Applied Chemical Engineering (2026) Volume 9 Issue 2 ISSN: 2578-2010 (O)
doi: 10.59429/ace.v9i2.5923

ORIGINAL RESEARCH ARTICLE

Sustainable development of recycled ultrafiltration membranes with bio-based antifouling coatings

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ABSTRACT

Fouling in Reverse Osmosis (RO) membranes reduces filtration efficiency and shortens membrane lifespan, generating significant end-of-life (EoL) waste. This study developed recycled ultrafiltration (UF) membranes from EoL RO membranes with natural chitosan-based antifouling coatings containing garlic, lime, and green tea extracts. The process involved RO cleaning, sodium hypochlorite (NaOCl) treatment for UF conversion, coating application, and performance evaluation via permeability, salt and humic acid rejection, and water contact angle tests. The chitosan-garlic (0.4 g, 30s immersion) coating achieved the best antifouling performance, showing the lowest contact angle (40.7°) and high rejection rates for humic acid and salts. Increasing coating duration from 30 s to 3 h improved salt rejection (e.g., Na_2SO_4 rejection increased from ~18% to ~34.9%) but reduced permeability due to pore narrowing. This approach improves membrane antifouling capability while supporting SDGs 3,6,9, and 12 by promoting sustainable water treatment and waste reduction.

Keywords: End-of-Life RO Membrane; Recycled Ultrafiltration; Antifouling Coating; SDG 6; SDG 12

ARTICLE INFO

Received: 27 February 2026
Accepted: 28 April 2026
Available online: 18 June 2026

1. Introduction

The use of reverse osmosis (RO) membranes is one of the primary approaches in water treatment systems, both for domestic and industrial purposes. However, the limited lifespan of RO membranes leads to the generation of end-of-life (EoL) membrane waste due to the accumulation of irreversible fouling, membrane damage, and various other causes[1]. According to Lejarazu et al.[2], it is predicted that by 2025, more than 2,000,000 tons of EoL RO membrane waste will be generated.

Ultrafiltration (UF) membranes are a pressure-driven separation process in which the separation of components in a liquid depends on the size and structure of the dissolved substances. UF membranes are typically characterized by a molecular weight cut-off (MWCO) in the range of approximately 1–100 kDa, although equivalent pore sizes are often reported between 1 and 100 nm. These membranes are designed to retain colloids, macromolecules, and microorganisms while allowing water and low-molecular-weight solutes to pass through [3]. UF systems generally operate at relatively low transmembrane pressures (0.1–5 bar), resulting in high permeability and low energy consumption. Due to these properties, UF membranes are widely applied in industrial wastewater treatment, surface water purification, food processing, and pretreatment for reverse osmosis systems [4]. In

recent years, the reuse of RO membranes that have reached their end of life (EoL) as UF membranes has received increasing attention. This approach is considered both economical and environmentally friendly, as it extends the service life of the membranes and reduces the amount of solid waste generated.

EoL membrane waste presents its own challenges, particularly concerning fouling issues that can reduce membrane performance efficiency. The recycling of RO membranes into UF membranes has been widely investigated. Sodium hypochlorite (NaOCl)-based oxidation is a commonly reported approach to remove the polyamide layer and convert RO membranes into UF membranes; however, the resulting membranes often exhibit limited fouling resistance, which restricts their long-term applicability[5]. Additionally, another study by Putri et al[6] reported that recycled membranes still possess high hydrophobicity, making them less capable of absorbing water and more susceptible to fouling. In this context, various innovations have been developed, one of which is the application of natural antifouling coatings derived from materials such as chitosan, garlic, lime, and green tea, all of which are known for their anti-adhesive and antibacterial properties. Chitosan contains amino groups that can inhibit the growth of microorganisms and biofilm formation on membrane surfaces[7]. Allicin compounds in garlic exhibit strong antimicrobial activity against fouling-causing bacteria[8]. Lime contains citric acid and flavonoids that also have antibacterial properties and can reduce surface fouling[9]. Moreover, polyphenols found in green tea extract have been proven effective in preventing biofilm formation and enhancing the antifouling properties of membrane surfaces[10].

Although several studies have investigated antifouling coatings and membrane recycling technologies separately, there remains a limited number of studies that integratively combine both approaches. Therefore, this research aims to develop UF membranes recycled from EoL RO membrane waste, coated with natural antifouling agents based on chitosan, garlic, greentea, and lime, in order to enhance fouling resistance and support sustainable membrane waste management.

2. Materials and methods

2.1. Materials and Tools

The tools used in this study included glassware (Pyrex), an analytical balance (OHAUS PX224/E Analytical Balance), electrical conductivity (RIO 6002 Conductivity), a cross-flow operating unit, ball filler (24.25A), glass funnel (Herma), watch glass, measuring flask 25 mL and 100 mL (Herma), measuring pipette 25 mL (Iwaki), measuring pipette 5 mL (Iwaki), spatula, and a magnetic stirrer. The materials used in this study included an RO membrane module, distilled water, Hydrochloric acid (HCL), Sodium hydroxide (NaOH), Sodium Dodecyl Sulfate (SDS), Sodium Hypochlorite (NaOCl), chitosan, acetic acid (1 wt%), green tea powder, lime extract, garlic extract, glutaraldehyde (0.25%), Sodium chloride (NaCl), Magnesium sulfate ($MgSO_4$), Sodium sulfate (Na_2SO_4), and Acetic acid (CH_3COOH) that obtained from Merck and local distributor.

2.2. Membrane Recycling Process

2.2.1. RO Cleaning Process

The RO cleaning process uses a solution of HCl, SDS, and NaOH. The membrane is soaked in the HCl solution for 16 hours. This soaking in HCl removes metal ions and inorganic deposits. It is then rinsed with distilled water to remove any remaining HCl solution. This is followed by soaking in a NaOH + SDS solution for 8 hours, then drying with tissue paper, and soaking again for 8 hours. The soaking in the NaOH + SDS solution removes oil, grease, and organic contaminants. The membrane is then dried with tissue paper to remove any residual cleaning solution. The cleaned membrane is ready for further conversion processes or filtration characteristic testing.

2.2.2. Conversion of RO to UF

The membrane recycling technique involves soaking the cleaned membrane in a NaOCl 0.6 wt% solution as an oxidant to remove the solid aromatic polyamide layer, which can convert it into a porous membrane. The soaking process was carried out for 5 days.

2.3. Coating Process

The coating process began by preparing a chitosan solution, where 100 mg of chitosan was dissolved in 100 mL of 1 wt% acetic acid solution. This solution was stirred at 400 rpm for 3 hours at room temperature until homogeneous. Green tea extract was then prepared by extracting 2 g of green tea powder in 100 mL distilled water at 80°C for 1 *hour*, followed by filtration using a 0.45 µm membrane filter. The green tea extract was mixed into the chitosan solution and stirred for 1 hour. A 0.25% (w/v) glutaraldehyde solution was also prepared as a crosslinking agent for the chitosan-based coating. For coating, a membrane was pre-wetted by soaking in distilled water for 24 hours to activate the membrane surface. The activated membrane was immersed in the chitosan solution mixed with green tea extract for coating at varying durations of 0, 1, 3, and 5 hours, with gentle agitation to ensure uniform coating.

Lime extract was prepared by squeezing the juice of fresh limes (40 mL) and mixing it with 100 mL of 1% acetic acid solution. The mixture was stirred using a magnetic stirrer until homogeneous. After homogenization, 1 g of chitosan was added to the solution and stirred for an additional hour. The PES UF membrane was dip-coated into the prepared chitosan-lime extract solution for 5 minutes and 10 minutes, with controlled and stable dipping speed to ensure even coating on the membrane surface. The membranes were immersed slowly into the solution and withdrawn at the same speed, allowing a thin, uniform coating to form.

Garlic extract was prepared by dispersing 0.2 g, 0.4 g, and 0.6 g of garlic powder in 100 mL of 1% acetic acid solution and stirring for 1 hour. After achieving homogenization, 0.25 g of chitosan was added gradually, followed by 0.0625 g of glycerin (25% w/w chitosan), and stirred for 8–10 hours. The coating process was initiated by dip-coating. The solution, which had been mixed with chitosan and the extract, was transferred to a larger container so that when the membrane is coated, the membrane is completely covered and there are no gaps in the membrane that are not coated by the coating. Then the membrane is dipped into the coating solution that is already in a large container, the immersion of the membrane is carried out on both sides of the membrane, namely the front and back. After the membrane is completely immersed in the coating solution, it is left to immerse for 1 hour to allow adequate interaction between the membrane surface and the coating, ensuring uniform coating application. This duration allows the membrane to achieve optimal coating characteristics, which can be observed in the subsequent test results.

2.4. Characterization

2.4.1. Permeability and Rejection

The dissolved membrane was then tested using a dead-end ultrafiltration cell system. The membrane was placed at the base of the module, and the water reservoir was kept full of distilled water. The water was fed into the module at a constant pressure of 2 bar, controlled by an adjustable pressure regulator, to ensure a steady flow rate through the membrane. The sample water then flowed through the membrane, with the permeate collected in a container every 5 minutes for 20 minutes. The volume of the permeate was measured to calculate the membrane flux (membrane permeability) and rejection performance.

2.4.2. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared (FTIR) Spectroscopy is a spectroscopic technique used to identify functional groups in a chemical compound based on the absorption of infrared radiation. This technique allows the observation of changes in chemical structure that occur due to treatment or modification of a material. FTIR

spectrum data was obtained using a PerkinElmer Spectrum IR instrument with a wavelength range of 4000 cm^{-1} to 450 cm^{-1}.

2.4.3. Water Contact Angle (WCA)

The Water Contact Angle (WCA) was measured using the Optical Contact Angle (OCA) technique to characterize the hydrophilicity or hydrophobicity of the membrane surface. The measurements were conducted using a OCA 25, and the contact angle was determined by placing a water droplet on the membrane surface. The angle between the surface of the membrane and the tangent to the water droplet was measured. The contact angle was measured at two different points on each membrane to ensure consistency. All measurements were repeated three times to obtain average data for each condition.

3. Results and Discussion

3.1. Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR test aims to analyze the chemical structure to confirm the modification of the coating on the membrane surface[11]. The FTIR spectra shown in **Figure 1** correspond to the test results under varying experimental conditions.

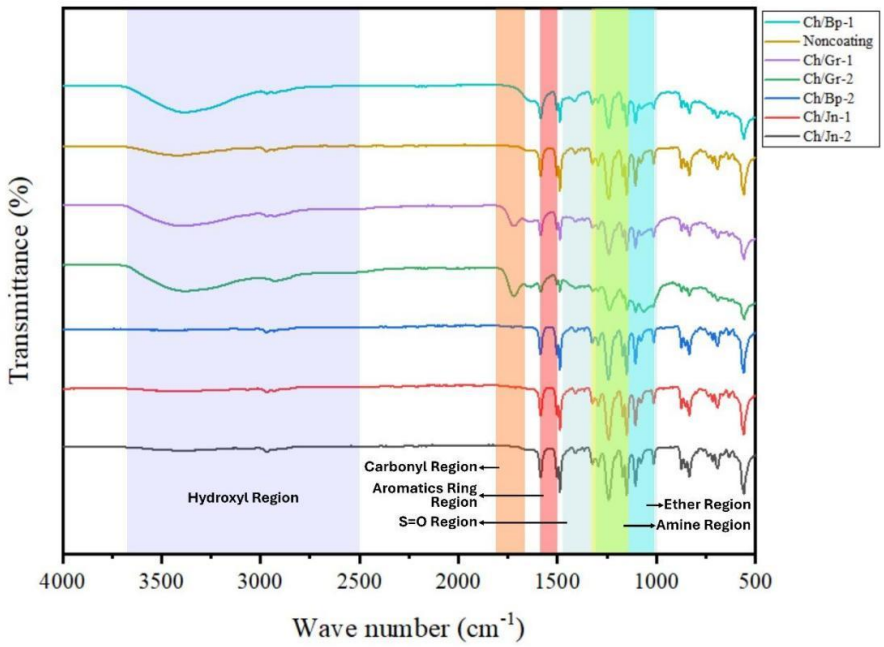

Figure 1. Fourier Transform Infrared Spectroscopy Graph.

The FTIR spectra show visible functional group differences between uncoated membranes and membranes coated with chitosan (Ch) and several natural ingredients, namely garlic (Bp), green tea (Gr), and lime (Jn). These samples are coded as follows: Ch/Gr-1 and Ch/Gr-2 are chitosan and green tea coatings with extraction times of 1 hour and 3 hours, respectively; Ch/Jn-1 and Ch/Jn-2 are chitosan and lime coatings with immersion times of 5 and 10 minutes; Ch/Bp-1 and Ch/Bp-2 are chitosan coatings with 0.2 g and 0.4 g of garlic, respectively.

The membrane coated with chitosan and green tea for 1 hour exhibited absorption bands at ~2965 cm^{-1}, attributed to C–H stretching vibrations of polysaccharide structures, and at ~1584 cm^{-1} corresponding to N–H bending of primary amine groups in chitosan. For the membrane coated for 3 hours, a slight shift in the C–H stretching band to ~2970 cm^{-1} was observed, while the N–H bending peak remained at ~1584 cm^{-1}. This minor shift suggests increased interaction between the coating components and the membrane surface, likely due to prolonged coating time leading to a more compact and homogeneous coating layer [11]. Chitosan contains amine

(-NH_2) and hydroxyl (-OH) groups, which are polar[12]. Therefore, the chitosan and green tea coatings, after 1 and 3 hours of application, resulted in a more uniform and chemically stable layer, as evidenced by the changes in FTIR spectra and enhanced surface interactions. The longer coating time allowed for greater crosslinking between chitosan molecules and green tea polyphenols, leading to a more robust membrane surface with improved antifouling properties.

After the coating process, the transmittance of several functional groups changed in the chitosan and lime membranes. The appearance of a band at 1720 cm^{-1}, characteristic of the carbonyl group (C=O), was only detected in the coated sample, suggesting the possible use of compounds with ester or ketone groups in the coating process. The band around 3390 cm^{-1} decreased in intensity, indicating a reduction in the O-H group due to masking by the coating or a chemical reaction. The increase in transmittance in the 2920-2970 cm^{-1} band associated with C-H stretching indicates the contribution of organic compounds from the coating material. Furthermore, the decrease in transmittance in the C-O and N-H bands indicates the involvement of these groups in the interaction with the coating material. This is consistent with research conducted by Daoud et al.[13], which showed that the formation of new carbonyl compounds due to oxidation can be observed through an increase in the intensity of the band around 1700-1726 cm^{-1}[13]. Thus, the results of the FTIR spectrum analysis indicate changes in the chemical structure of the membrane surface after the coating process, characterized by the appearance of a carbonyl band (C=O) and changes in the intensity of several other bands. As the coating time increases, the intensity of peaks associated with polar groups tends to decrease, indicating the effectiveness of the surface coating.

The chitosan and garlic membrane coatings show the presence of bioactive compounds from garlic, such as allicin and other organic sulfur compounds. In the membrane with garlic extract coating (0.2 and 0.4 g) there is an increase in peak intensity (O-H) around 3400-3500 cm^{-1}, appearing wider and more intense than the non-coating membrane. This comes from phenolic compounds and water molecules derived from garlic extract which increase the hydrophilicity of the membrane surface. And plays an important role in increasing hydrophilicity in reducing fouling and increasing membrane permeability[8]. At the peak of 1000-1200 cm^{-1} which is a characteristic of the (S=O) group of organosulfur compounds such as allicin, it is seen in the membrane with garlic extract coating more intensely than the non-coating membrane. This indicates that the active compound of garlic is successfully seen on the membrane surface, this compound has strong antibacterial properties so it can inhibit bacterial growth on fouling membranes[14]. Then at 1500-1650 cm^{-1} functional groups related to aromatic ring strain (C=C). This was found in the garlic extract-coated membrane, which was more intense than the non-coated membrane. This compound is another phenolic compound in garlic that contributes to antibacterial activation. Membranes with the added coating exhibited significant chemical changes on the membrane surface. These changes demonstrate the success of this study, which used the addition of garlic extract coating to the membrane, increasing the membrane's antibacterial, antibiofouling, and hydrophilic properties.

3.2. Filtration Performance

3.2.1. Rejection of Salt

Based on test data, the coating process on the ultrafiltration membrane significantly impacted its salt solution rejection. Rejection values for all three types of salt (NaCl, $MgSO_4$, Na_2SO_4) increased with increasing coating duration and the amount of additives. This indicates that the coating process can reduce the membrane's pore size and change its surface properties to be more selective to dissolved ions. In NaCl solution, membrane rejection increased from approximately 2% (without coating) to over 9% after 1 hour of coating and reached 23% for the membrane with the addition of 0.4 g of garlic extract. This phenomenon is caused by the narrowing of the pores caused by the polymer coating and the positive charge from the protonation of the amine group ($-NH_3^+$) in chitosan, which exerts a repulsive force on Cl^- ions. The thicker the coating layer, the higher the

rejection rate for monovalent ions such as Cl^-. For the $MgSO_4$ solution, the rejection value showed an increasing trend after 3 hours of coating, reaching 16%. However, after 1 hour of coating, rejection decreased slightly (4.9%) compared to the uncoated condition (7.3%). This is likely due to the interaction between the positively charged Mg^{2+} ions and the negatively charged membrane at the beginning of the coating process, thereby reducing the repulsive force against SO_4^{2-} ions. With longer coating durations, the polymer layer formed becomes denser and is able to block dissolved ions from entering through the membrane pores, thus increasing rejection. The most prominent phenomenon was seen in the Na_2SO_4 solution. Rejection increased from 2.4% (without coating) to 18% (1 hour of coating) and reached 34.9% after 3 hours of coating. SO_4^{2-} ions, which have a double charge and a larger hydration radius, experience stronger electrostatic repulsion, making it more difficult for them to pass through the membrane. Thus, increasing the coating thickness strengthens the rejection mechanism through both pore narrowing (size exclusion) and charge interaction (Donnan exclusion). Overall, these data indicate that coating duration and coating amount are directly proportional to salt rejection, although this results in decreased permeability due to smaller pores. This phenomenon aligns with the basic principle of nanofiltration membranes, where a trade-off occurs between permeability and selectivity[15].

3.2.2. Humic acid Rejection

Based on the test results, the concentration of humic acid in the permeate decreased significantly after the membrane was coated. On the uncoated membrane, the humic acid concentration was recorded at 20 ppm with an absorbance of 0.821. After coating for 1 hour, the concentration dropped to 16 ppm (absorbance of 0.652), while after 3 hours of coating, the humic acid concentration further decreased to 13 ppm (absorbance of 0.533). This decrease indicates that the coating process is able to improve the membrane's ability to retain complex organic molecules such as humic acid. This phenomenon is caused by changes in the properties of the membrane surface due to the chitosan coating which is rich in amino groups ($–NH_2$) which are protonated to ($–NH_3^+$) in acidic conditions, resulting in a positive charge on the membrane surface. Humic acid, which at neutral pH has a negative charge due to the presence of carboxylic groups ($–COO^-$), experiences electrostatic repulsion (Donnan exclusion) when passing through a positively charged membrane[14]. In addition, the coating layer also reduces the pore size of the membrane, thereby increasing the size exclusion mechanism for large organic molecules. The combination of chitosan and natural extracts (e.g., garlic or lime extract) provides antibacterial and anti-biofouling effects that can reduce the adhesion of organic particles to the membrane surface. According to Rinaudo[16], chitosan has antibacterial activity and the ability to form a film that can increase the physical and chemical durability of the membrane. Also states that the antibiofouling coating can inhibit biofilm formation, which often exacerbates fouling by organic compounds. This explains why the decrease in humic acid concentration in coated membranes is higher than in non-coated membranes. A longer coating duration results in a denser and more homogeneous protective layer on the membrane surface. A thicker layer can seal most pores, increasing the membrane's selectivity in rejecting humic acid. However, this effect can also reduce water permeability due to greater flow resistance, creating a trade-off between permeability and rejection.

3.3. Membrane Hydrophilicity

Hydrophilicity is the ability of a surface to interact with water, which is determined by measuring the contact angle[17]. The WCA indicates the degree of wetness of the membrane surface[18]. A membrane contact angle value less than 90° indicates higher hydrophilicity, meaning the membrane can absorb water droplets on the membrane surface on the other hand, a contact angle value greater than 90° indicates higher membrane hydrophobicity, indicating that water droplets are unable to spread across the membrane surface[19]. The results of the WCA measurements are shown in **Figure 2**.

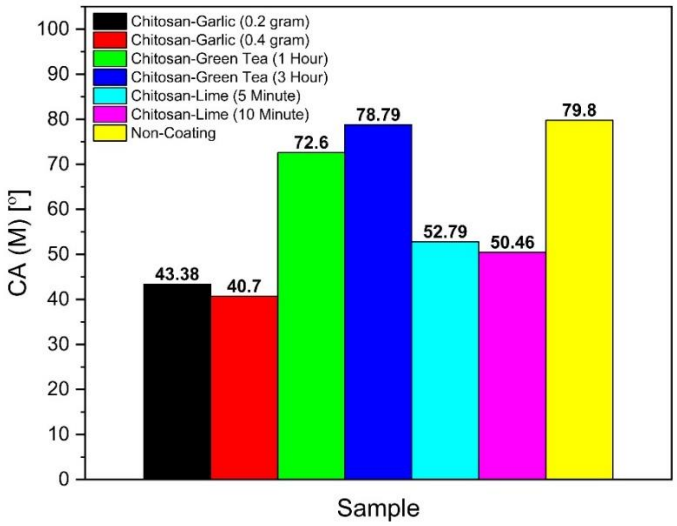

Figure 2. Water Contact Angle Graph.

Based on **Figure 2**, the initial WCA before coating was 79.8°. The initial water contact angle of the uncoated membrane was measured at 79.8°, which is consistent with previous findings by Putri et al.[6], who reported a contact angle of 78.9° for pristine Polyethersulfone (PES) membranes. Such values indicate moderate hydrophobicity, which can limit water permeability and increase the risk of membrane fouling. According to the graph, the lowest contact angle was observed in the chitosan–garlic sample with 0.4 g of garlic and 30 seconds of immersion time, while the highest contact angle was found in the chitosan–green tea sample with 3 hours of immersion, The decrease from 79.8° to 40.7° indicates a significant improvement in surface hydrophilicity, which is beneficial for water filtration applications due to enhanced water permeability and reduced fouling potential.

The decrease in WCA on the coated membrane is attributed to the addition of chitosan, which contains polar amine (-NH_2) and hydroxyl (-OH) groups[20]. Furthermore, according to Ding et al.[21], the incorporation of garlic extract can also reduce the WCA. This is due to the hydrogen bonding interactions between the active compounds in the extract and the polymer matrix. Overall, the combination of chitosan and garlic extract at an optimal concentration and immersion time enhances membrane hydrophilicity, indicating its potential for applications in water purification system.

3.4. Sustainability and Environmental Benefits

Natural coatings derived from garlic, lime, and green tea extracts offer a sustainable antibiofouling solution for the development of ultrafiltration membranes, with key advantages including their renewable, edible, food-grade, and non-toxic properties. Garlic extract contains phenolic compounds and allicin, which have been shown to reduce bacterial adhesion, such as Escherichia coli and Bacillus subtilis by more than 90% when applied to cellulose acetate membranes, while also enhancing hydrophilicity without leaving harmful residues[22]. Meanwhile, lime extract is rich in flavonoids that act as natural antibacterial agents capable of inhibiting Streptococcus mutans activity[23]. Green tea extract, abundant in catechins, also exhibits strong antibacterial and antioxidant properties, forming a hydrophilic layer that effectively suppresses biofilm formation. The Chitosan-Oligosaccharides Layer (COL)/ Poly (glycidyl methacrylate) PGT/PES membrane modified with green tea extract has demonstrated 93.5% filtration efficiency against E. coli and antibacterial activity exceeding 98% against both E. coli and S. mutans, making it superior to conventional PES membranes in terms of antifouling performance[12]. Ultrafiltration membranes are widely applied in everyday life for drinking water purification, domestic and industrial wastewater treatment, protein separation in the food industry, and in pharmaceuticals for sterilization and purification of biological compounds[24,25]. The

development of these natural bio-based coatings directly contributes to the achievement of SDGs 3 (Good Health and Well-being), SDGs 6 (Clean Water and Sanitation), SDGs 9 (Industry, Innovation and Infrastructure), and SDGs 12 (Responsible Consumption and Production) by providing safe, environmentally friendly technologies aligned with sustainable consumption and production practices.

4. Conclusion

Recycled ultrafiltration membranes converted from end-of-life reverse osmosis modules and coated with chitosan-based natural extracts (garlic, lime, green tea) exhibited substantially improved antifouling performance and surface hydrophilicity. The chitosan–garlic formulation (0.4 g garlic, short immersion) produced the best hydrophilicity (WCA reduced to 40.7°) and delivered significant reductions in humic acid permeation and improved salt rejection compared with uncoated recycled membranes. Increasing coating duration and additive loading increased salt and humic-acid rejection (e.g., Na_2SO_4 rejection up to ~34.9% after 3 h) but reduced permeability due to pore narrowing, demonstrating a clear trade-off between selectivity and flux. Overall, the combined recycling + bio-based coating approach successfully enhances membrane performance while advancing circularity and sustainability in membrane management.

Recommendations for future development

1. Optimize coating formulation and protocol
   Perform systematic optimization of chitosan concentration, extract loading (e.g., garlic), and immersion time to find the best compromise between permeability and rejection for target applications.
2. Long-term fouling and cleaning cycles
   Evaluate long-term performance under realistic cross-flow conditions using model and real wastewaters, including repeated fouling/cleaning cycles and flux-recovery tests to assess durability.
3. Antimicrobial and biofilm testing
   Quantify antibacterial activity and biofilm inhibition against representative fouling organisms (e.g., *E. coli, Pseudomonas spp*.) and measure biofilm formation under dynamic conditions.
4. Mechanical, chemical stability and leaching assessment
   Test coating adhesion, mechanical robustness, chemical resistance (e.g., exposure to oxidants, pH extremes), and potential leaching of bioactive compounds to ensure safety and longevity.
5. Advanced surface and structural characterization
   Use Scanning Electron Microscopy (SEM)/ Atomic Force Microscopy (AFM), X-ray Photoelectron Spectroscopy (XPS), FTIR (detailed), and zeta-potential measurements to correlate coating morphology, thickness and surface charge with filtration performance and fouling behavior.

Author contributions

Author Contributions. Rr. Dewi Putri conceptualized the research, provided overall supervision of the study, and contributed to the manuscript writing and editing. Maharani Kusumaningrum and Ima Winaningsih contributed to the manuscript writing and proofreading. Nisrina Hasna Nabil, Adhika Bintang Syahputra, Ridha Suryaning Sukma, Shofiah Zulfa Putri, Shandy Alif Fahrezi, and Nathan Aditya were responsible for data collection and data processing.

Acknowledgments

This research was funded by the Faculty Research fund through the DPA FT UNNES 2025 scheme with contract number 62.14.4/UN37/PPK.05/2025. The authors gratefully acknowledge the financial support provided by the Faculty of Engineering, Universitas Negeri Semarang.

Conflict of interest

The authors declare no conflict of interest.

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