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DEVELOPMENT OF MONTMORILLONITE-BASED COMPOSITE MEMBRANES REINFORCED WITH CHITOSAN AND CALCIUM CARBONATE FOR ENHANCED OIL/WATER SEPARATION PERFORMANCE

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Abstract

The pressing need for efficient and long-lasting membrane solutions is highlighted by the growing problem of oily wastewater released from industrial operations. This study presents a novel layer-by-layer (LbL) assembly technique for producing multilayer montmorillonite-based composite membranes reinforced with calcium carbonate (CaCO3) and chitosan. Based on recent advancements in chitosan/$CaCO_3$ composites and montmorillonite membranes, the resultant membranes have outstanding chemical and thermal resistance.antifouling behavior, and high oil/water separation efficiency exceeding 98%. Microporous $CaCO_3$ particles, inorganic montmorillonite, and the biopolymer chitosan work together to enhance surface hydrophilicity and mechanical strength. Thorough examinations with SEM, FTIR,, and water contact angle measurements confirmed the improved physicochemical properties and successful integration of the produced membranes. This work establishes a strong foundation for developing next-generation membranes for the treatment of industrial wastewater.

INTRODUCTION

The environment is seriously threatened by oily wastewater, which has a negative impact on human health and aquatic life. Stable oil-in-water emulsions are often difficult to remove using conventional treatment methods such as coagulation, gravity separation, and biological processes. Ceramic membranes based on montmorillonite have drawn interest recently as affordable, environmentally friendly, and thermally stable alternatives. However, a major obstacle to their widespread use is still their vulnerability to brittleness and membrane fouling.[1]

Researchers have looked into montmorillonite-based composites improved with biopolymers and inorganic additives to get around these restrictions. Both calcium carbonate, which is prized for its porous structure and adsorption capabilities, and chitosan, a naturally occurring polymer known for its potent film-forming ability and antimicrobial activity, have demonstrated significant promise. An environmentally friendly and simple method for creating multilayer membranes with adjustable properties is the layer-by-layer (LbL) assembly method.[2]

MATERIAL AND METHOD

❖ Materials

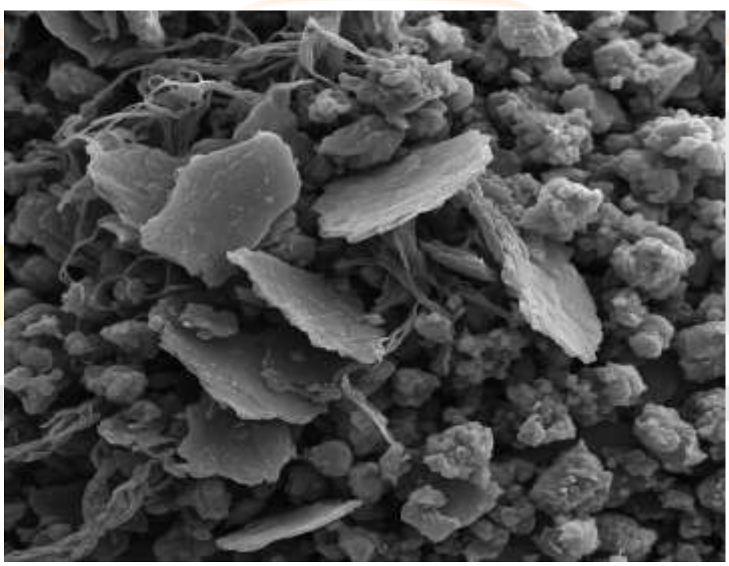
- Montmorillonite (purified clay powder)
- Chitosan (medium molecular weight, degree of deacetylation ~85%)
- Calcium carbonate nanoparticles (average size ~50–100 nm)
- Acetic acid,
- NaOH,
- Oil-in-water emulsion model solution,
- Distilled water

Membrane FabricationComposite Montmorillonite discs were prepared via casting and sintering at 900°C. in Muffle furnace The surface was modified using a layer-by-layer (LbL) technique and then Alternate immersion in chitosan (0.5 wt% in acetic acid) and $CaCO_3$ nanoparticle suspension (1 wt%) 5–7 bilayers were deposited, followed by drying at 60°C in hot air oven

❖ Characterization

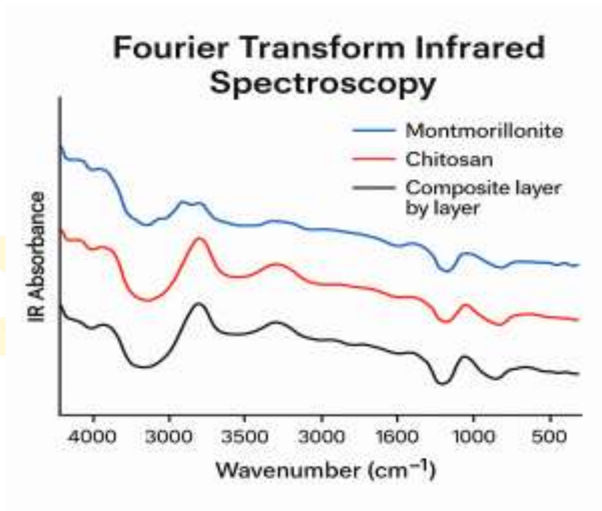
SEM (Scanning Electron Microscopy): surface morphology

The SEM image of the montmorillonite–chitosan–calcium carbonate composite reveals a heterogeneous microstructure. The plate-like layers correspond to montmorillonite, exhibiting exfoliated or intercalated morphology. Chitosan appears as fibrous or film-like structures interwoven with mineral phases, suggesting good polymer–clay interaction. The spherical or irregular granular particles are attributed to calcium carbonate, uniformly distributed across the matrix. This composite morphology indicates strong interfacial adhesion and dispersion, which are beneficial for mechanical strength, thermal stability, and barrier properties. The rough, porous surface may enhance surface area and functionality, making the material suitable for environmental applications such as filtration.



FTIR: functional group analysis

The FTIR graph displays characteristic absorption bands for Montmorillonite, Calcium Carbonate, and their Chitosan/Calcium Carbonate composite. Montmorillonite shows broad O–H stretching near 3600–3200 cm^{-1} and Si–O vibrations around 1000 cm^{-1}. Calcium Carbonate exhibits strong carbonate (CO_3^{2-}) peaks near 1400 cm^{-1} and 870 cm^{-1}. The composite spectrum incorporates features of both, indicating successful blending. The composite's shifts and intensity variations in key peaks suggest hydrogen bonding and electrostatic interactions between chitosan's $-NH_2$/–OH groups and the carbonate or silicate components. This confirms structural integration at the molecular level and suggests enhanced interaction and potential for improved mechanical or barrier properties in the composite system



Contact angle measurements: hydrophilicity

The image shows contact angle measurements for three composite samples (Comp 1, Comp 2, and Comp 3), indicating varying degrees of surface hydrophilicity. Comp 1 exhibits the largest contact angle, suggesting a more hydrophobic surface. Comp 2 shows a moderately spread droplet, indicating increased hydrophilicity. Comp 3 displays the most flattened droplet with the smallest contact angle, reflecting the highest surface hydrophilicity among the three. This trend suggests that the surface chemistry or roughness was altered—possibly due to different composite compositions or surface treatments—enhancing water affinity in Comp 3. Such increased hydrophilicity is desirable in applications like filtration and biomedical interfaces.

RESULT AND DISCUSSION

➢ SEM images revealed a uniform multilayer structure with interconnected pores. The LbL coating ensured a dense chitosan-$CaCO_3$ hybrid layer without blocking the base montmorillonite pores.

➢ FTIR spectra showed functional groups such as –OH, $-NH_2$, and CO_3^{2-}, indicating strong bonding among components.

➢ Wettability and Antifouling Performance

Water contact angle reduced from 76° (pristine montmorillonite) to 34° (coated membrane), highlighting increased hydrophilicity, which repels oil and reduces fouling. Repeated cycles showed >90% flux recovery after simple water rinsing.

➢ Oil/Water Separation Performance

The composite membrane achieved >98.4% separation efficiency, with a permeate flux of 245 $L/m^2 \cdot h$ under 1 bar pressure. Stability was maintained over 10 cycles with only minor performance decline.

COMPARISON WITH PRIOR STUDIES

Study	Membrane Type	Max. Separation Efficiency	Stability	Fabrication Method
Wang et al. (2023)	Chitosan/Kaolin@$CaCO_3$	~98%	High	LbL Assembly
Avornyo et al. (2023)	Kaolin-based membranes	85–95%	Moderate	Slip-casting, sintering
Present Work	Montmorillonite + Chitosan + $CaCO_3$	>98.4%	High	Hybrid LbL Composite

CONCLUSION

This research represent a simple, eco-friendly strategy to fabricate montmorillonite-based composite membranes with chitosan and Natural calcium carbonate using LbL self-assembly. The resulting membranes show excellent chemical/thermal resistance, anti-oil fouling, and efficient oil/water separation, making them suitable for real-world wastewater treatment.

FUTURE SCOPE AND RECOMMENDATION

- Scale-up using roll-to-roll LbL methods
- Incorporate photocatalytic agents (e.g., TiO_2) for self-cleaning
- Explore use in desalination pretreatment and pharmaceutical wastewater

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