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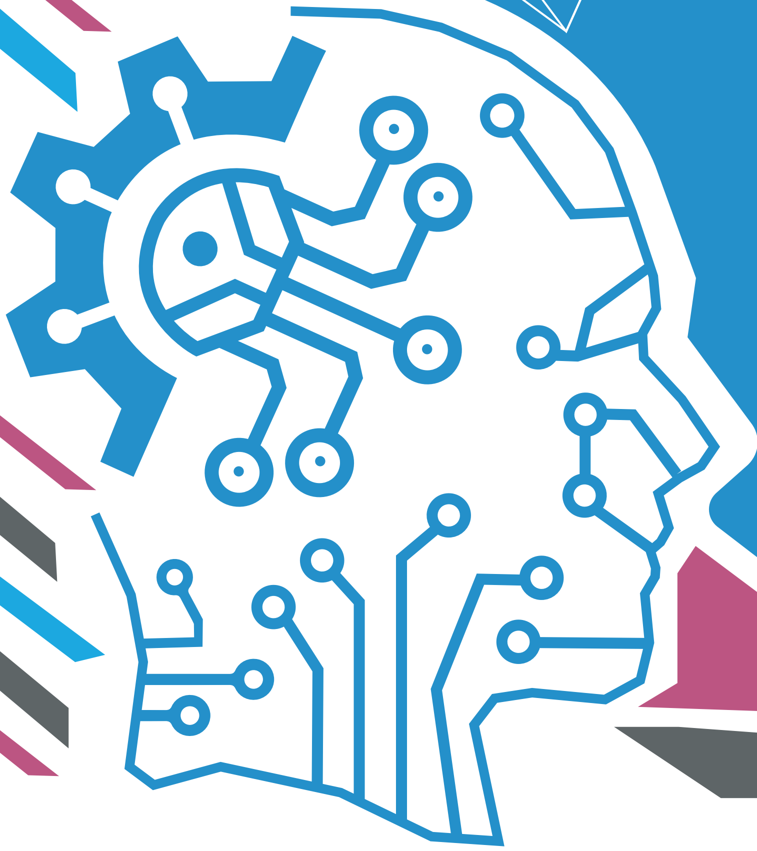
THE 14TH INTERNATIONAL INVENTION, INNOVATION & DESIGN COMPETITION 2025



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EVOSHELL PLA/PCL: EGGSHELL-DERIVED BIOCOMPOSITE MEMBRANES FOR OIL/WATER REMEDIATION

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ABSTRACT

Oil-in-water (O/W) emulsions in domestic and industrial effluents pose a multifaceted challenge due to their stability, multi-droplet size, and low resistance to traditional treatment methods. In this study, composite microfiltration membranes were developed to separate oil droplets from O/W emulsions efficiently. Fine particles from chicken eggshell waste were chemically modified via esterification and directly incorporated into a polylactic acid/polycaprolactone (PLA/PCL) matrix, forming a homogeneous, porous, and asymmetric membrane with a micro/nano-hierarchical structure that enhances surface hydrophobicity. The membranes were fabricated using a combination of plate casting, non-solvent-induced phase separation (NIPS), acid treatment, and ambient drying. The membranes were comprehensively characterised, including chemical structure, morphology, crystallinity, mechanical strength, and surface wettability. The filtration performance was evaluated using an O/W mixture and a surfactant-stabilised O/W emulsion. This work demonstrates a sustainable approach to valorising biowaste eggshells while providing a high-performance membrane for efficient oil/water separation.

Keywords: esterification, polymer composite, thin membrane, hydrophobic, oil/water separation

1. INTRODUCTION

The rapid growth of industrial activities and urbanisation has led to significant environmental challenges, particularly the contamination of water bodies with oil and hydrophobic pollutants. Efficient, eco-friendly oil/water separation methods are critical for ensuring environmental sustainability. In recent years, eggshells, a widely available agricultural waste, have emerged as a promising material for fabricating filtration membranes and adsorbents to address these challenges (Othaman et al., 2024). Conventional wastewater treatment methods, such as chemical coagulation and gravity-based settling, often fail to remove oil droplets and emulsions entirely, and their reliance on chemical additives can result in secondary pollution and inefficiency. As regulatory standards tighten, there is an increasing demand for more effective, sustainable, and economically viable solutions.

Membrane-based separation technologies have gained significant attention due to their high efficiency, minimal chemical usage, and lower energy requirements. Hydrophobic-oleophilic composite membranes are particularly attractive for oil/water separation as they can selectively adsorb and remove oil droplets from water (Li et al., 2024; Zhang et al., 2025; He et al., 2021). Recent advancements in membranes fabricated from various polymers and biomass (Zhong, Tao, & Gong, 2021; Chen et al., 2019), including composite membranes, have demonstrated excellent hydrophobicity and potential for oil recovery.

This study explores the influence of physicochemical properties, including filler loading, surface wettability, and mechanical strength, on the oil/water separation performance of PLA/PCL composite membranes.

2. METHODOLOGY

2.1 Preparation of Composite Membranes

Chicken eggshell waste was processed into microparticles (<63 μm) and chemically modified via stearic acid esterification to enhance hydrophobicity. These modified eggshell particles (MESP) were incorporated in various loadings (5–30%) into a PLA/PCL polymer blend (3:2 ratio) with dichloromethane as solvent. The polymeric solution was cast on a glass plate and underwent the non-solvent-induced phase separation (NIPS) in distilled water. In addition, the PLA/PCL membrane of 30% MESP loading was subjected to surface treatment with glacial acetic acid.

2.2 Characterisation of Composite Membranes

Various characterisation techniques, including FT-IR, FESEM-EDX, AFM, XRD, contact angle analysis, and tensile testing, were utilised to evaluate the membranes' physicochemical, morphological, and mechanical properties. The membranes' performance was assessed for oil/water separation using immiscible and surfactant-stabilised emulsions under gravity-driven and pressurised conditions. Oil removal efficiency and flux were quantified, and membrane reusability was demonstrated through ethanol backwashing.

3. FINDINGS

3.1 Membrane Characterisation

The FTIR analysis revealed the presence of key functional groups, confirming the hydrophobic nature of the composite membranes. Notable peaks included those associated with the ester group (–C=O) and methyl groups ($–CH_3$), indicating the successful incorporation of modified eggshell particles (MESP). XRD results showed increased crystallinity with higher MESP loadings, revealing characteristic diffraction peaks of the pseudo-orthorhombic unit cell of PLLA during a slow solvent-induced precipitation process. Two peaks at about 29° and 39° (2θ) attributed to PLLA, respectively, corresponding to the (2 0 0) and (1 1 3) planes of the orthorhombic crystal structure. The micrograph images in Figure 1 illustrate that the surface of the PLLA/PCL/MESP membranes had micro- and nano-sized craters, improving dispersity and matrix-filler adhesion due to the reduced polarity of MESP and enhanced interaction with the polymer matrix. The contact angle measurements of water contact angle (WCA) and oil contact angle (OCA) indicated a significant increase in hydrophobicity (WCA ~ 132° and OCA ~ 23°) with higher MESP content compared to pristine PLA/PCL membrane (WCA ~ 95° and OCA ~ 42°).

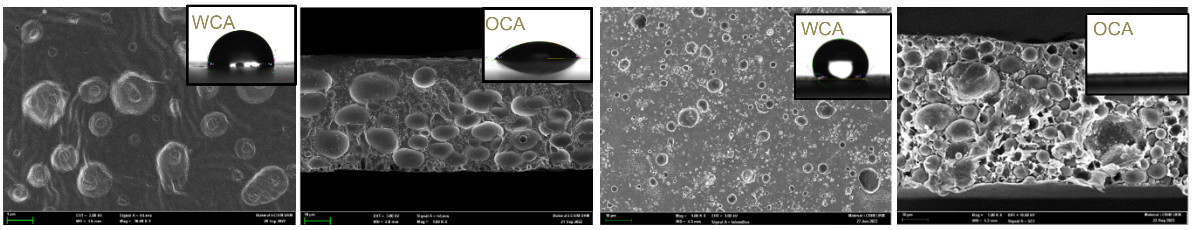

Figure 1 Surface and cross-section morphology micrographs with observed water contact angle (WCA) and oil contact angle (OCA) of (a)(b) pristine PLA/PCL membrane (PPM-0%) in comparison with (c)(d) PLA/PCL membranes of 30% MESP loading (PPM-30%).

3.2 Oil/Water Separation Performance

Oil/water separation experiments demonstrated the effectiveness of PLA/PCL/MESP composite membranes in filtering oil from immiscible mixtures. The PPM-30% membrane exhibited high separation efficiency, with over 80% separating oil from water. The highest efficiency (89.8%) was achieved by PPM-30%, which was treated with glacial acetic acid, making these membranes highly promising for oil recovery and wastewater treatment applications. Table 1 presents the correlation between the physicochemical and mechanical properties of PLA/PCL/MESP membranes and their oil/water separation efficiency

Table 1 Physical, mechanical properties and oil/water separation efficiency of PLA/PCL/MESP membranes

Composite Membrane	Thickness[b] (mm)	Pore diameter$_{ave}$ (μm)	Tensile Strength$_{ave}$ (MPa)	Young Modulus$_{ave}$ (MPa)	Separation Efficiency (%)$_{ave}$

(% MESP Loading)					O/W Mixture	O/W Emulsion
PPM-0%	0.07±0.01	0.145	-	-	9.6	10.9
PPM-10%	0.09±0.01	0.876	7.6172	218.5	12.2	13.8
PPM-15%	0.09±0.01	0.459	7.4377	134.8	14.9	16.4
PPM-20%	0.09±0.01	1.792	7.7213	370.1	23.8	22.7
PPM-25%	0.09±0.01	2.990	7.7123	427.8	43.5	58.4
PPM-30%	0.09±0.01	3.446	7.6046	210.3	75.3	82.2
PPM-30%[a]	0.11±0.01	3.724	7.4335	45.6	78.9	89.8

[a] Surface treatment using glacial acetic acid.
[b] Self-supporting.

4. CONCLUSION

In conclusion, optimising the physicochemical properties of composite membranes is critical for their performance in oil/water separation. Surface treatment with glacial acetic acid for PLA/PCL membranes at higher MESP loading enhances membrane thickness, increases water contact angle, and reduces oil contact angle, improving hydrophobicity and oleophilicity. The surface treatment also increases membrane roughness and pore diameter. While tensile strength and Young's Modulus decrease with higher MESP loading, the separation performance significantly improves.

REFERENCES

Othaman, R., Shahdan, S. N., Abdullah, N. A., Anuar, F. H., & Baharum, A. (2024). Effect of silane treatment on eggshell powder incorporated in poly (L-lactic acid) composite membrane. *Polymer-Plastics Technology and Materials, 63*(15), 2141–2150. https://doi.org/10.1080/25740881.2024.2365299

Li, K., Feng, G., Li, G., Zhang, Z., Xiang, J., Jiao, F., Chen, T., & Zhao, H. (2024). Structurally integrated janus polylactic acid fibrous membranes for oil-water separation. *Journal of Water Process Engineering, 68*, 106525. https://doi.org/10.1016/j.jwpe.2024.106525

Zhang, X., Liu, W., Gao, B., & Zong, W. (2025). A review on hydrophobic materials in oil-water separation. *Microchemical Journal*, 113455. https://doi.org/10.1016/j.microc.2025.113455

He, N., Li, L., Chen, J., Zhang, J., & Liang, C. (2021). Extraordinary Superhydrophobic Polycaprolactone-Based Composite Membrane with an Alternated Micro–Nano Hierarchical Structure as an Eco-friendly Oil/Water Separator. *ACS Applied Materials & Interfaces, 13*(20), 24117–24129. https://doi.org/10.1021/acsami.1c03019

Zhong, L., Tao, H., & Gong, X. (2021). Superhydrophobic Poly(l-lactic acid) Membranes with Fish-Scale Hierarchical Microstructures and Their Potential Application in Oil–Water Separation. *Langmuir, 37*(22), 6765–6775. https://doi.org/10.1021/acs.langmuir.1c00858

Chen, C., Weng, D., Mahmood, A., Chen, S., & Wang, J. (2019). Separation Mechanism and Construction of Surfaces with Special Wettability for Oil/Water Separation. *ACS Applied Materials & Interfaces, 11*(11), 11006–11027. https://doi.org/10.1021/acsami.9b01293

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