

Solvent effect on film microstructure

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Research Article

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Abstract:

The replacement of conventional solvents with more sustainable alternatives remains a significant challenge in the fabrication of cellulose acetate (CA) membranes, particularly in controlling membrane morphology. In this study, ethyl lactate (EtLa) and dimethyl carbonate (DMC), together with acetone as a borderline green solvent, were investigated as more sustainable alternatives for the preparation of CA films and compared with conventional solvents, including N-methyl-2-pyrrolidone (NMP), N,N-dimethylformamide (DMF), and N,N-dimethylacetamide (DMAc). The membranes were prepared by non-solvent-induced phase separation (NIPS) and characterized in terms of morphology, pore structure, crystallinity, and wettability. This study systematically investigates the relationship between solvent properties, phase-inversion kinetics, and the resulting CA membrane morphology, highlighting the importance of considering solvent volatility and solvent–nonsolvent interactions when selecting more sustainable solvents for membrane fabrication.

Keywords: cellulose acetate; green solvents; membrane morphology; phase inversion; solvent selection; membrane fabrication

Introduction

Cellulose acetate (CA) is one of the most widely used and researched cellulose derivatives especially in membrane technologies, packaging industry and biomedical applications (Goñi-Ciaurriz et al. 2025; Lindsey et al. 2025; Alkaron et al. 2026; Gopika et al.). In the case of membranes, CA has become one of the most predominant polymers used for the fabrication of both porous and dense membranes. The widespread use of CA is largely attributed to its numerous advantageous properties. CA combines good film forming ability, moderate hydrophilicity, tunable mechanical strength and partial biodegradability. (Fischer et al. 2008; Kalita and Hakkarainen 2023; El-Badry et al. 2025) It is also thermally and chemically stable and nontoxic. Another advantage is its easy processability which makes it adaptable into different forms such as powder, sheets, fibers and hollow fibres. (Vatanpour et al. 2022). Despite its wide application, CA also has certain limitations, such as low chemical resistance, moderate thermal stability, lower water flux, and susceptibility to fouling (Sango et al. 2023; Waleed et al. 2025; Chen et al. 2025). Nevertheless, some of these limitations can be addressed by modifying the fabrication process.

The preparation of membranes is also closely related to various parameters such as the molecular weight of the polymer, the choice of solvent, the concentration of the polymer solution, the humidity of the environment during preparation, the evaporation time, the composition of the coagulation bath, and also the drying method. (Fu et al. 2008; Ghaffarian et al. 2015; Majumder et al. 2019; Mohammed Ali et al. 2020). The choice of solvent is one of the most critical aspects influencing the resulting structure of the membrane. Among the most commonly reported are N-methyl-2- pyrrolidone (NMP), dimethylacetamide (DMAc), dimethylformamide (DMF) and tetrahydrofuran (THF). However, because of their know toxicity and ecotoxicity risks, there is a continuous transfer toward green chemistry principles. These principles provide framework for reducing the environmental impact of chemical processes. Between these principles, safer solvent use is mentioned. (Amoneit et al. 2024) Therefore green solvents as an alternative to organic solvents have been introduced. Among the different classes of green solvents, bio-based solvents represent a particularly attractive alternative due to their availability from renewable sources, relatively low cost, and commercial availability. Among these, ethyl lactate (EL) and dimethyl carbonate (DMC) have been reported as promising commercially available bio-based solvents for cellulose acetate. (Doble and Kruthiventi 2007) Acetone (Acet), although often classified as a conventional solvent, can be considered a transitional or borderline green solvent depending on its production route and the overall outlook. (Soroko et al. 2011; Kurada et al. 2020)

Although several comparative studies exist, they are typically limited to a narrow selection of solvents or lack a systematic connection between solvent physical-chemical properties, polymer–solvent interactions, and the resulting membrane structure. In this context, the present work aims to provide a systematic comparison across a broader solvent spectrum, ranging from conventional to more sustainable alternatives. At the same time, membrane preparation is carried out using simple and reproducible procedures to maintain relevance for

potential scale-up. This approach enables a consistent evaluation of solvent effects while establishing a link between solvent characteristics, phase inversion behavior, and the resulting morphology and properties of CA membranes.

Materials and methods

Materials

Commercial grade cellulose acetate (CA, 50 kDa molecular weight and 39.7 wt.% acetyl content) was supplied by Sigma-Aldrich Co. *N*-methyl-2-pyrrolidone (NMP) with a purity $\geq 99\%$ ($d = 1.028 \text{ g mL}^{-1}$, at 25°C), *N,N*-dimethylacetamide (DMAc) with a purity $\geq 98\%$ ($d = 0.937 \text{ g mL}^{-1}$, at 25°C), dimethylformamide (DMF) with a purity $\geq 99\%$ ($d = 0.948 \text{ g mL}^{-1}$, at 25°C) and tetrahydrofuran (THF) with a purity $\geq 99.8\%$ ($d = 0.887 \text{ g mL}^{-1}$, at 25°C) were all purchased from Penta Chemicals Unlimited Czech Republic and used as conventional non-biodegradable solvents. As green solvents acetone with a purity $\geq 99\%$ ($d = 0.79 \text{ g mL}^{-1}$, at 25°C) by Avantor ScienceCentral, ethyl lactate with a purity $\geq 99\%$ ($d = 1.03 \text{ g mL}^{-1}$, at 20°C) and dimethyl carbonate with a purity $\geq 99\%$ ($d = 1.07 \text{ g mL}^{-1}$, at 20°C) were supplied by Sigma-Aldrich Co. Deionized water was used for all prepared solutions as a coagulation bath.

Membrane preparation

CA solutions were prepared using NMP, DMAc, DMF, THF, EtLa, DMC, and Acet as solvents. Based on preliminary studies, the concentration of 8 wt% was selected for all solutions. The required amounts of CA and the respective solvent were added to reagent bottles and stirred under ambient laboratory conditions or, depending on the solvent, under mild heating up to 50°C until a homogeneous solution was obtained. The prepared solutions were subsequently left to degas to remove entrapped air bubbles. Each solution was then casted onto a stainless-steel substrate using a $300 \mu\text{m}$ casting knife. The resulting films were allowed to undergo solvent evaporation in a fume hood at $22 \pm 2^\circ\text{C}$ and $35 \pm 5\%$ RH until nearly dry. The partially dried films were subsequently immersed in a demineralized-water coagulation bath. The membranes were then washed twice with fresh demineralized water. Finally, the washed films were freeze-dried and subsequently analyzed.

Characterization of the morphology of the films

The cross-sectional morphologies of the membranes were examined using the JEOL JSM-7610F Plus scanning transmission electron microscope (STEM). The samples were scanned at an accelerating voltage of 15 kV using secondary electron detection. Before the STEM analysis, the samples were sputtered with 20 nm platinum layer using sputtering advice SC 7620 mini (Edlin, Libis). The distribution of pore sizes and the porosity of the samples were then determined from the SEM images in ImageJ programme (National Institutes of Health, USA). (He et al. 2025)

Characterization of the structural properties

Fourier-transform infrared (FT-IR) spectroscopy (Nicolet 6700, Thermo Nicolet) was performed in the range of $400\text{--}4000 \text{ cm}^{-1}$ using 32 scans in ATR mode with baseline correction to identify membrane components and verify the absence of residual solvents. To better understand the nature of polymer–solvent interactions, FTIR analysis was additionally performed using a solvent subtraction approach. The spectra were obtained by measuring the polymer solutions against both air and pure solvent backgrounds and subsequently comparing them with the spectra of pristine CA powder and the corresponding solvents. Comparison of the spectra acquired under different background conditions enabled the distinction between solvent-derived contributions and spectral changes associated with polymer–solvent interactions.

To determine crystallinity XRD patterns were obtained using a Rigaku SmartLab diffractometer (Rigaku, Tokyo, Japan) equipped with a D/teX Ultra 250 detector. Cu $K\alpha$ radiation ($\lambda_1 = 0.154059 \text{ nm}$, $\lambda_2 = 0.154441 \text{ nm}$) operated at 40 kV and 30 mA was used as the X-ray source. The incident and diffracted beam optics were equipped with 5° Soller slits. The incident slit was fixed at 1° divergence, while the diffracted beam slits were fixed at 5 and 10 mm. The thermal behavior of the cast films was studied by differential scanning calorimetry (DSC) using a TA 2000 calorimeter under a nitrogen atmosphere (50 mL/min). The samples were subjected to a heating ramp from -80 to 280°C at 10°C/min . The crystalline fraction of the cast materials, χ , was calculated by dividing the melting enthalpy of the samples by the enthalpy of 100% crystalline triacetate cellulose, which is $58.8 \text{ J}\cdot\text{g}^{-1}$, (Cerqueira et al. 2006). To compare the porosity and wettability, water contact angles were measured using a contact angle analyzer (OCA 15, DataPhysics).

Hansens solubility parameters

Hansens solubility parameters (HSP) have been investigated and used as a framework for the prediction of the interaction between each solvent and CA (Hansen 2007). The concept of HSP is based on the cohesive energy of a substance. Cohesive energy describes the energy required to overcome the attractive intermolecular forces that hold the molecules of a substance together. In case of a solvent, it can be defined as the energy required to separate the solvent molecules into the gas phase. (Glasser and Sheppard 2016) For comparing the substances of different molecular sizes, the cohesive energy is expressed per unit of molar volume as the cohesive energy density. HSP divides cohesive energy density (δ) into three distinct contributions: dispersion forces (δD), polar interactions (δP), and hydrogen bonding (δH) as described by equation (1). Each of the forces are presented for the CA as well as the solvents and nonsolvent used in table 1. (Hansen 2007)

$$\delta^2 = \delta D^2 + \delta P^2 + \delta H^2 \quad (1)$$

Each substance can thus be represented as a point in a three-dimensional parameter space defined by these components. The affinity between a solvent and a solute is evaluated by calculating the distance between their respective coordinates in this space (R_a) described by equation (2); smaller distances indicate greater compatibility and a higher likelihood of dissolution.

$$R_a^2 = 4(\delta D_1 - \delta D_2)^2 + (\delta P_1 - \delta P_2)^2 + (\delta H_1 - \delta H_2)^2 \quad (2)$$

Based on this, the Relative Energy Difference (RED) parameter can be introduced to indicate the position of a solvent within the Hansen solubility space as described by equation (3). The lower the RED value is, the better solvent for the polymer.

$$RED = R_a/R_0 \quad (3)$$

Table 1 Hansen solubility parameters (HSP) - dispersion (δD), polar (δP), and hydrogen-bonding (δH) contributions of CA, the solvents and nonsolvent investigated for film preparation (Hansen 2007)

Chemicals	δ disp	δ polar	δ hydrogen
Cellulose Acetate (CA)	17.1	13.1	9.4
Water	15.5	16.0	11.3
NMP	18.0	12.3	7.2
DMAc	16.8	11.5	10.2
DMF	17.4	13.7	11.3
THF	16.8	5.7	8.0
EtLA	16.0	7.6	12.5
DMC	15.5	3.9	9.7
Acet	15.5	10.4	7.0

Results and discussion

The vapor pressure of the casting solvent provides an important first indication of expected behavior during membrane formation. Accordingly, increasing solvent vapor pressure is expected to accelerate solvent loss during casting, reduce the amount of residual solvent available for subsequent solvent–nonsolvent exchange, and thereby modify the extent and kinetics of phase inversion process. However, the kinetics of phase inversion may also be governed by the miscibility of the solvent with nonsolvent. (Manholi and Athiyanathil 2022)

The experimental morphologies observed by cross-sectional SEM broadly support these relationships. The highly volatile solvents underwent particularly rapid evaporation during casting. Consequently, prior to immersion into the coagulation bath, the resulting films appeared to be almost dry indicating that only a residual amount of solvent remained available to participate in the subsequent phase-inversion process. This substantially reduced the

influence of the coagulation bath and miscibility of these solvents with nonsolvent and resulted in comparatively dense morphologies. The observations indicate that, at sufficiently high solvent volatility, rapid evaporation can suppress the development of the porous structure typically associated with immersion precipitation. (Guan et al. 2006)

The observed casting and coagulation behavior therefore suggests a two-stage influence of solvent volatility. During casting, vapor pressure controls the extent of solvent evaporation and consequently determines the residual solvent content of the film at the onset of coagulation. During immersion, this residual solvent content determines the extent to which solvent–nonsolvent exchange and phase separation can proceed. However, the kinetics are also influenced by the miscibility of solvent-nonsolvent.

Overall, the results indicate that increasing solvent vapor pressure generally leads to a decrease in membrane porosity. However, this relationship is not linear and cannot fully account for the observed differences in pore morphology. Instead, vapor pressure acts primarily by regulating the residual solvent content prior to immersion, which subsequently controls the kinetics and extent of phase inversion. The final membrane morphology is therefore determined by the coupled effects of solvent evaporation during casting and solvent–nonsolvent exchange during coagulation as well as the miscibility of solvent-nonsolvent. This distinction is particularly important for the “green” solvents EtLa and DMC, whose intermediate volatility produces morphologies that cannot be predicted from vapor pressure alone. The results consequently highlight that solvent selection for phase-inversion membrane fabrication must consider not only volatility, but also the subsequent coagulation behavior and the associated mass-transfer kinetics which could be however further researched by the optimization of the composition of coagulation bath.

Pore size and porosity

The quantitative analysis of the cross-sectional SEM images further confirms the strong influence of solvent selection on the resulting membrane morphology. The findings from both average pore size and porosity further support the conclusion that solvent vapor pressure primarily influences membrane morphology indirectly by controlling the amount of solvent retained in the polymer film before immersion. The retained solvent then determines the extent of solvent–nonsolvent exchange and while also depending on the miscibility of solvent-nonsolvent. Consequently, the kinetics and degree of phase separation during coagulation are influenced. Therefore, while increasing solvent volatility generally tends to reduce the overall porosity, the resulting pore size and morphology depend on the coupled effects of solvent evaporation and coagulation-induced phase inversion.

Solvent influence on structural properties of the films

The spectra indicate that the overall chemical structure of CA remained preserved after processing with different solvents. However, variations in band intensities suggest differences in intermolecular interactions between CA and the respective solvents. The observations indicate that all investigated solvents acted as effective solvents for CA promoting the unwrapping and expansion of the polymer chains within the solvent environment without inducing chemical degradation or structural alteration of the polymer backbone. Overall, the FTIR analysis suggests that solvent treatment primarily induced dipole-dipole interactions of the acetyl groups of cellulose acetate while affecting the conformational organization and packing of the polymer chains.

The thin polymeric films were also tested on XRD to detect the crystallinity and their differences. Overall, all samples exhibited predominantly amorphous structures. Although XRD is widely used to assess the crystallinity of polymers, the significant contribution of the amorphous halo to the diffractogram may compromise the accuracy of crystallinity calculations. Consequently, DSC was additionally used to determine the degree of crystallinity, while also enabling the evaluation of the thermal properties of the films. The nature of the solvent had a pronounced effect on the melting enthalpy and, consequently, on the degree of crystallinity of the prepared films. The findings from DSC show that both rapid solvent evaporation and slow phase inversion suppress the formation of crystalline domains, resulting in predominantly amorphous films with crystallinity below 10%. In contrast, solvents with longer evaporation times and faster phase inversion promoted the formation of more ordered crystalline regions. Nevertheless, all prepared films remained predominantly amorphous as seen in XRD, although the observed differences in crystallinity are reflected in their microstructure and pore-forming behavior. Thermal behavior also responds to the crystallinity percentage, where the samples with higher show higher melting temperatures than the others which indicates that with higher crystallinity of the film, the melting temperature increases (Cheng et al. 2026).

Wettability

The water contact angle is used to determine the hydrophilicity of a membrane and it has significant impact on the permeation rate. It should be noted that during contact angle measurements, rapid absorption of water into the membrane was observed while maintaining their structural integrity without dissolution. Consequently, the contact angle decreased rapidly within seconds. The results are indicating that wettability is primarily governed by the intrinsic properties of the polymer, while the choice of solvent has a limited direct influence on surface wettability, especially because there is only residual amount (Tretinnikov 1999). However, the solvent significantly affects membrane morphology, which in turn can influence the apparent contact angle through differences in water uptake and penetration.

Conclusions

This study demonstrates that cellulose acetate (CA) film morphology is governed by the combined effects of vapor pressure inducing solvent volatility and retention of solvent in the casted film, solvent–polymer interactions, and solvent–nonsolvent exchange and their miscibility. While NMP, DMAc and DMF provided the most favorable solvent–polymer compatibility, the results also demonstrate that the greener alternatives ethyl lactate (EtLa) and dimethyl carbonate (DMC) can successfully be used for CA film preparation and provide distinct morphological outcomes. DMF and DMAc produced the most porous structures, whereas THF and acetone resulted in dense morphologies due to rapid solvent evaporation. EtLa produced relatively large pores with moderate porosity, indicating its potential as a greener solvent for porous CA membrane applications. In contrast, DMC generated a comparatively dense structure with limited porosity, suggesting its potential for applications where denser CA films are desired. Overall, the findings demonstrate that greener solvents do not merely represent substitutes for conventional solvents but can provide distinct opportunities for tailoring CA membrane morphology. Their selection should therefore consider both solvent–polymer compatibility and phase-inversion kinetics for the targeted application.

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Conflict of interest

The authors have no relevant financial or non-financial interests to disclose.

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Additional information

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