater rejection of bromate ions (BrO_3^-) up to 92% at a concentration of 5% MCC, which is associated with increased hydrophilicity and optimal pore size (Abdullah et al., 2024). The addition of MCC to CA membranes (isolated from various lignocellulosic biomass such as water hyacinth, sengon wood, and kapok fiber) has been shown to increase the tensile strength of the membrane (up to 3.05 $N \cdot mm^{-2}$ at the addition of 1% MCC) (Suyambulingam et al., 2025). MCC also significantly increases the hydrophilicity of the membrane (Ciriminna et al., 2024). For example, CA membranes containing MCC show a decrease in the water contact angle from 65∘ (pure CA membrane) to as low as 52∘ (Ciriminna et al., 2024). This increase in hydrophilicity is due to the large number of –OH groups in the MCC structure that form hydrogen bonds with water molecules. In addition, the addition of MCC increases the membrane porosity from 82% (pure CA) to up to 88%. The composite membrane can significantly increase water flux, from 43 $L \cdot m^{-2} \cdot h^{-1}$ (pure CA) to between 82 and 88 $L \cdot m^{-2} \cdot h^{-1}$. This increase occurs because the hydrophilic nature of MCC accelerates the exchange of solvents and non-solvents during the phase inversion process, resulting in the formation of more pores. The incorporation of 1% MCC into PVDF membranes was reported to result in a significant increase in water flux, from 16.76 $L \cdot m^{-2} \cdot h^{-1}$ to approximately 27 $L \cdot m^{-2} \cdot h^{-1}$, and achieves humic acid rejection of up to 99%. MCC is also used to strengthen nanofiber membranes made by electrospinning. In PVP/lecithin nanofibers, the addition of MCC increased mechanical properties (tensile strength) by up to 279%. MCC was added as a filler to increase mechanical strength, without causing significant changes in fiber diameter (due to the parallel shape of MCC rods in the electrospun fibers) (Rafieian et al., 2020).Although the study focused on nanofiltration and protein adhesion (such as blood plasma products), this increase in strength and stability can be used as a basis for pressurized water treatment applications. Furthermore, MCC was also successfully isolated from various inexpensive lignocellulosic biomass sources, including oil palm empty fruit bunches, water hyacinth, sengon wood, and kapok fiber. showed good thermal stability (cellulose degradation occurs at temperatures of 300°C to 350°C) (Suyambulingam et al., 2025; Varma et al., 2025). For example, MCC from water hyacinth has

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the highest decomposition temperature of up to 317°C. The morphological characteristics of MCC show a highly crystalline structure (Crystallinity Index/CrI often between 55% to 80%), abundant pores, and OH groups that facilitate interaction with water (Zhu et al., 2025).

Although previous studies have reported the synthesis of cellulose acetate (CA) from microcrystalline cellulose (MCC) derived from oil palm empty fruit bunches (OPEFB) and highlighted its use in membrane-related applications, most of them primarily focus on acetylation feasibility or composite membrane performance, with limited attention to process efficiency under mild reaction conditions and application-oriented material design. In addition, the reported degree of substitution (DS) and degree of acetylation (DA) are often presented as isolated values without sufficient discussion of their implications for membrane-relevant properties. In this study, pure CA is deliberately synthesized as a fundamental precursor material, recognizing its role as the primary polymer matrix governing solubility, hydrophilicity, and processability in membrane fabrication. The main contribution and objective of this work lie in the optimization of an efficient and mild acetylation process (3 h, 25 °C) to produce membrane-grade cellulose acetate with a high degree of substitution (≈ 2.50) and degree of acetylation (≈ 39.41%), while explicitly establishing structure–property relationships relevant to membrane material design**.** This objective is supported by FTIR evidence of hydroxyl group reduction and characteristic acetate carbonyl formation. While the use of OPEFB-derived cellulose inherently supports sustainability considerations, the core scientific focus of this study is placed on process efficiency and material quality, providing a robust basis for rational membrane material development and subsequent MCC–CA composite membrane fabrication (Islam et al., 2023; Abdullah et al., 2024; Grzybek et al., 2024; Sango et al., 2024; Qi et al., 2025).

2. Method

2.1. Materials and Methods

The main material used in this study is Microcrystalline Cellulose (MCC), which has been ground as much as 6 grams using a blender. The microcrystalline cellulose (MCC) used in this study was derived from oil palm empty fruit bunches (OPEFB) through prior delignification and partial hydrolysis, resulting in a cellulose-rich material with high purity and reduced non-cellulosic components. MCC is typically characterized by micron-scale particle size and a relatively high crystallinity index (CrI), generally ranging from 55% to 80%, due to the removal of amorphous regions during hydrolysis. These physicochemical characteristics play a crucial role in the acetylation process, as smaller particle size enhances surface area and reagent accessibility, while high crystallinity promotes more controlled and homogeneous substitution of hydroxyl groups. Acetylation is known to initiate preferentially in the amorphous domains before progressing to the crystalline regions; therefore, the combination of high purity, suitable particle size, and elevated crystallinity facilitates efficient acetylation under mild reaction conditions. Similar effects of MCC properties on acetylation efficiency and degree of substitution have been widely reported in cellulose-based membrane and composite studies (Zahran et al., 2020; Alsohaimi et al., 2023; Grzybek et al., 2024; Sango et al., 2024; Suyambulingam et al., 2025; Varma et al., 2025). Next, the raw material is put into a three-neck flask assembled with a magnetic hot plate stirrer (stirred tank reactor). Then, 150 ml of glacial acetic acid is added to the three-neck flask, and the stirrer is turned on to mix the solution until homogeneous, with a stirring process that lasts for 3 hours. After that, 45 ml of anhydrous acetic acid and 9 drops of concentrated sulfuric acid catalyst are added to the mixture, with a temperature of 25°C and different reaction time variations (2 hours, 3 hours, and 4 hours). After the reaction was completed, 6 ml of distilled water and 15 ml of glacial acetic acid were added to the solution, and the reaction was allowed to proceed for 30 minutes. Then, 3 grams of sodium acetate were added to the solution, and the process was allowed to proceed for 5 minutes. After the reaction was completed, the mixture was washed with tap water to remove the odor of acetic acid, and then soaked in methanol for 10 minutes. The solution was then filtered, dried in an oven at 50°C, ground, and sieved. All chemicals used for the acetylation process were purchased from Sigma-Aldrich. All experiments were performed three times separately, and the average values are presented to avoid the influence of individual differences and to prove the reliability of the results.

The reaction temperature was controlled using a thermostated reaction setup and continuously monitored with a calibrated digital thermometer to maintain a constant temperature of 25±1 °C throughout the acetylation process. Minor temperature fluctuations within this range are not expected to significantly affect the degree of substitution (DS) and degree of acetylation (DA), as the acetylation reaction is primarily governed by reagent availability and reaction time under mild conditions. Nevertheless, larger temperature deviations may influence acetylation kinetics and diffusion of acetylating agents, potentially leading to variations in DS and DA, as reported in previous cellulose acetylation studies (Islam et al., 2023; Grzybek et al., 2024).

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2.2. Determining the Degree of Acetylation and Substitution

To determine the degree of acetylation and substitution using the titration process, first weigh 0.3 grams of cellulose acetate and dissolve it in 25 mL of acetone. After that, add 2-3 drops of PP indicator and titrate with 0.1 N NaOH to a faint pink endpoint. Then, calculate the degree of acetylation and the degree of substitution.

2.3. Fourier Transform Infrared Spectroscopy Analysis

The functional groups and chemical composition of CA were confirmed by Fourier transform infrared spectroscopy (FTIR) analysis. FTIR spectroscopy was employed as the primary structural characterization technique because the main objective was to confirm chemical modification through acetyl group substitution rather than morphological changes. FTIR, together with titration-based determination of the degree of acetylation (DA) and degree of substitution (DS), is widely regarded as a reliable and sufficient approach to validate the successful conversion of cellulose to cellulose acetate. Complementary techniques such as scanning electron microscopy (SEM) are mainly used to examine surface morphology and pore structure, which are more relevant at the membrane fabrication stage. Since acetylation primarily alters the chemical functionality of cellulose without inducing distinct morphological features at the particle scale, SEM analysis was considered beyond the scope of the present study. Similar characterization strategies have been commonly adopted in cellulose acetate synthesis studies focusing on chemical structure and substitution efficiency (Islam et al., 2023; Grzybek et al., 2024; Sango et al., 2024).

This analysis is based on differences in absorption bands (peaks) at specific wavenumbers, indicating the presence of characteristic functional groups in each compound. Sample preparation involved creating a thin pellet by mixing CA with potassium bromide (KBr) at a 1:100 ratio. The samples were recorded in the wavenumber region of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans per sample to optimize the signal-to-noise ratio.

3. Result and Discussion

3.1. Degree of Acetylation and Degree of Substitution

The results of the study indicate that the acetylation process of cellulose from OPEFB produces cellulose acetate with characteristics suitable for membrane applications. Titration results indicate that the volume of NaOH used in the titration process ranged from 27 mL to 28 mL, with an average degree of acetylation of 39.41% and a degree of substitution of 2.50. These results are within the expected optimal range of 36.5% - 42.2% for membrane applications. A comparison of the research results and reference or literature values is presented in Table 1. Based on the comparative data, the acetylated MCC showed a trend towards increasing degrees of acetylation (DA) and degrees of substitution (DS) with increasing reaction time. This increase indicates that the acetylation process is more optimal at longer reaction times, as the number of hydroxyl groups (-OH) in the cellulose that react with acetic anhydride increases, resulting in an increased number of bound acetyl groups (Grzybek et al., 2024; Qi et al., 2025).

Table 1. Effect of reaction time on the degree of acetylation (DA) and degree of substitution (DS) of cellulose acetate

Based on results				Based on Literature			
Reaction time (h)	NaOH Volume (mL)	Degree of Acetylation (%)	Degree of Substitution	Reaction time (h)	NaOH Volume (mL)	Degree of Acetylation (%)	Degree of Substitution
2	27	38.70	2.43	1.5	24	36.8	2.40
3	27.5	39.41	2.50	2	25	38.5	2.55
4	27.5	39.41	2.50	3	26	40.1	2.66

The optimum condition was achieved at a reaction time of 3 hours, with a DA value of 39.41% and a DS of 2.50. This value indicates good efficiency, considering that this achievement requires a relatively short reaction time compared to the highest value in the literature, where a DS of 2.66 was only obtained at a reaction time of 3 hours with different conditions. In addition, the results of this study show that at reaction times of 3 hours and 4 hours, the DA and DS values are relatively stable without significant increases, so that a reaction time of 3 hours can be considered the most efficient condition.

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The obtained DS values also meet the minimum criteria (>2.5) for the application of cellulose acetate as a filtration membrane material or biodegradable film. The similarity of the research results with the literature also indicates that the method used has a good level of validity. Small differences in DA and DS values between this study and the literature may be caused by variations in process parameters, such as the concentration of the NaOH solution, the purity of the cellulose raw material, and temperature control during the acetylation reaction (Abdullah et al., 2024; Qi et al., 2025; Wang et al., 2024; Wang et al., 2025).

The literature benchmarks employed in this study were selected based on the comparability of the resulting degree of acetylation (DA) and degree of substitution (DS), rather than identical reaction conditions, as these parameters are intrinsic indicators of cellulose acetate quality and membrane applicability. Differences in catalysts, solvent systems, or reaction temperatures among studies do not diminish the relevance of DA and DS for evaluating process efficiency and material performance. Furthermore, the DS range obtained (2.4–2.5) is expected to directly influence membrane morphology during phase inversion by balancing polymer solubility and hydrophilicity, thereby promoting controlled solvent–nonsolvent exchange and the formation of asymmetric porous structures. Such structure–property relationships between DS, hydrophilicity, and pore formation are well established in cellulose acetate membrane literature (Islam et al., 2023; Wang et al., 2024; Grzybek et al., 2024; Sango et al., 2024; Qi et al., 2025).

3.2. FTIR examination

The obtained FTIR spectrum provides information on the chemical structure changes that occur during the cellulose isolation and acetylation process. Figure 1 shows that the FTIR spectrum of OPEFB (black color) has a strong absorption band around ~3330 cm^{-1}, which indicates the O–H stretching of the hydroxyl group, indicating the presence of compounds containing lignocellulose. The band at ~2918 cm^{-1} is the aliphatic C–H stretching of the methyl and methylene groups. Meanwhile, the absorption at ~1730 cm^{-1} indicates the presence of carbonyl groups (C=O) from hemicellulose or ester compounds. The peaks at ~1240 cm^{-1} and ~1030 cm^{-1} indicate the presence of C–O–C bonds from the ether and secondary alcohol structures, which are common in natural lignin and cellulose components. After the cellulose isolation process, the FTIR spectrum of cellulose (red color) shows significant changes.

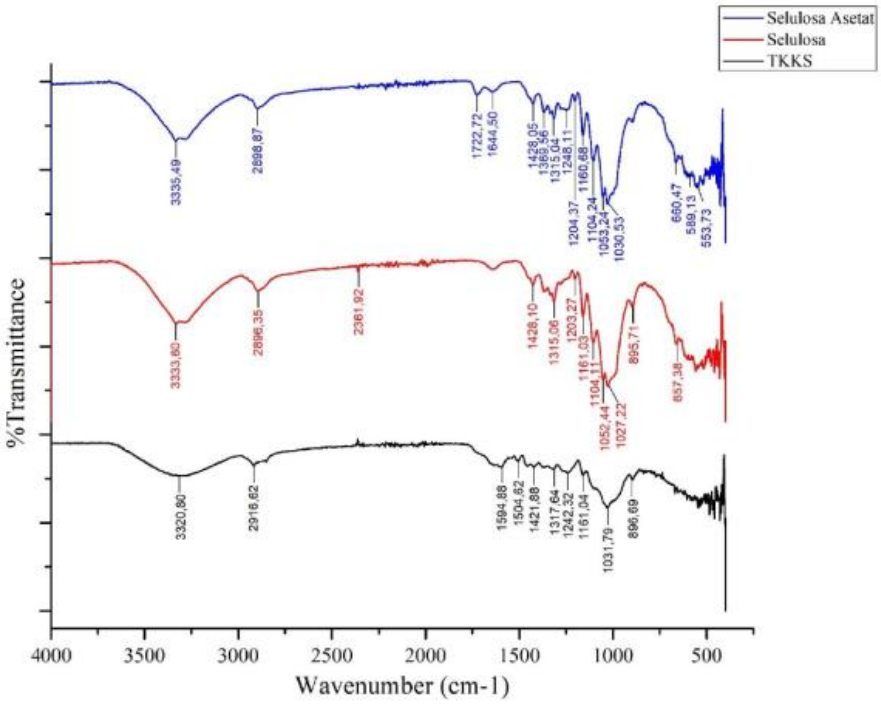

Figure 1. FTIR test results on cellulose acetate, cellulose, and empty fruit bunches of oil palm

The peak at ~1730 cm^{-1}, carbonyl groups disappeared, indicating that hemicellulose and lignin had been largely degraded or dissolved during the delignification and bleaching processes. A strong peak at ~3330 cm^{-1} remains, indicating the presence of the typical cellulose O–H group. Meanwhile, the band at ~2890 cm^{-1} indicates the C–H stretching of the alkyl group. The presence of a sharp peak at ~1050 cm^{-1} also indicates the presence of C–O stretching of secondary alcohols and ethers in the glucopyranose chain of cellulose. In the cellulose acetate spectrum (blue color), a new

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absorption band appears at around ~1740 cm^{-1}, which is characteristic of the carbonyl (C=O) stretching of the acetyl group, indicating that the acetylation reaction successfully formed acetate groups in cellulose. In addition, the intensity of the O–H band around ~3340 cm^{-1} decreased compared to pure cellulose, indicating that most of the hydroxyl groups had reacted with acetic anhydride. The peak at ~1240 cm^{-1} corresponding to the C–O stretching of the acetate group also became stronger, strengthening the evidence for successful acetylation.

To further support the qualitative FTIR interpretation, a semi-quantitative analysis based on relative peak intensity changes was also considered. In particular, the relative increase in the carbonyl (C=O) stretching band at approximately 1740 cm^{-1} and the concurrent decrease in the hydroxyl (O–H) stretching band around 3330–3340 cm^{-1} provide indirect evidence of successful acetylation. Although absolute quantification of functional groups by FTIR is limited without calibration and internal normalization, the increasing C=O/O–H intensity ratio has been widely used as a semi-quantitative indicator of acetyl group substitution in cellulose-based materials. The observed spectral trends in this study are consistent with the titration results, which indicate a high degree of acetylation and substitution, thereby reinforcing the reliability of the chemical modification interpretation (Islam et al., 2023; Grzybek et al., 2024; Sango et al., 2024).

4. Conclusion

This research successfully produced cellulose acetate from OPEFB-based MCC through an effective acetylation process under simple operating conditions. The obtained acetylation and substitution degrees were within the optimal range for membrane materials, with the best conditions achieved at a reaction time of three hours. FTIR analysis showed clear changes in chemical structure, characterized by the appearance of carbonyl groups from acetate and a decrease in the intensity of hydroxyl groups, thus confirming the success of the acetylation process. These findings demonstrate that MCC from local lignocellulosic biomass can be processed into cellulose acetate suitable for membrane technology applications, while supporting efforts to ensure sustainability and independence in membrane material production in Indonesia.

Despite the promising results, this study is limited to the synthesis and chemical characterization of cellulose acetate as a precursor material, without membrane fabrication or performance evaluation. Consequently, direct experimental validation of structure–performance relationships could not be established at this stage. In addition, the acetylation process was conducted at laboratory scale, and further studies are required to address scalability, solvent recovery, and economic feasibility. These limitations highlight the need for future work focusing on membrane fabrication, morphological analysis, and long-term performance assessment toward industrial application.

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