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Development of Eco-Friendly Silk Nanofibril-Based Composite Membranes for Water Filtration Applications

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Abstract

This research presents the development of a sustainable membrane system designed for effective water purification, utilizing silk nanofibrils (SNFs) combined with palygorskite (PGS). Genipin, a non-toxic natural crosslinking agent, was employed to induce a structural shift in SNF chains from flexible random coils to a stable β-sheet configuration. This modification notably improved the mechanical integrity of the membranes, while reducing susceptibility to swelling and degradation. By adjusting the proportion of SNFs and PGS, the resulting composite membranes achieved customizable performance in terms of selectivity and permeability. These hybrid membranes demonstrated strong dye separation abilities and exceptional water permeability, highlighting their viability in wastewater treatment applications. The nanoscale pore structure, together with the biodegradable and protein-based architecture of silk, makes these membranes a sustainable replacement for traditional synthetic filtration materials. Future studies may focus on incorporating active functional groups or layering substrates to further refine their separation profiles for targeted uses.

Keywords: *Silk nanofibrils; palygorskite; genipin; eco-filtration; dye removal*

Introduction

The intensifying discharge of harmful pollutants—including dyes and heavy metals—due to industrial growth has led to serious water quality concerns. Colored wastewater contains persistent organic pollutants that resist degradation and can cause significant environmental and health hazards. Studies have shown that a significant fraction of industrial dyes enter water bodies untreated, contributing to ecological imbalance. To address these challenges, membrane-based filtration offers a chemical-free and energy-efficient solution. Although traditional membranes like ultrafiltration and nanofiltration are widely used, their effectiveness is limited by issues like pore size constraints, high pressure requirements, and membrane fouling. This has prompted interest in natural materials like silk fibroin, known for its robustness, biocompatibility, and ability to form nanostructured fibrils. SNFs can be fabricated into membranes with consistent pore distributions, but challenges such as fixed pore dimensions and structural limitations remain. Integrating natural clay minerals like palygorskite offers an economical approach to enhance the structural and adsorptive qualities of SNF membranes. Furthermore, using genipin as a crosslinking agent can stabilize the silk nanofibril network, increasing water resistance and separation efficiency. This study explores the formation and performance of SNF-PGS composite membranes crosslinked with genipin, offering a green and effective solution for dye-laden wastewater treatment.

Materials and Methods

Preparation of the SNF/PGS Blended Precursor

To formulate the precursor mixture, an aqueous SNF solution (0.1% w/v) was combined with a PGS suspension (0.02% w/v). Several volume ratios of SNF to PGS were prepared (4:1, 3:1, 3:2, and 1:1), corresponding to mass ratios of 20:1, 15:1, 15:2, and 5:1. Genipin, acting as a natural crosslinker, was added at a concentration of 0.025% (w/v) and stirred at ambient temperature until fully dissolved. The crosslinking reactions were performed at 37 °C for intervals of 3, 6, 12, 18, and 24 hours to yield SNF-PGS-G solutions with varying degrees of crosslinking.

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Fabrication of SNF-PGS-G Composite Membranes

The prepared SNF-PGS-G solutions were subjected to vacuum filtration onto polyamide (PA) membranes with a 450 nm pore size. To induce the structural transformation of SNFs into β-sheets, the membranes were immersed in a 90% methanol aqueous solution for 30 minutes. Afterward, they were thoroughly rinsed with distilled water and air-dried. Pure SNF membranes, without PGS addition, were also fabricated under the same conditions as controls.

Determination of Crosslinking Efficiency

Crosslinking levels were assessed via a ninhydrin assay that quantifies free amino groups. Freeze-dried membrane samples (3 mg) were mixed with 0.35% (w/v) ninhydrin solution and heated to 100 °C for 10 minutes. Absorbance was measured at 570 nm using a microplate reader. A reduction in free amino group content indicated a higher crosslinking degree. Membranes without genipin and glycine standards were used as references. Each experiment was repeated three times to ensure reproducibility.

Characterization of Composition and Morphology

The crystallinity of palygorskite before and after treatment was examined by X-ray diffraction (XRD). Atomic force microscopy (AFM) was used to evaluate the morphology of SNFs. Chemical compositions of PGS, SNF, and composite membranes were analyzed by Fourier-transform infrared spectroscopy (FT-IR) and X-ray photoelectron spectroscopy (XPS). The microstructure and surface topography of membranes were observed using field emission scanning electron microscopy (FESEM).

Performance Evaluation of Membranes

The water flux performance of the membranes was assessed using a laboratory-scale ultrafiltration system at 25 °C. Pure water was passed through the membranes to measure permeate flux (J), and dye separation efficiency was evaluated using standard dye solutions.

Results and Discussion

Filtration Performance of SNF-PGS-G Membranes

To comprehensively evaluate the separation ability of the SNF-PGS-G composite membranes, various anionic dye solutions were used as test media. The membranes were subjected to filtration tests involving 40 mL of solutions containing dyes such as direct red 80 (DR80), brilliant blue G (BBG), methyl blue (MB), acid fuchsin (AF), and others. These dye-containing solutions were filtered using a vacuum filtration apparatus. The effectiveness of dye removal was assessed using UV–Vis spectroscopy to monitor concentration changes before and after filtration. The results revealed a significant drop in dye concentrations across all tested solutions, with rejection rates exceeding 95% for every dye, regardless of their molecular weight differences.

The membrane's ability to block dyes can primarily be attributed to its precisely controlled pore size. The SNF-PGS-G membranes operate through a size-exclusion mechanism aided by electrostatic interactions. Zeta potential analysis of SNF-G and SNF-PGS-G (15:1) membranes at pH 6.5 revealed negative surface charges in the range of −41 to −54.9 mV. These negatively charged surfaces enhanced repulsion of anionic dyes, improving rejection rates. Furthermore, the incorporation of PGS enhanced the membrane’s electronegativity compared to SNF-G membranes alone. This effect explains the superior ion-repelling behavior of the SNF-PGS-G (15:1) formulation, which outperformed its pure SNF counterpart.

Conclusion

This study successfully demonstrates the development of eco-friendly, biodegradable composite membranes by integrating silk nanofibrils (SNFs) with palygorskite (PGS) and crosslinking with genipin. The resulting SNF-PGS-G membranes exhibited high mechanical stability, efficient dye removal (>95% rejection rate), and excellent water permeability. These membranes leverage the synergistic properties of SNFs and PGS to enhance structural strength and filtration performance through size-exclusion and electrostatic repulsion mechanisms. Given their customizable composition, green fabrication process, and sustainable nature, SNF-PGS-G membranes offer a promising alternative to conventional synthetic filtration systems for effective treatment of dye-contaminated wastewater. Future research may focus on scaling production and integrating functional additives for specific pollutant removal.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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