

POME Dye Removal Performance during Pervaporation using Lignin Nanoparticle Integrated into Polyamide Thin Film Composite Membranes

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ABSTRACT

Palm oil mill effluent (POME) contains recalcitrant, dye-like chromophoric compounds that are difficult to remove using conventional treatment, leading to persistent colour pollution and limiting safe discharge or reuse. Although polyamide thin-film composite (PA-TFC) membranes offer strong selectivity, their performance in high-strength wastewater is often constrained by the permeability-selectivity trade-off and susceptibility to fouling. To address this gap, this study investigates a bio-based modification strategy by integrating lignosulfonate-derived lignin particles into the polyamide selective layer to enhance pervaporation-based decolorization. Lignin-modified PA-TFC membranes were fabricated via interfacial polymerization with molar ratio MPD: TMC for 0.00925:0.0188 and 0.25% of lignosulfonate. It was then evaluated for pervaporation flux, colour rejection, and chemical stability during POME treatment. Chemical and structural characterization by FTIR indicated changes in surface functional groups consistent with enhanced intermolecular interactions between lignin and the polyamide matrix. The lignin-integrated membrane achieved an improved separation performance, reaching a flux of $17 \pm 0.8 \text{ kg.m}^{-2}.\text{h}^{-1}$ with up to 85% colour rejection, outperforming the pristine polyamide membrane. The performance enhancement is attributed to improved hydrophilicity and a more favourable selective-layer microstructure enabled by the homogeneous distribution of lignin particles and hydrogen-bonding interactions within the polyamide network. Overall, incorporating lignin as a renewable additive provides a promising route to develop higher-performance PA-TFC membranes for pervaporation treatment of POME, supporting more sustainable industrial wastewater management and reducing colour-related environmental impacts.

Keywords: dye removal, nanoparticle lignin, pervaporation, polyamide thin film composite, POME.

1. INTRODUCTION

Palm oil mill effluent (POME) is a highly polluting wastewater generated during crude palm oil processing, particularly from sterilization, clarification, and hydro-cyclone operations [1]. It is characterized by its high organic load, suspended solids, and the presence of recalcitrant and chromophoric dye-like compounds derived from degraded lignocellulosic biomass [2]. These dye compounds not only contribute to the deep brown coloration of POME but also exhibit high stability, resistance to biodegradation, and potential toxicity, making them difficult

to remove using conventional treatment technologies such as anaerobic digestion, sedimentation, or filtration [3]. With increasingly stringent environmental regulations and sustainability standards in the palm oil sector [4], advanced and environmentally benign technologies are needed to reduce colour, chemical oxygen demand (COD), and complex organics to acceptable discharge limits [5].

Membrane-based separation is promising due to its operational simplicity, high efficiency, and scalability [6]. However, pressure-driven processes such as ultrafiltration,

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nanofiltration, and reverse osmosis often face severe fouling, limited selectivity toward specific dyes, and high energy demand, especially for high-organic-load streams such as POME [7,8]. Pervaporation, which combines permeation and evaporation, offers an alternative route for separating organics or dyes from aqueous solutions [9–12]. Polyamide thin-film composite (PA-TFC) membranes are attractive because they provide good chemical stability and strong rejection [4,5,13], yet their dense selective layer can restrict water permeability, limiting throughput for high-strength wastewater such as POME [14,15]. Therefore, tailoring the active-layer structure and surface chemistry of PA-TFC membranes is essential.

Recent membrane development has increasingly incorporated functional nanomaterials to improve hydrophilicity, permeability, and fouling resistance [14]. Prior studies have also explored lignin and lignin-derivative incorporation in polymeric membranes, reporting improved surface hydrophilicity and antifouling behaviour due to abundant hydroxyl/phenolic groups, as well as changes in membrane morphology and transport when lignin-based additives are introduced during fabrication [16–18]. Lignosulfonate-derived lignin nanoparticles are particularly attractive because they are renewable and can form intermolecular interactions with polyamide chains, enabling them to act as structural modifiers during interfacial polymerization and potentially tune active-layer morphology and transport pathways [19,20].

However, a key gap remains: systematic studies are still limited on integrating lignosulfonate-derived lignin nanoparticles directly into the PA-TFC active layer for POME decolorization via pervaporation, and on whether this strategy can increase dye rejection without the common penalty of reduced flux under highly fouling POME conditions. Accordingly, this study develops lignin nanoparticle-integrated PA-TFC membranes via interfacial polymerization and evaluates their pervaporation performance for removing dye-like

compounds from POME, focusing on the effects of lignin incorporation on functional group formation, membrane morphology, flux, and dye rejection.

2. RESEARCH METHODS

2.1. Material and Tools

The materials used in this study included palm oil mill effluent (POME) samples obtained from local CPO industry of Central Kalimantan. Al₂O₃ tubular ceramic substrate (length: 20.0 mm; inner diameter: 5.7 mm; outer diameter: 9.65 mm; pore size: 100-200 nm; external surface area: 17.1 cm²) were imported from Ceramic Oxide Fabricators, Australia. The chemicals used were sodium lignosulfonate (95%, Spectrum Chemical), m-phenylenediamine (MPD, flakes, 99%, Sigma-Aldrich), trimesoyl chloride (TMC, flakes, 98%, Sigma-Aldrich), distilled water, n-hexane (>99,5%; Sigma-Aldrich), and polyaluminum chloride (PAC) coagulant (99%, Sigma-Aldrich).

2.2. POME Pretreatment

Pre-treatment of POME wastewater was carried out to reduce the membrane workload caused by the high content of impurities in the water. The pre-treatment employed a coagulation-sedimentation method using PAC as the coagulant agent follows to research of Elma [21]. A dosage of 2.5 mL of 10% PAC solution was added to 500 mL of POME, followed by stirring for 16 minutes at speeds of 100 rpm and 40 rpm, respectively as shown in Figure 1. The coagulation-flocculation procedure was conducted according to the method described by Allerdings [22].



Figure 1. Coagulation and sedimentation of POME.

2.3. Monomer, Lignin and Alumina Preparation

The membrane fabrication process began with the preparation of the MPD and TMC monomers, the lignin additive, and Al₂O₃ tubular ceramic substrate. MPD was dissolved in deionized water at a molar concentration of 0.00925 M to form the aqueous phase, while TMC was dissolved in n-hexane at a concentration of 0.0188 M to form the organic phase; both solutions were stored in separate containers. The lignin additive was prepared via a wet treatment method in which liginosulfonate powder was mixed with n-hexane and stirred continuously for 3 hours at room temperature, followed by drying through solvent evaporation. The dried lignin particles were then added to the MPD aqueous solution at varied concentrations of 0.25% (w/v). Two beakers containing the MPD-lignin solution and the TMC solution were prepared for application onto the Al₂O₃ substrate.

2.4. Interfacial Polymerization and Inner Coating onto Alumina Tubular Substrate

The synthesis of PA-TFC membranes is primarily achieved through IP, a widely adopted technique for fabricating selective PA layers on porous substrate [23], particularly in reverse osmosis (RO) [24] and nanofiltration (NF) applications [25]. This reaction occurs at the boundary between two immiscible phases: an aqueous phase containing an amine monomer of MPD, and an organic phase containing an acid chloride monomer of TMC [23]. The IP process involves several key steps: (1) a nucleophilic substitution where the amine group attacks the carbonyl carbon of TMC; (2) the formation of an amide bond, accompanied by the release of hydrogen chloride (HCl) as a by-product; (3) continued polymerization as additional MPD molecules react with TMC, producing a cross-linked PA network and releasing HCl and water; (4) the final product is a dense and highly interconnected PA layer

that functions as the active selective barrier of membrane.

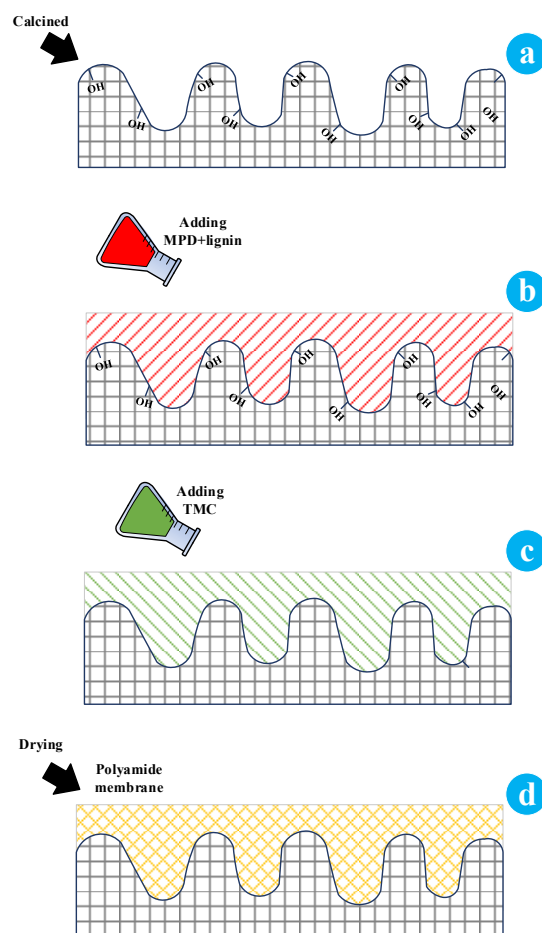


Figure 2. Schematic of Interfacial Polymerization [26].

The formation of lignin-modified PA-TFC membranes was carried out through the stages illustrated in Figure 2. The IP and inner coating processes were conducted simultaneously. The Al₂O₃ tubular substrate to be coated was first prepared by cutting it to a length of 5 cm, followed by washing with ethanol and gently scrubbing to remove chemical impurities. It was then calcined at 800 °C for 8 hours to eliminate physical impurities. The MPD-lignin solution was introduced into the inner channel of the Al₂O₃ tubular substrate and allowed to remain inside the tube for 2 minutes. After that, the solution was drained. The same procedure was then applied to the TMC solution for 1 minute. Then, the membrane was rinsed with n-hexane. Finally, the PA layer formed on the Al₂O₃ tubular substrate was then dried at 80

°C for 1 hour. The completed membrane was cooled for 24 hours and immersed vertically in distilled water until further use.

In this study, the resulting aromatic PA backbone, formed from MPD and TMC, is further functionalized with lignosulfonate via covalent ester bond formation between the hydroxyl (–OH) groups of lignosulfonate and the acid chloride (–COCl) groups of TMC [7,25]. This reaction yields a branched PA structure, where lignosulfonate units are grafted onto the polymer matrix, carrying sulfonate (–SO₃[–]) groups that introduce a negative surface charge [8,27]. The presence of these functional groups enhances membrane hydrophilicity, alters surface charge characteristics, and potentially modifies both morphology and network density, thereby improving the efficiency of membrane in separating organic pollutants [28,29].

Lignin was incorporated at 0.25% (w/v), equivalent to approximately 0.0047 M, which was intentionally kept lower than the MPD concentration. This design follows the common approach in thin-film nanocomposite PA-TFC fabrication, where additives are introduced at relatively low loading because they function as modifiers rather than primary monomers in the interfacial polymerization (IP) reaction [17,18]. Using a lower lignin concentration helps prevent disruption of PA network formation and avoids excessive viscosity or particle aggregation that could introduce defects, while still enabling lignin functional groups to interact with the forming polyamide matrix and tune the active-layer structure

2.5. Pervaporation for Dye Removal of POME

The prepared membrane was utilized in a pervaporation system to treat coagulated POME wastewater. The pervaporation setup is illustrated in Figure 3. The membrane was installed in a dead-end configuration, with one end sealed and the other connected to a cold trap linked to a vacuum pump operating at a pressure of 1.5 kPa. The feed water used

in the process was coagulated POME, which was pumped into the system. The membrane was rinsed with distilled water between sample changes.

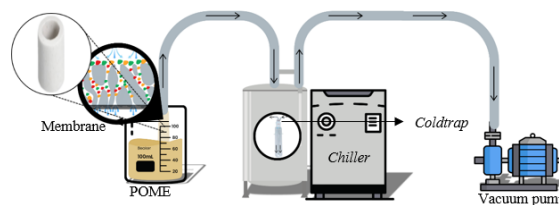


Figure 3. Pervaporation schematic.

The performance of the PA/Lig TFC membrane was evaluated based on the calculation of permeate flux and contaminant rejection in accordance with water quality standards. The flux was determined using the equation $F = m / (A \cdot \Delta t)$, where m is the mass of the permeate (kg), A is the active surface area (m²), and Δt is the duration of the pervaporation process. Meanwhile, the rejection of organic contaminants in POME was calculated using the equation $R = (C_f - C_p) / C_f \times 100\%$, where C_f and C_p represent the contaminant concentrations before and after treatment, respectively.

3. RESULTS AND DISCUSSION

3.1. Membrane Characterization

Figure 4 compares FTIR spectra of lignin, pristine PA, and lignin-modified PA. The pristine PA membrane exhibits the characteristic bands typically reported for IP between MPD and TMC, indicating successful formation of the PA selective layer [23]. After lignin incorporation, the main amide-related features remain present, suggesting that the overall PA framework was preserved and that the additive did not suppress PA formation. This observation is consistent with previous reports on PA-TFC modification, where suitable additives can be introduced without eliminating the fundamental amide signatures, provided that the interfacial polymerization remains dominant [17,18].

Several spectral changes were observed in the lignin-modified PA. First, the intensity in the fingerprint region near 1032 cm^{–1}

increased relative to pristine PA. This region is commonly associated with C-O stretching vibrations and can also include contributions from sulfonate-related vibrations in lignosulfonate-containing materials; therefore, the increase is qualitatively consistent with the presence of lignosulfonate-derived functionalities within the selective layer [17,18]. Second, a slight increase in the broad band around 3400 cm^{-1} suggests additional O-H stretching contributions, which is also consistent with lignin-rich hydroxyl groups [17,18]. While a band around 1604 cm^{-1} may reflect aromatic ring vibrations and/or carbonyl-related contributions, FTIR alone cannot conclusively confirm the formation of new covalent ester bonds between lignin -OH and TMC-COCl. Importantly, no distinct new peaks uniquely attributable to ester formation were observed, and the interpretation should therefore remain conservative.

In this study, the FTIR results are used qualitatively to support (i) the preservation of the PA backbone and (ii) the presence of lignin-associated functional group contributions, rather than definitive proof of new covalent bond formation. This limitation is in line with common practice in membrane studies, where FTIR supports functional group identification but is typically insufficient as a standalone technique to prove new bond formation without complementary evidence.

Overall, the FTIR data indicate that lignin was incorporated into the PA selective layer without disrupting the characteristic PA structure and that additional hydrophilic functionalities likely contribute to the modified surface chemistry. These trends are consistent with literature describing lignin-based additives as hydrophilic modifiers that can improve surface properties relevant to antifouling and aqueous transport, although the property link should be interpreted as supportive rather than conclusive based solely on FTIR.

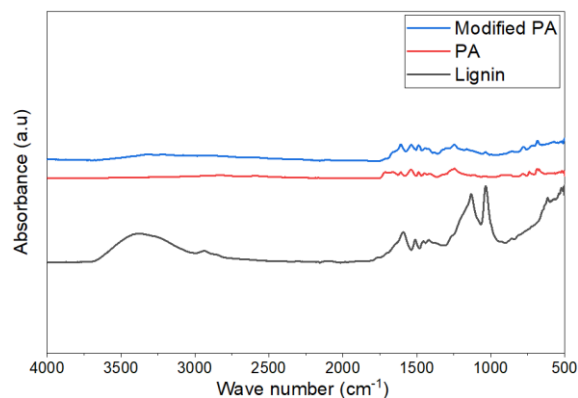


Figure 4. FTIR Spectra of lignin, polyamide and modified polyamide membrane.

Figure 5 compares the surface morphology of pristine PA and lignin-modified PA membranes. The pristine PA surface shows a rough, non-uniform morphology with densely packed nodular/granular features, which is frequently reported for PA selective layers formed under rapid interfacial polymerization [13,15]. Such roughness can increase the number of potential attachment sites for organic foulants and is often correlated with higher fouling propensity in complex wastewaters [9,30,31]. In contrast, the lignin-modified membrane exhibits a more uniform surface with more regularly distributed nodular features. This suggests that lignin presence may have influenced local interfacial polymerization dynamics and selective-layer growth, producing a more organized surface structure. Similar morphology moderation has been observed in thin-film nanocomposite PA membranes when hydrophilic additives are well dispersed and compatible with the forming PA network [14,32–34]. Consequently, the SEM results support that lignin incorporation altered the selective-layer morphology in a direction that may be favorable for permeability and fouling resistance; however, the expected performance benefits should be validated primarily through transport and rejection results.

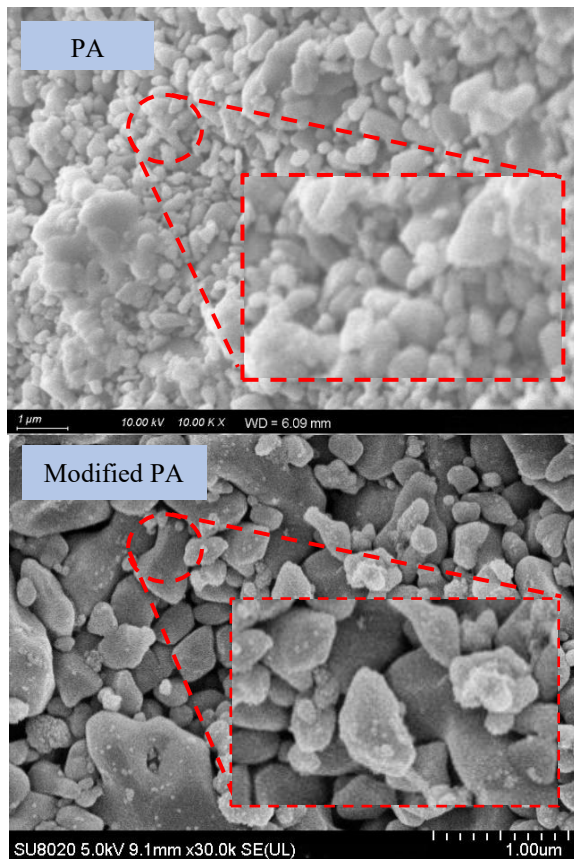


Figure 5. SEM morphology of membrane.

Overall, the observed morphological changes demonstrate that lignin incorporation effectively modifies the PA selective layer, resulting in a more homogeneous surface structure that is expected to improve membrane permeability and antifouling performance compared to the pristine PA membrane.

3.2. Pure Water Fluxes

Figure 6 shows that lignosulfonate modification increased the pure water flux. The pristine PA membrane exhibited a flux of approximately $18 \text{ kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$, which is consistent with the formation of a dense PA selective layer that imposes transport resistance [6–8,15]. With 0.25% (w/v) lignin, the flux increased to $21 \pm \text{kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$.

This improvement is consistent with prior studies where hydrophilic modifiers increase water uptake and facilitate water transport in PA-TFC structures, provided that additive dispersion is adequate and does not create non-selective defects [14,32–34]. In this work, the concurrent SEM observation of a

more uniform surface supports the interpretation that lignin likely acted as a compatible hydrophilic modifier rather than a defect-forming filler at the selected loading. Nevertheless, the flux increase is interpreted conservatively as resulting from combined effects of modified surface chemistry and selective-layer microstructure rather than a single mechanism.

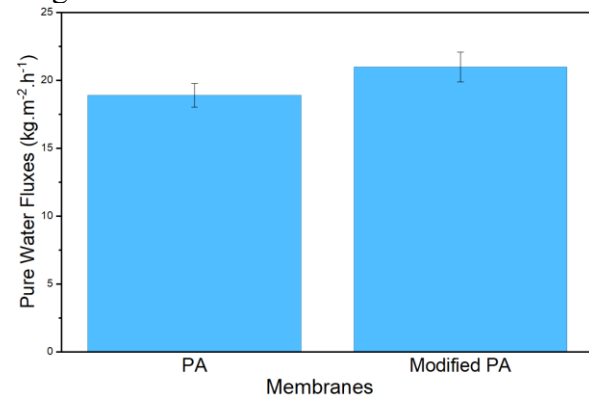


Figure 6. Pure water fluxes.

3.3. Flux and Rejection of POME During Pervaporation

Figure 7 shows POME before and after coagulation-sedimentation using PAC (500 mg/L). The visual change from dark reddish-brown to pale yellow indicates a substantial reduction of suspended matter and a portion of colour-causing compounds, which is consistent with the known role of coagulation in removing colloids and some chromophoric fractions prior to downstream polishing [35].

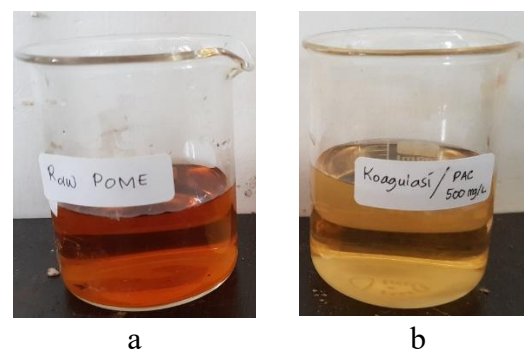


Figure 7. a) raw POME and b) POME after coagulation-sedimentation.

Colour changes before and after pervaporation were quantified using the Platinum-Cobalt (Pt-Co) method, where absorbance at 465 nm was correlated to Pt-

Co concentration as shown in Figure 8. This procedure explicitly defines the rejected component as colour (Pt-Co units), representing dye-like chromophoric compounds in POME, consistent with standard water colour assessment practices [36]. The linear regression ($y = 0.0003x - 0.0034$; $R^2 = 1$) indicates excellent linearity over the calibration range, enabling conversion of absorbance to Pt-Co units for POME samples.

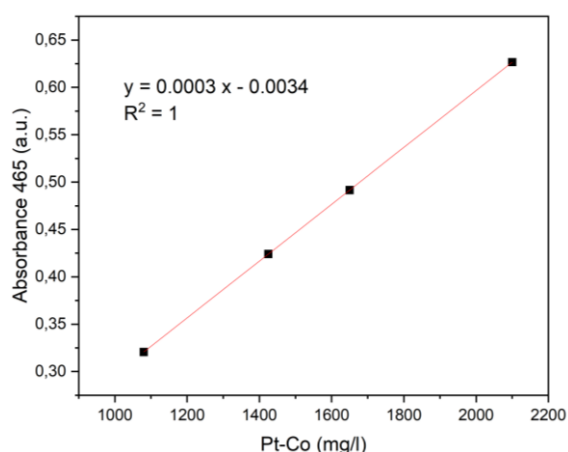


Figure 8. Correlation of POME color with 0-500 mg/l Pt-Co.

Figure 9 presents pervaporation flux and colour rejection. The lignin-modified membrane achieved a higher flux ($17 \pm 0.8 \text{ kg.m}^{-2}.\text{h}^{-1}$) than pristine PA ($13 \pm 1.1 \text{ kg.m}^{-2}.\text{h}^{-1}$), while also providing improved colour separation. This trend is significant because PA selective layers typically face a permeability–selectivity trade-off; improvement in transport often comes at the expense of rejection when modifications introduce non-selective pathways [14,32–34]. The combined increase in flux with maintained/improved colour rejection suggests that, at 0.25% (w/v), lignin acted primarily as a compatible hydrophilic modifier that improved water transport without creating dominant non-selective defects. This interpretation aligns with reports that lignin-derived additives can enhance hydrophilicity and surface functionality, which may support improved interaction with polar contaminants and improved water transport in modified

membranes [17,18]. However, mechanistic statements are presented cautiously: improved rejection is more reasonably attributed to changes in surface chemistry and microstructure rather than definitive chemical bonding inferred solely from FTIR.

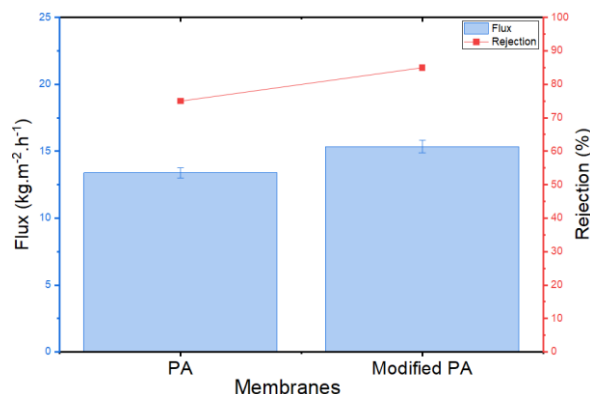


Figure 9. Flux and rejection of POME.

Colour concentration changes and regulatory context. Figure 10 summarizes colour concentration changes (Pt-Co, mg.L^{-1}) across treatment stages. Raw POME showed very high colour ($8811.33 \text{ mg.L}^{-1}$), which decreased after coagulation ($7044.67 \text{ mg.L}^{-1}$), confirming that coagulation alone provided partial colour removal. Subsequent membrane treatment reduced colour markedly to 1578 mg.L^{-1} for PA and further to 844 mg.L^{-1} for PA-lignin/TFC. These data confirm that membrane-based polishing substantially improves decolorization beyond coagulation alone. Nevertheless, the final colour levels remain above stringent discharge targets ($<15 \text{ Pt-Co}$), indicating that additional optimization (e.g., lignin loading range, interfacial polymerization conditions, or staged treatment configurations) is required to meet regulatory limits. Similar constraints are widely recognized in POME treatment, where multiple stages are often needed to achieve strict discharge standards, especially for colour [35,37].

Comparison with previous POME colour removal approaches. Table 1 provides broader context by comparing reported colour removal efficiencies for various methods. While oxidation and electrocoagulation-based methods often

achieve high decolorization (90–100%), they typically require acidic conditions, high chemical/energy input, and/or generate sludge, which can reduce sustainability at scale [35,38–40]. Pressure-driven membranes can provide high-quality permeate but are frequently limited by severe fouling in high-organic-load streams like POME [41]. Within this landscape, the

present pervaporation approach using a lignin-integrated PA-TFC membrane offers a membrane route that avoids high hydraulic pressure while achieving 85% colour rejection and practical flux, supporting its potential as a more environmentally responsive polishing strategy for dye-like contaminants in POME.

Table 1. Comparative study of POME colour removal.

POME Treatment Method	Colour Removal (%)	Advantages	Limitations	Ref.
Fenton Oxidation	90%	Effective degradation of chromophoric compounds; operates at ambient temperature	Requires acidic pH (~3); high chemical consumption	[38]
Coagulation–Flocculation	90%	Simple operation; low energy requirement	High sludge production; often requires post-treatment	[35]
Electrocoagulation (EC)	96-100%	Very high decolorization efficiency; relatively fast process	High energy and electrode consumption; pH-sensitive	[39]
Integrated EC + Peroxidation (ECP)	96.77%	Enhanced removal efficiency and improved biodegradability	Requires handling of residual H ₂ O ₂ ; potential metal sludge formation	[40]
Ultrafiltration	High (exact % not always specified)	Good permeate quality; potential for water reuse	Severe membrane fouling with POME; frequent cleaning required	[41]
Pervaporation using membran PA-lignin/TFC	85% rejection; flux 21 kg.m ⁻² .h ⁻¹	High selectivity without high hydraulic pressure; lignin modification improves hydrophilicity and active layer stability	Long-term fouling and operational stability require further evaluation	This study

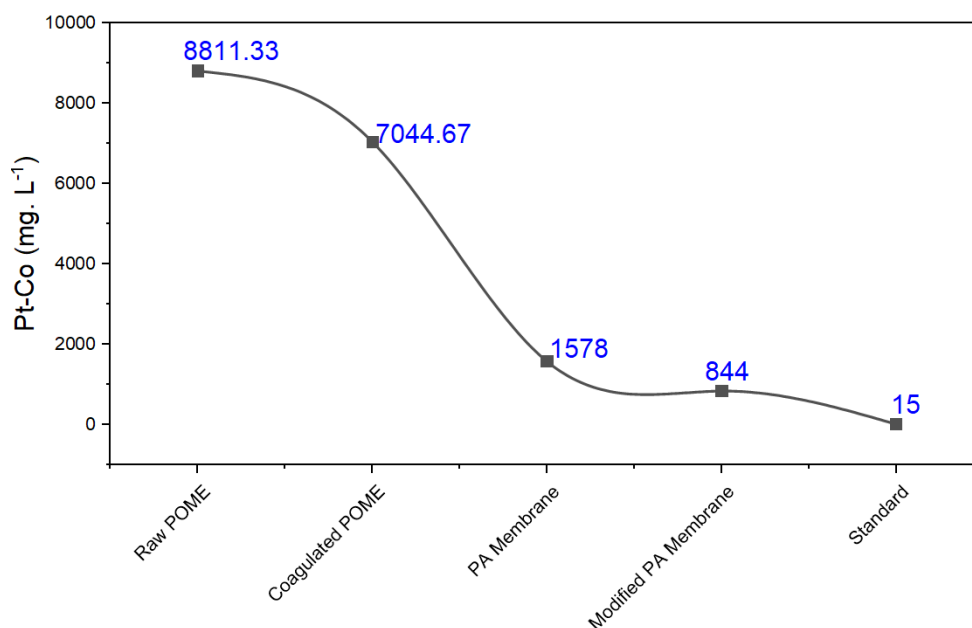


Figure 10. POME color change in correlation with standard Pt-Co solution (mg/L).

4. CONCLUSION

This study developed and evaluated a lignosulfonate-derived lignin-integrated polyamide thin-film composite (PA-lig/TFC) membrane for POME treatment via pervaporation, targeting improved dye/colour removal while maintaining practical transport performance. FTIR and SEM results qualitatively indicated that lignin incorporation did not disrupt the formation of the polyamide selective layer and altered the chemical environment and morphology of the active layer. The FTIR changes (e.g., increased intensity near 1032 cm^{-1} and slight broadening around 3400 cm^{-1}) are consistent with lignin-associated functional group contributions, although FTIR alone is insufficient to confirm formation of new covalent ester bonds. SEM observations showed a more uniform surface morphology for the modified membrane compared with pristine PA, suggesting that lignin acted as a structural modifier during interfacial polymerization. Performance testing demonstrated that lignin modification improved transport and separation outcomes. The pure water flux increased from 18 $\text{kg.m}^{-2}.\text{h}^{-1}$ (PA) to $21 \pm 1.1 \text{ kg.m}^{-2}.\text{h}^{-1}$ (PA-lig/TFC). During POME pervaporation, the modified membrane achieved a flux of $17 \pm$

$0.8 \text{ kg.m}^{-2}.\text{h}^{-1}$ with up to 85% colour rejection, outperforming the pristine PA membrane ($13 \pm 1.1 \text{ kg.m}^{-2}.\text{h}^{-1}$). Colour concentration decreased substantially from 8811.33 mg.L^{-1} in raw POME to 844 mg.L^{-1} after coagulation and PA-lignin/TFC pervaporation, demonstrating effective polishing of dye-like chromophoric contaminants. Although the final colour level remains above strict discharge standards ($<15 \text{ Pt-Co}$), the results indicate that lignin is a promising biodegradable modifier for enhancing PA-TFC membrane performance and sustainability in complex wastewater treatment. The limitations of this study include the need to examine a broader lignin loading range (including concentrations closer to MPD on a molar basis) to distinguish optimal network modification from potential aggregation effects. In addition, the interfacial polymerization was conducted at room temperature without induction (e.g., controlled heating), which may limit reaction kinetics and maximum amide bond formation. Finally, the specific reaction pathway between lignin hydroxyl groups and TMC carbonyl groups cannot be confirmed by FTIR alone due to competing reactions and the absence of distinct new peaks. Future work should include expanded

lignin concentrations, controlled IP conditions, and complementary surface/chemical analyses to strengthen mechanistic understanding and further improve colour removal toward regulatory compliance.

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