

Polycaprolactone Blended Cellulose Acetate Thin Film Composite Membrane for Dairy Waste Treatment Using Forward Osmosis

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Abstract

In recent years new sustainable technology for wastewater treatment has emerged, and among them, Forward osmosis (FO) has gained importance. FO utilizes osmotic pressure difference across the semipermeable membrane as the driving force to concentrate the wastewater. Further, the surface and physical properties of the semipermeable FO membrane play a crucial role during the FO process to reduce the internal concentration polarization. In general, FO membranes are prepared using cellulose acetate (CA) polymer due to their high hydrophilic nature. However, CA membranes are mechanically unstable for the FO process. Hence, to increase the mechanical strength and flexibility of CA, other polymers are blended along with it. In this present study, we have prepared a Phase-Inversion membrane using CA blended with polycaprolactone (PCL) polymers. Further, to increase the hydrophilicity of the membrane, a thin film composite (TFC) layer of polyamide is coated using interfacial polymerization. To increase the antifouling properties of the membrane, Graphene oxide (GO) and copper oxide (CuO) nanoparticles (NPs) are incorporated inside the TFC matrix. The prepared NPs and membrane were characterized using Fourier-transform infrared spectroscopy (FTIR), Wide-angle X-ray scattering (WAXD), and contact angle. Further, the GO-CuO incorporated TFC coating has improved the hydrophilicity and antifouling properties of the membrane. It was observed that the water flux has increased up to 5 LMH, and reverse solute flux has reduced to 4 GMH. Further, the membrane was utilized to concentrate in situ prepared dairy waste. It was observed that after 60 mins of the FO process, the concentration of dairy waste has increased to 23%, with a concentration factor of 0.903. Thus, prepared TFC phase inversion membrane is potential for dairy wastewater treatment.

1. Introduction

The essential contribution to sustaining a healthy environment is water. Through works, it has been established that about two-thirds of the earth's surface is enclosed with water, out of which only 2.5% is fresh water and an unbiased 0.3% is accessible for human consumption. The increase in population, industrialization, and improved living standards has upsurged the demand for freshwater, thereby making people aware of its importance and vulnerability in terms of quality. This issue can be addressed by incorporating a proper wastewater treatment methodology. The traditional wastewater treatment approaches include filtration, coagulation-flocculation, or biological treatment. However, among these various methods, the most acknowledged technique is Membrane technology. Membrane technology has shown significant progress in the last decades owing to its easy accessibility and environmental friendliness. Several membrane processes that have been tried earlier for treating wastewater are ultrafiltration (UF), microfiltration (MF), nanofiltration (NF), membrane distillation (MD), and reverse osmosis (RO). Amongst these, RO has received a greater appreciation for water reclamation due to its higher efficiency. This technology was anticipated to bridge the gap between economics and sustainability.(Obaid et al. 2016)

Dairy processing is now one of the largest sources of industrial wastewater due to rapid industrialization. Approximately 60-80 percent of the water consumed in the industry is contaminated. The production of

several food items from raw milk resulted in the formation of a diverse run-off stream with qualitative and quantifiable variations, which is restricted for further reuse. The primary characteristics of dairy wastewater are high temperature, high organic content, and a wide pH range. To avoid environmental damage, the wastewater from the dairy industries must be treated. Milk production results in the production of wastewater containing similar constituents. The type of treatment required is heavily influenced by the composition and flow rate of the wastewater. Because of the high energy requirements and significant fouling of membranes, pressure-driven membrane processes cannot be used to treat this contaminated water. Also, this pressure-driven process entails a large energy requirement and is costly. A new energy-efficient technique, Forward Osmosis (FO), was introduced to encounter water stresses cautiously.

Forward Osmosis is an evolving energy-efficient membrane separation technology proposed in the 1970s. It trails the natural phenomenon of osmosis, involving the passage of water crossways towards the membrane from the low concentration region, known as feed solution, to the high concentration region, known as draw solution. (Cath et al. 2006) This movement of water molecules across the membrane due to the osmotic pressure gradient between the two solutions. The main advantage of FO is that it does not require any external pressure, which aids in the minimal fouling of membranes in comparison to other pressure-driven processes that are already in use. The membrane used in the FO process a crucial role in water treatment. Cellulose acetate (CA) FO membrane was also frequently used until lately when Thin Film Composite (TFC) membranes took the FO platform showing an enhanced water flux performance along with better salt rejection. The currently used FO membranes are designed by forging a thin film polymerization over the base membrane as a support layer. Optimization studies conducted on the TFC membranes reported that a base layer with a mixture of different morphologies greatly affected the membrane performance. A further insight revealed that though TFC membranes have superior selectivity, the membranes suffer serious internal concentration polarization (ICP), limiting their applications.

The synthesis of chemically stable high-performance FO membranes is still in its early phases of development. The unique advantages offered by CA make it worthwhile to investigate further into its improvement that might have been unnoticed in the past. New studies and advancements in the TFC membranes recommend producing a thin porous support layer for vindicating the effect of ICP, thereby sustaining the top dense selective layer for effective water flux. In search of a better strategy for membrane structure, a new polymer Polycaprolactone (PCL), was found useful. PCL is a biodegradable, biocompatible, and hydrophobic polymer used for various tissue engineering applications. This polymer exhibits great strength and elasticity. To address the issues concerning CA and TFC membranes, we chose to integrate PCL into the CA solution in the present study. The argument for this approach includes (1) incorporating PCL into CA enhanced its mechanical strength drastically. (2) the presence of PCL can be tailored accordingly to the hydrophilicity needs of the support layer for reducing the ICP. (3) No study has reported this combination for preparing FO membranes so far. Also, GO, and CuO NPs are incorporated to increase the antifouling properties. Hence, the potential benefit of low fouling of FO technology is utilized to concentrate dairy wastewater.

2. Materials And Method

2.1. Preparation of nanoparticles

Graphene oxide is prepared using the modified hummers method.(Chandran et al. 2021) In general, 2 g of graphite was washed subsequently with water and ethanol to remove any contamination. Further, the graphite (Sigma) was oxidized using a mixture of H_2SO_4 and H_3PO_4 (9:1, Merck India) with the appropriate amount of $KMnO_4$ (Merck India) and stirred at $60^\circ C$ for 12 h. After the oxidation reaction, the acid slurry was poured into an ice-cold distilled water with 20% H_2O_2 (Merck India). The yellow color appeared confirmed the formation of GO NPs. Further, the yellow-colored suspension was separated and washed using distilled water and ethanol subsequently. The obtained mass after washing was dried in a vacuum oven at $60^\circ C$. Similarly, the CuO was prepared using the sole gel method.(Etefagh et al. 2013) To prepare the precursor, an appropriate amount of copper nitrate (Merck India) was added to a mixture of ethanol and water (1:1). The citric acid (Merck India) was used as the reducing agent and ethylene glycol as the stabilizer. The mixture refluxed for 4 h at $100^\circ C$, and the obtained black powder was calcinated at $600^\circ C$.

2.2. Preparation of FO membrane

The membrane was cast using the Phase-Inversion method. In general, 18 wt. % of cellulose acetate (CA-Sigma) was dissolved in an approximate volume of DMF (Merck India) and acetone (Merck India) (7:3). After 4 h of stirring, the polymer solution was poured over the glass substrate, cast using a doctors' blade, and immersed in a water bath for membrane precipitation. To remove the solvent, the precipitated membrane was washed with DI water. Similarly, CA-PCL blend membrane with 20 wt. % of PCL (Sigma) was fabricated using a similar method. To increase the hydrophilicity of the membrane, an interfacial polymerization (IP) process was used to form a polyamide selective layer on the upper surface of the CA-PCL membrane. The IP reaction was carried out using m-Phenylenediamine (MPD-Sigma) and 1,3,5-Benzenetricarbonyl trichloride (TMC-Sigma). The as-prepared CA-PCL membrane was kept on a glass plate and a 2.0 wt.% MPD aqueous solution was emptied over it. The poured solution was then reserved for 2 minutes to ensure proper saturation, followed by careful removal of excess solution. Following the drying of the aqueous solution, the top surface of the membrane was brought into contact with a 0.1 percent TMC/n-hexane solution for 1 minute. The resultant CA-PCL-TFC (Thin Film Composite) membrane was washed and stored in DI water after drying at room temperature for several minutes and then in the oven for 20 minutes at $70^\circ C$. The Thin Film Nanocomposite (TFN) membrane was prepared using GO and CuO. The MPD solution with varying concentrations of GO wt.% (0, 0.005, 0.01, 0.02) was taken, followed by TMC, the solution for the preparation of TFN. The prepared TFN membranes were named CP0.005, CP0.01, CP0.02, respectively. Similarly, as prepared CuO NPs were incorporated in TFN as depicted in Scheme 1. The fabricated membrane is named CA-PCL membrane as CP, Thin-film composite as CPT, Graphene doped TFC membrane as CPTG, and CuO NPs doped TFC membrane as CPTGC.

2.3. Characterization

Morphology of NPs was investigated using FESEM (Model: Gemini SEM 300 Brand: Carl Zeiss). Surface functionality and crystallinity were studied using FTIR (PerkinElmer Frontier MIR) and WAXD (PANalytical). Structure defects of GO were investigated using Raman spectroscopy (532 nm laser-Horiba Labram HR Evo). The blend formation of CA-PCL was investigated using FTIR (PerkinElmer Frontier MIR) and WAXD (PANalytical). The water contact angle of the membrane was measured (Kyowa, DMS-401, Japan) to gain a piece of knowledge about the hydrophilicity of the membranes. The contact angles conveyed are an average of five measurements done at different locations on the individual membrane samples. Information regarding the mechanical stability, tensile strength, and percentage elongation of the membrane was obtained using Universal Testing Machine (UTM- Shimadzu, AG-X plus, Japan).

2.4. Performance of membrane

The performance evaluation of the membrane is made using a lab-scale FO setup. The FO arrangement involves a cross-flow cell, two pumps for the circulation of individual feed and draws solutions, respectively, and a weighing balance on both sides to measure the amount of water passed. The effective area of the FO cell used is $150 \times 100 \text{ mm}^2$. Spacers were used along with the membrane in the cell to increase the mass flow rate. The membrane performance is evaluated in the AL-FS mode, i.e., where the selective polyamide layer of the membrane faces the feed side. The forward osmosis tests were conducted using DI water as the feed solution and NaCl as the draw solution. The hydraulic pressures of both solutions were the same ($<1.0 \text{ kg/cm}^2$). Volumetric flow rates through the cell were maintained at 40 L/hr. Initial volumes of feed and draw solution were 3 ml and 1 ml, respectively. After the start of the process, the changes in the weight on both sides were recorded. If the feed side, in any case, shows an increase in weight, then the process should be stopped immediately. Otherwise, the process is continued for a certain period until a constant conductivity value of the feed solution is observed. The water flux J_w ($\text{Lm}^{-2}\text{h}^{-1}$) through the membrane is calculated using the Eqn.

$$J_w = \frac{\Delta V}{S \Delta t} \quad (1)$$

Where ΔV (in liters) is the volume change of the feed solution, S (in m^2) is the area of the membrane available for flux (in $\text{Lm}^{-2}\text{h}^{-1}$) and Δt (in hours) is the time interval during which the volume changes by amount ΔV . During each run, volumes of the draw solution were documented before and after testing. A graph was plotted for feed solution representing its conductivity against the concentration. This change in conductivity of the feed solution for each run was used to evaluate the reverse salt flux (J_s , $\text{gm}^{-2}\text{h}^{-1}$, GMH) of the draw solution.

$$J_s = \frac{\Delta C_t * V}{\Delta t * A_m} \quad (7)$$

where ΔC_t (g/L) and V (L) are the changes of salt concentration and feed solution volume, respectively.

3. Result And Discussion

3.1. Nanoparticles

The FTIR spectrum of graphite and GO is depicted in Figure 1a, and the broadening of the peak for GO in a range of $3000\text{-}3500\text{ cm}^{-1}$ is attributed to the stretching vibration of the hydroxy group (O-H). Further, the new peaks observed for GO at 1726 , 1626 , and 1040 cm^{-1} correspond to C-O and C=O bonds of the carboxyl group (Mural et al. 2015). Thus confirm the formation of hydrophilic GO formation. Figure 1b shows the WAXD of prepared GO and CuO. The CuO NPs has shown the characteristic peak at 35.62° and 38.62° , which corresponds to the (002) and (111) plane of CuO NPs. Similarly, graphite has shown the characteristic peak at 26.53° , which corresponds to the (002) plane. Further, the GO NPs have shown a characteristic peak at 10.45° , which attribute to the (001) plane of GO. This shift in WAXD indicates the formation of GO NPs. Further, Figure 1c corresponds to the Raman spectroscopy of Graphite and GO NPs. The peak observed at ~ 1580 , and 1348 cm^{-1} for graphite corresponds to the G and D bands. The G band is attributed to the sp^2 domain of graphite, and the D band corresponds to defects present. It is observed from Figure 1c that the intensity of the D band has increased for GO compared to graphite. This indicates intense oxidation of graphite to GO (Chandran et al. 2021). The surface morphology of GO NPs showed a sheet-like structure with curved edges, as depicted in Figure 1d.

3.2. Membrane characterization

FTIR spectrum as depicted in Figure 2a of pristine CA showed a typical adsorption band at 1732 cm^{-1} , 1368 cm^{-1} , 1229 , and 1034 cm^{-1} which attributed to the C=O, $-\text{CH}_2$, and C-O-C vibration from the acetyl group, respectively (Joshi et al. 2015). Similarly, the FTIR spectrum for pristine PCL shows the characteristic peaks at 2943 and 2864 cm^{-1} attributing the stretching band of the CH_2 group in the polymer chain. Further, for PCL, the asymmetric deformation and symmetric wagging of CH_3 are observed at 1472 and 1366 cm^{-1} bands. The asymmetric and symmetric stretching of C-O-C and C-O is also observed in 1294 , 1163 , and 1108 cm^{-1} bands. The CA-PCL blends showed steady alteration in FT-IR peaks. It is observed from the FTIR spectrum of the CA-PCL blend that the characteristic peak intensity of CA observed at 1229 and 1034 cm^{-1} has gradually increased with the addition of PCL polymer; also, a measured shift in the peaks was observed. Similarly, the characteristic peak observed at 1732 cm^{-1} which corresponds to C=O, has shifted to a lower value. These results indicated that the PCL has effectively interacted with CA to form a blend (He et al. 2021). Similarly, Figure 2c corresponds to the WAXD of CA, PCL, and CA-PCL. It is observed that CA has shown only a prominent peak at 22.5° which attribute to the (200) plane of cellulose II structure (Rouhani and Fashandi 2018). Also, the WAXD of CA indicates lower crystallinity. Similarly, neat PCL has shown the characteristic peak at 21.45° , 22° , and 23.8° , which corresponds to the (110), (111), and (200) plane of orthorhombic crystalline PCL (Sun et al. 2018). Further, the WAXD peak of CA-PCL has shown both peaks at 21.45° , and 22.19° indicate the CA-PCL blend formation (Palacios Hinestroza et al. 2020). Figure 2c corresponds to the measured water contact angle, it can be seen that the contact angle of pristine CA is $\sim 59^\circ$, which characterizes the hydrophilic nature of the polymer. Blending the hydrophobic PCL polymer (contact angle $\sim 83.69^\circ$) into CA, increases its contact angle to 69.7° , thereby decreasing its hydrophilicity by 16.67%. This CA-PCL blended membrane

can be used in the forward osmosis process to address the issue caused by the pristine CA membrane. The tensile strength and percentage elongation of the prepared neat and blend membrane are depicted in Figure 2d. Three samples were tested for each individual CA, PCL, and CA-PCL membrane, and an average value was estimated and compared. From Figure 2d, it is observed that the tensile strength of neat CA is very less as compared to the PCL, demonstrating its lower resistance to deformation. The subsequent blending of PCL in the neat CA to obtain the CA-PCL membrane exhibits a drastic improvement in its tensile strength. Also, the results display that blending has increased the elastic behavior of pristine CA by 395%.

As discussed early, the blending of PCL polymer will enhance the mechanical properties of CA polymer. However, the hydrophilicity of the membrane decreases due to the hydrophobic nature of PCL. To overcome this, a thin film layer of polyamide is coated over the CA-PCL blend. Also, GO and CuO NPs were incorporated inside the thin film to form a thin film nanocomposite. The FTIR spectrum was employed to investigate molecular-level interaction. The FTIR spectrum, as depicted in Figure 3a, corresponds to Phase-Inversion CP, CPT, CPTG, and CPTGC membrane. It is observed from Figure 3a that the Phase-Inversion CP and CPT membrane has comparable peaks. The TFC coated CPT membrane has shown new peaks at 1649, 1611, and 1541 cm^{-1} attributed to the N-H bending aromatic amide ring and C=O stretching in -CO-NH- group, correspondingly. (Park et al. 2017)

Further, the peak located at 1661 cm^{-1} in the CPTG membrane is associated with the C=O stretching of the carbonyl group. This spectrum confirms the formation of new amide bonds between the NH_2 group of MPD and OH groups of the GO. Further, there can be observed the presence of a new peak at 700 cm^{-1} and a subsequent shift in the range of 1200 cm^{-1} which can be attributed to the presence of copper oxide. The relative hydrophilicity of both the polyamide coated and nanoparticles incorporated membrane was measured by the Contact angle shown in Figure 3b. Time-dependent measurement of water contact angle demonstrates that the nanoparticles doped membranes, CPTGC have a better wettability than the CP membrane. The amalgamation of nanoparticles has reduced the contact angle of the CPT membrane from $\sim 67^\circ$ to $\sim 32^\circ$, thus increasing its hydrophilicity by 52%. This increase in the hydrophilic property of the membrane was desired to reduce the antifouling behavior and increase the water flux performance.

3.3. Membrane performance

The prepared CPT membrane was tested for different concentrations of draw solute (0.1, 0.5, and 1 M of NaCl), as shown in Figure 4a. It was observed that as the concentration of draw solute increased, the water flux through the membrane increased, indicating an increase in the driving force for the process. Similarly, an increasing trend was observed for the reverse solute flux. However, 0.5 M NaCl was selected as a draw solution due to high water flux and low reverse solute flux. Further, it was observed that when GO (0.005 wt. %) is incorporated into the thin film matrix, the water flux changed from 4.75 LMH to 21.48 LMH at 0.5 M NaCl as the draw solution. Further, increase in the concentration of GO to 0.01 wt.% lead to an average water flux of 26.635 LMH. This increase in water flux for the membrane could be attributed to an increase in the hydrophilicity of the membrane due to the presence of more hydroxyl groups in graphene oxide. After increasing the graphene oxide concentration beyond 0.01 wt.%, a reduction in water

flux was observed, which might be because of the fact that a higher concentration of GO could have led to its aggregation in the polyamide layers of the membrane.

The optimized GO (0.01wt.%) doped membrane was later assimilated with copper oxide (CuO) on the membrane's same active layers to further increase the hydrophilicity, antifouling, and water flux properties the membrane. The water flux and reverse solute flux performance comparison of the CP-TFC (CPT), CP-TFC-GO (CPTG), and CP-TFC-GO-CuO (CPTGC) membrane against 0.5 M NaCl as draw solute is shown in Figure 5a and b. From the figure displayed it can be concluded that after incorporating both the nanoparticles, the water flux of the membranes increased by the factor of 42. Two batches of fresh laboratory-made dairy wastewater samples were processed by the forward osmosis using a draw solute concentration of 0.5 M NaCl in the AL-FS mode. Firstly, before treating the wastewater, an antifouling study was conducted between the only GO doped and GO-CuO membranes. The better performance membrane could be used for further concentration study. As can be seen from Figure 5c, the water flux declines rapidly with time for both the membranes, but when the membrane was used again after its washing (conducted inline), it was seen that the GO-CuO doped membrane was able to recover its flux, then GO doped membrane. Also, through the literature study, it is found that as the hydrophilicity of the membrane is increased, its capacity to repel the hydrophobic protein increases, thereby increasing the fouling resistance. Therefore, this copper oxide doped membrane was used for concentrating dairy wastewater. The concentration of organic matter in both feed and draw sides were effectively characterized using the TOC (Total Organic Carbon) analyzer. During the FO process conducted for nearly 80 minutes, the feed concentration increased from ~30ppm to ~37ppm, thus, concentrating the solution up to 23% and a concentration factor of 0.903.

4. Conclusion

This study investigates the applicability of the novel (CA+PCL) prepared Forward Osmosis membranes towards concentrating dairy wastewater. After conducting an optimization study for an appropriate blending of two polymers, the membrane was successfully fabricated using Phase-Inversion methods. Blending PCL into CA significantly improved the mechanical strength of the neat CA membranes. This blended membrane was then effectively modified to prepare a TFC membrane, thereby improving the membrane's hydrophilicity and stability, which resulted in the optimal desired low reverse solute flux performance of the phase inversion membrane. Further surface modification of this optimized membrane leading to the efficient formation of Thin Film Nanocomposite (TFN) integrating Graphene oxide and Copper oxide with improved hydrophilicity and antifouling properties was used to concentrate the dairy wastewater, positively showing a concentration factor of 0.91.

Declarations

Authors Contributions

Akash M. Chandran and Dr. Prasanna Kumar S Mural created the concept and interpreted the data. Ekta Tayal conducted the literature survey and carried out the experiments. Graphical design and manuscript

preparation by Akash M. Chandran.

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Availability of data and materials

The information will be provided upon request.

Ethical Approval

Not applicable

Consent to Participate

Not applicable

Consent to Publish

Not applicable

Competing Interests

The authors declare no competing interests.

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Figures

Figure 1

(a) FTIR spectrum graphite and GO, (b) WAXD of CuO, graphite and GO, (c) Raman spectrum of graphite and GO, and (d) FESEM of GO.

Figure 2

(a) FTIR spectrum, (b) WAXD, (c) Water contact angle, and (d) tensile and elongation of CA, PCL, and CA-PCL membrane.

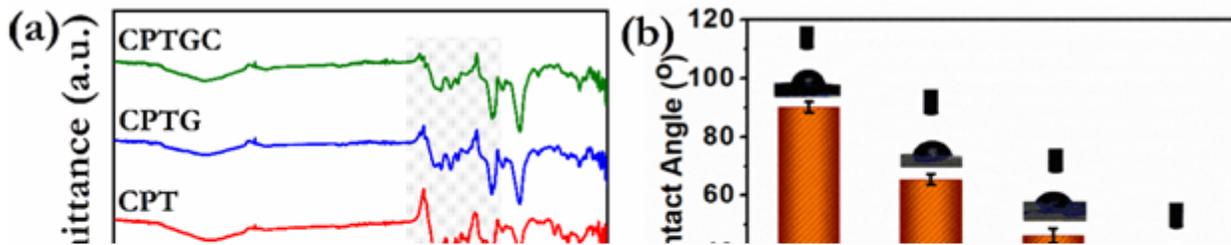


Figure 3

FTIR spectrum and water contact angle of CP, CPT, CPTG, and CPTGC membrane.

Figure 4

(a) Water flux and reverse solute flux of CPT membrane at different concentration of draw solute solution, (b) Water flux, and (b) reserve solute flux of GO incorporated TFC membrane (CPTG).

Figure 5

(a) Water flux, and (b) reverse solute flux performance of CPT, CPTG, and CPTGC membrane against 0.5 M NaCl as draw solute, (c) antifouling study for CPTG and CPTGC membranes, and (d) Rejection performance for dairy wastewater.

Supplementary Files

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- [Scheme1.png](#)