

Influence of Acetyl Content and Degree of Substitution on the Structural Properties of Cellulose Acetate for Pressure Retarded Osmosis

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Abstract

Cellulose acetate (CA) has been extensively utilized as a membrane material in pressure retarded osmosis (PRO) owing to its adjustable hydrophilicity, mechanical robustness, and chemical stability. Although earlier research has highlighted the role of acetylation in modifying the properties of CA, the specific impact of acetyl content and degree of substitution (DS) on structural features pertinent to PRO applications is still not well understood. The present study aims to fill this gap by systematically investigating CA derived from different lignocellulosic feedstocks. The purpose is to assess the relationship between acetyl content, DS, and the resulting structural properties of CA, and to determine the most appropriate biomass precursor for the development of PRO membranes. Cellulose was isolated from oil palm empty fruit bunches (EFB), rice straw, and waste paper, followed by acetylation. The obtained CA was characterized using Fourier-transform infrared spectroscopy (FTIR) to confirm esterification, while acetyl content and DS were quantitatively determined. Commercial CA served as the reference standard. The results demonstrated marked differences among the feedstocks. Waste paper yielded cellulose monoacetate (DS = 1.37; acetyl 26.9%), exhibiting incomplete acetylation with weak ester absorption. EFB produced cellulose diacetate (DS = 2.80; acetyl 43.0%), displaying spectral features comparable to commercial CA. Rice straw resulted in cellulose triacetate (DS = 3.91; acetyl 51.6%), indicating the most extensive substitution and superior structural quality. Higher DS was positively associated with enhanced thermal stability, reduced hydrophilicity, and improved mechanical performance, all of which are critical for PRO operation.

Keywords: Cellulose Acetate, Biomass, Degree Of Substitution, Acetyl Content, Pressure Retarded Osmosis (PRO)

INTRODUCTION

Pressure Retarded Osmosis (PRO) has emerged as a promising technology for osmotic energy conversion, utilizing the salinity gradient between two aqueous solutions to generate renewable “blue energy” (Loeb, 2002; Skilhagen et al., 2008). The efficiency of PRO systems strongly depends on the structural and chemical characteristics of the membranes employed, particularly their ability to balance water permeability, salt selectivity, and mechanical stability (Achilli et al., 2009). Among various polymeric materials, cellulose acetate (CA) has attracted continuous attention due to its abundance, biodegradability, cost-effectiveness, and well-established fabrication routes for asymmetric membranes (Achilli et al., 2009).

The performance of CA membranes is largely governed by the extent of acetylation of the cellulose backbone, which is typically described in terms of acetyl content and degree of substitution (DS). Acetyl content refers to the percentage of hydroxyl groups on the anhydroglucose unit substituted by acetyl groups, while DS indicates the average number of hydroxyl groups replaced per glucose unit, ranging from 0 (native cellulose) to 3 (cellulose triacetate) (Puls et al., 2011). Variations in these parameters directly influence the hydrophilic–hydrophobic balance, crystallinity, solubility, and mechanical strength of CA (Sanda et al., 2025; Science, 2019). Consequently, tailoring the acetyl content and DS offers a versatile strategy to optimize CA

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membranes for PRO applications ([Bolorunduro & Gasser, 2024](#); [Helfer et al., 2014](#)).

Spectroscopic and chemical analysis methods are frequently applied to evaluate the structural modification of CA. Fourier-transform infrared spectroscopy (FTIR) provides valuable information on functional groups, enabling qualitative and semi-quantitative estimation of acetyl substitution, while titration or elemental analysis allows more precise determination of acetyl content ([Beckers et al., 2003](#)). These analytical techniques not only validate the chemical structure of CA but also serve as predictive tools for understanding membrane performance in PRO systems ([Helfer et al., 2014](#)).

Despite the long-standing use of CA in forward osmosis and desalination membranes, limited studies have systematically correlated acetyl content and DS with the structural properties most relevant to PRO ([Altaee et al., 2018](#)). Addressing this gap is critical, as CA membranes with optimized substitution levels could provide enhanced water flux, reduced reverse salt transport, and improved long-term stability under PRO operating conditions.

In this work, cellulose acetate with varying acetyl content and DS was characterized by FTIR spectroscopy, acetyl content determination, and substitution analysis to establish the relationship between chemical composition and structural features. The findings are expected to provide fundamental insights into the design of CA membranes with tailored properties, thereby contributing to the advancement of PRO technology for sustainable energy generation.

LITERATURE REVIEW

Pressure Retarded Osmosis (PRO) has emerged as a promising technology for converting salinity gradient energy into usable power. This process utilizes the osmotic pressure difference between two solutions, in which a semi-permeable membrane selectively allows water transport while retaining ions. The efficiency of PRO systems is highly dependent on membrane properties, making the development of advanced polymeric materials a crucial aspect of research and industrial application ([Achilli & Childress, 2010](#)).

Among various polymers, cellulose acetate (CA) has been widely used due to its affordability, renewability, and favorable mechanical properties. In particular, cellulose triacetate (CTA) membranes have been established as benchmarks in PRO studies, often fabricated in hollow-fiber configurations to withstand high osmotic gradients while maintaining substantial power density ([Kakihana et al., 2021](#)). The functional performance of CA membranes is closely related to their acetyl content and degree of substitution (DS). Acetyl content reflects the proportion of acetyl groups, while DS describes the average number of hydroxyl groups substituted per anhydroglucose unit (ranging from 0 to 3). These parameters significantly influence membrane hydrophilicity, crystallinity, solubility, and pore structure, which directly affect permeability and selectivity ([Kakihana et al., 2021](#)).

For characterization, Fourier Transform Infrared Spectroscopy (FTIR) is one of the most widely applied methods. The characteristic absorption bands of carbonyl (C=O) and ether (C–O–C) groups provide insights into substitution levels and structural modifications. FTIR is often complemented with titration, XRD, SEM, and thermal analysis for a more comprehensive understanding of structure–function relationships ([Wu et al., 2014](#)).

Although CA-based membranes have been extensively investigated, many studies emphasize operational performance under different PRO conditions rather than establishing a systematic link between acetyl content, DS, and structural attributes. Bridging this gap is crucial to improve membrane stability, enhance water permeability, and reduce internal concentration polarization, major challenges in PRO applications ([Vatanpour et al., 2022](#)). Therefore, integrated studies combining chemical analysis (acetyl content, DS), FTIR characterization, and structural evaluation (SEM, XRD, TGA, contact angle) are essential. Such approaches will provide a more fundamental

understanding of how chemical composition governs the structure and performance of CA membranes, enabling the rational design of next-generation membranes for sustainable osmotic energy conversion ([Achilli et al., 2009](#)).

RESEARCH METHOD

Cellulose Synthesis

Cellulose was isolated from rice straw, empty fruit bunches (EFB), and wastepaper through a sequential chemical treatment. Initially, each feedstock was subjected to acid hydrolysis using 10% H_2SO_4 solution at a ratio of 10 mL per gram of biomass under reflux at 100 °C for 40 min. The hydrolyzed suspensions were filtered and rinsed with distilled water until reaching neutral pH. The retained solids were subsequently treated with 5% NaOH solution (10 mL per gram) at 100 °C for 60 min to eliminate hemicellulose and lignin, followed by washing to neutrality. A bleaching step was then performed using 30% H_2O_2 solution (10 mL per gram) at 95 °C for 60 min. After thorough washing, the purified residues were oven-dried at 60 °C for 24 h, yielding cellulose from straw, EFB, and wastepaper as separate fractions.

Cellulose Acetylation

The cellulose obtained from each feedstock was converted into cellulose acetate via acetylation. In a typical procedure, 10 g of purified cellulose was dispersed in 125 mL of glacial acetic acid and stirred at 50 °C for 60 min. Subsequently, 15 drops of concentrated H_2SO_4 were introduced as a catalyst, followed by the addition of 75 mL acetic anhydride, and the mixture was further stirred at 40 °C for 2 h. After the reaction period, the solution was kept undisturbed for 24 h to ensure complete acetylation. To stabilize the system, 125 mL of glacial acetic acid was added, and the mixture was stirred again at 50 °C for 60 min. The resulting product was repeatedly washed with distilled water preheated to 60 °C until a neutral pH was obtained. Finally, the neutralized cellulose acetate was oven-dried at 60 °C for 24 h to yield cellulose acetate powder.

FTIR Analysis

Fourier Transform Infrared (FTIR) spectroscopy of the cellulose acetate sample was conducted using a Perkin-Elmer UATR Spectrum Two instrument. FTIR is a reliable analytical technique for identifying functional groups and elucidating the chemical structure of materials. The obtained spectra revealed characteristic absorption bands corresponding to ester carbonyl ($\text{C}=\text{O}$) and hydroxyl ($-\text{OH}$) groups, confirming the successful acetylation of cellulose. These spectral features provide valuable insights into the degree of substitution and overall chemical composition, which are critical parameters in evaluating the physicochemical properties and potential applications of cellulose acetate.

FINDINGS AND DISCUSSION

Finding

Determination of the degree of substitution (DS)

1.0 g of CA sample was weighed accurately and transferred to a 250 mL conical flask, and then 40 mL of 75% aqueous ethanol were added. The flask, loosely stoppered, was heated to 55 °C for 1 h for better swelling of the sample. Then 40 mL of 0.5 mol.L⁻¹ NaOH solution, accurately measured, was added to each sample and the mixture was again heated at 55 °C for 1 h. The same procedure was also done for a control system (without CA). The flasks were stoppered tightly and allowed to stand at room temperature for 72 h. The excess alkali in the sample and control was titrated with 0.5 mol.L⁻¹ HCl solution using phenolphthalein as an indicator. An excess amount of acid was added (1 mL) and the alkali was allowed to diffuse from the regenerated cellulose overnight. The

disappearance of the pink color indicated complete neutralization of the alkali. The small excess of acid was then back titrated with 0.5 mol.L⁻¹ NaOH solution to a phenolphthalein end point (until the solution had acquired a faint pink color). To reduce errors and confirm the results, each experiment was carried out in triplicate. The acetyl content (% A) was calculated according to eqn (1).

$$\%A = \frac{[(DC)Na + (AB)N] \times 4.305}{W} \quad (1)$$

Where, A and B are the volumes (mL) of NaOH solution required for the titration of the sample and blank, respectively; C and D are the volumes (mL) of HCl solution required for the titration of the sample and blank, respectively; Na and No are the normalities of HCl solution and NaOH solution, respectively; and W is the mass of the CA sample used. The % A value obtained from eqn (3) was used to calculate the DS according to eqn (2).

$$DS = \frac{162 \times \% A}{(430543 \times \% A)} \quad (2)$$

Discussion

Cellulose Acetylation

The characterization of cellulose acetate (CA) synthesized from different biomass feedstocks is presented in Figure 1. The cellulose acetate derived from empty fruit bunches (EFB) of oil palm in Figure 1.a exhibits a light brown coloration with a relatively fine and compact texture. This brownish appearance is mainly attributed to residual lignin and other impurities that were not completely removed during the cellulose isolation and acetylation processes (De France et al., 2020). In contrast, cellulose acetate obtained from rice straw in Figure 1.b shows a darker brown color and a coarser texture compared to that from EFB. This observation is strongly influenced by the high silica content in rice straw, which is difficult to remove completely and consequently affects both the morphology and coloration of the final product (Seif Sahandi et al., 2019).

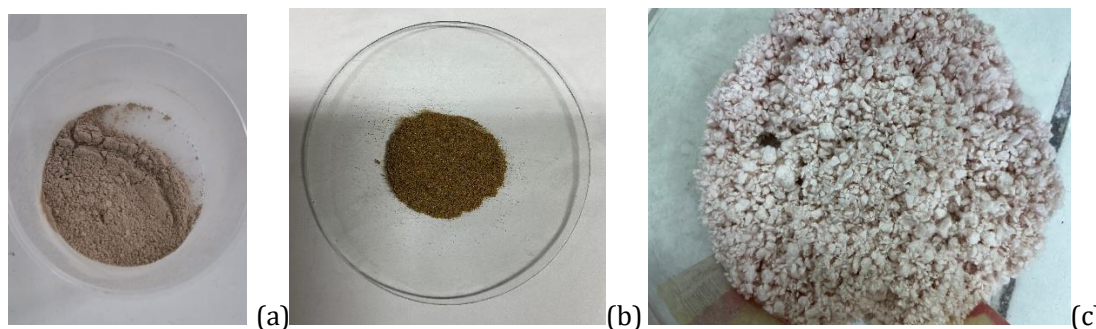


Figure 1. (a) Cellulose Acetate from empty fruit bunches of oil palm (EFB); (b) Cellulose Acetate from Rice Straw; (c) Cellulose Acetate from Waste Paper.

Meanwhile, cellulose acetate synthesized from waste paper in Figure 1.c displays a lighter, whitish-yellow color with a porous and aggregated structure. Compared to EFB and rice straw, waste paper tends to produce cellulose acetate with a brighter appearance due to its low lignin content, resulting from prior bleaching during paper manufacturing. However, its porous morphology indicates structural rearrangements during acetylation, which differ from the other two raw materials (Singh, 2015). Overall, the variations in color and morphology among the three cellulose acetate products are influenced by the type of raw material, particularly the presence of

lignin, hemicellulose, and mineral components. These findings indicate that waste paper shows promising potential for producing cellulose acetate with better color quality, while EFB and rice straw require further optimization to minimize impurities and improve the final product characteristics (Alencar et al., 2022).

FTIR Analysis

The FTIR analysis was conducted to validate the formation of cellulose acetate derived from various raw materials, namely waste paper, empty fruit bunches of oil palm (EFB), and rice straw, with commercial cellulose acetate used as the reference. The FTIR from Figure 1. spectrum of cellulose acetate derived from waste paper exhibited absorption bands at 3333 cm^{-1} (O–H stretching), 2902 cm^{-1} (C–H stretching), and in the range of $1158\text{--}1030\text{ cm}^{-1}$, corresponding to C–O–C vibrations. However, no clear peak was observed in the $1730\text{--}1750\text{ cm}^{-1}$ region, which is the key indicator of acetyl group formation. This suggests that the acetylation process was incomplete, resulting in a low degree of substitution, and the structure still closely resembles native cellulose rather than cellulose acetate (Bahmid et al., 2013).

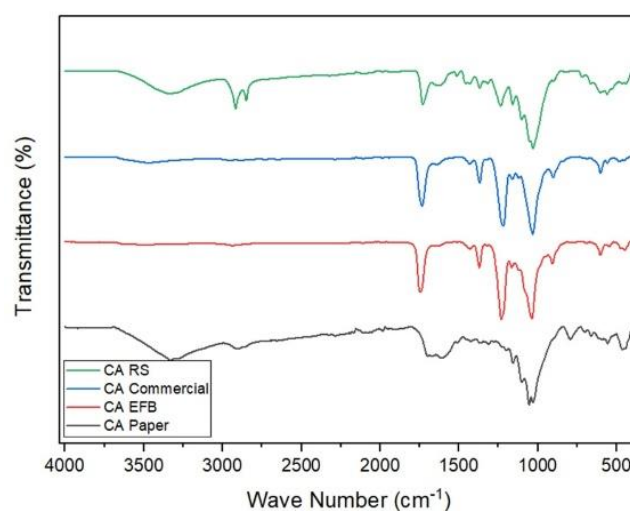


Figure 2. FTIR spectra of the cellulose Acetate

In contrast, the FTIR spectrum of cellulose acetate synthesized from EFB clearly showed a strong absorption at 1746 cm^{-1} , assigned to the C=O stretching of the ester group, and a peak at 1231 cm^{-1} corresponding to C–O stretching of the ester. These features were also observed in commercial cellulose acetate (1734 cm^{-1} and 1219 cm^{-1}), confirming that cellulose from EFB had undergone successful acetylation. Additional supporting bands such as those at 2935 cm^{-1} (C–H stretching), 1370 cm^{-1} (C–H bending), and $1165\text{--}1036\text{ cm}^{-1}$ (C–O–C) further validate the conversion of cellulose into cellulose acetate (Beckers et al., 2003).

Similarly, the cellulose acetate obtained from rice straw displayed the characteristic peaks of acetyl groups at 1729 cm^{-1} (C=O ester) and 1236 cm^{-1} (C–O ester). However, the O–H stretching band at 3336 cm^{-1} remained relatively strong, indicating that a considerable number of hydroxyl groups were not substituted. This result suggests that the acetylation process in rice straw was partially successful, yielding cellulose acetate with a lower degree of substitution compared to EFB. Overall, the FTIR comparison indicates that commercial cellulose acetate, as expected, shows the most consistent spectral features with literature values, including the ester-specific peaks at 1734 cm^{-1} and 1219 cm^{-1} . Among the synthesized samples, cellulose acetate from EFB exhibits the closest similarity to the commercial standard, followed by rice straw, while cellulose acetate from

waste paper is the least consistent due to the absence of ester carbonyl absorption (Wu et al., 2014; Yan et al., 2021).

Therefore, the order of spectral similarity to commercial cellulose acetate can be summarized as follows:

- Commercial cellulose acetate (reference standard) > Cellulose acetate from EFB (most comparable to the standard) > Cellulose acetate from rice straw (moderately comparable, partial acetylation) > Cellulose acetate from waste paper (least comparable, acetylation not optimal).

Determination of the degree of substitution (DS)

Cellulose acetate can be classified into three categories: cellulose monoacetate ($0 < DS < 2$; acetyl content $< 35\%$) and cellulose diacetate ($DS 2.0-2.8$; acetyl content $35-43.5\%$) (Ashter, 2018). Meanwhile, Cellulose triacetate ($DS > 2.9$) (Ashter, 2018). A higher DS reflects a higher degree of acetylation, which is typically associated with improved physicochemical properties such as melting point and solubility.

Table 1. Classification of Cellulose Acetate Based on DS and Acetyl Content

Source Material	DS	Acetyl Content (%)	Solubility	Classification
Waste Paper	1.37	26.875	Soluble in water	Monoacetate
Oil Palm EFB	2.80	43.0	Limited solubility in organic solvents	Diacetate (upper limit, close to triacetate)
Rice Straw	3.91	51.6	Insoluble in water, soluble in organic solvents	Triacetate

The results show that cellulose acetate derived from waste paper exhibited an acetyl content of 26.875% with $DS = 1.37$ can be seen in Table 1. These values fall within the monoacetate category, indicating that the acetylation process was incomplete. This finding is consistent with the FTIR spectra, which lacked a strong ester carbonyl absorption band at $1730-1750\text{ cm}^{-1}$. In contrast, cellulose acetate synthesized from oil palm empty fruit bunches (EFB) exhibited an acetyl content of 43% with $DS = 2.8$. This value lies at the upper boundary of the diacetate category and approaches the triacetate range. This indicates that the acetylation process for EFB was relatively efficient, with most hydroxyl groups successfully substituted by acetyl groups.

Cellulose acetate obtained from rice straw showed the highest acetyl content, reaching 51.6%, with $DS = 3.91$. Cellulose with a DS higher than 2.9 is classified as cellulose triacetate. Therefore, the result indicates that the synthesized cellulose acetate from rice straw can be categorized as cellulose triacetate, which generally possesses higher thermal stability, lower hydrophilicity, and improved mechanical properties compared to diacetate (Kakihana et al., 2021). These findings are consistent with the FTIR analysis, in which EFB and rice straw exhibited more pronounced ester-specific peaks ($C=O$ and $C-O$ stretching) compared to waste paper. Therefore, rice straw and EFB appear to be more suitable raw materials for producing cellulose acetate, both in terms of FTIR spectral validation and degree of substitution values (Ashter, 2018; Science, 2019).

CONCLUSIONS

The study demonstrates that the acetyl content and degree of substitution (DS) significantly influence the structural properties and potential performance of cellulose acetate (CA) in pressure retarded osmosis (PRO) applications. Cellulose acetate synthesized from different biomass feedstocks exhibited distinct physicochemical characteristics due to variations in lignin, hemicellulose, and mineral content.

FTIR analysis confirmed that CA derived from oil palm empty fruit bunches (EFB) and rice straw underwent more successful acetylation compared to waste paper. The strongest ester-specific absorption bands observed in EFB- and rice straw-based CA validated their higher substitution levels. Determination of DS further supported this finding, where EFB produced cellulose diacetate at the upper boundary (DS = 2.80; acetyl content = 43.0%), while rice straw yielded cellulose triacetate (DS = 3.91; acetyl content = 51.6%). Conversely, waste paper resulted in cellulose monoacetate (DS = 1.37; acetyl content = 26.9%), reflecting incomplete acetylation.

From a structural perspective, higher DS values were associated with reduced hydrophilicity, improved thermal stability, and enhanced mechanical integrity. These attributes are particularly advantageous for PRO membranes, which require a delicate balance between water permeability and salt rejection, alongside sufficient mechanical robustness under hydraulic pressure. Hence, rice straw, with its triacetate characteristics, presents the most promising raw material for producing CA membranes suitable for PRO, while EFB also offers considerable potential.

In conclusion, acetyl content and DS play a pivotal role in determining the structural and functional properties of cellulose acetate for PRO. Rice straw and EFB represent promising feedstocks for high-performance membrane materials, provided that further optimization and performance validation are carried out.

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LIMITATIONS & FURTHER RESEARCH

This study is limited by the variability of cellulose sources, the narrow range of acetyl content and degree of substitution examined, and the use of controlled laboratory conditions that may not fully reflect industrial environments. The characterization methods were restricted to conventional techniques, while advanced analyses could provide deeper molecular insights. In addition, membrane performance was only evaluated in short-term PRO tests without considering long-term stability, fouling, or hydrodynamic factors that influence large-scale applications.

However, further research is needed to bridge the gap between laboratory-scale synthesis and practical membrane fabrication. Future studies should focus on:

1. Optimization of Acetylation Processes: Refining reaction parameters to maximize acetyl content and minimize residual impurities, particularly for lignin- and silica-rich feedstocks such as EFB and rice straw.
2. Membrane Performance Evaluation: Investigating the transport properties of CA membranes (water flux, salt rejection, and power density) under PRO operating conditions.
3. Blending and Composite Approaches: Exploring the incorporation of nanoparticles, hydrophilic additives, or copolymers to fine-tune the permeability-selectivity trade-off and improve antifouling properties.

4. Long-Term Stability Testing: Assessing chemical, thermal, and mechanical durability of CA membranes with different DS values in simulated PRO environments.
5. Sustainable Feedstock Utilization: Expanding the use of agricultural and industrial waste-derived cellulose for large-scale, cost-effective, and environmentally friendly CA production.

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