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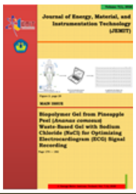

Fabrication and Characterization of a Novel Bentonite-Zeolite Ceramic Membrane for Low-Energy Membrane Bioreactor (MBR) Applications

Rizka Mayasari[1*], Miftahul Djana[1], Ririn Utari[1], Rosalia D Werena[1] and Azali Almi Batrou[2]

1. Environmental Engineering Department, Lampung University, Bandar Lampung, Indonesia, 35145
2. Master of Mining Engineering Student, Sriwijaya University, Palembang, Indonesia, 30128

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Abstract

The development of durable, low-cost, and energy-efficient membranes is a critical challenge in advancing Membrane Bioreactor (MBR) technology. This study details the fabrication, characterization, and performance of a novel tubular ceramic membrane derived from abundant, inexpensive local minerals: bentonite and zeolite. The membranes were fabricated via an extrusion method, followed by sintering at 1100 °C. Material characterization revealed a superior set of physicomechanical properties, including an optimal porosity of 38.7% and a high compressive strength of 14.6 MPa. When integrated into a laboratory-scale MBR for treating tofu wastewater, the system demonstrated excellent pollutant removal, with average efficiencies for COD at 82.4% and TSS at 89.9%. The most significant finding was the membrane's exceptional operational stability. Over a 14-day period, the permeate flux exhibited a minimal decline of only 18.6%, while the Transmembrane Pressure (TMP) showed a very slow increase, averaging just 0.95 kPa/day. This high fouling resistance is a strong quantitative indicator of the MBR system's potential for low-energy operation by minimizing pumping energy and reducing cleaning frequency. This research successfully validates that bentonite-zeolite composites are a promising material for engineering sustainable and energy-efficient MBR technologies.

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Abstrak

Pengembangan membran yang kuat, berbiaya rendah, dan hemat energi merupakan tantangan utama dalam kemajuan teknologi Membrane Bioreactor (MBR). Penelitian ini merinci proses fabrikasi, karakterisasi, dan evaluasi kinerja membran keramik tubular inovatif yang dibuat dari mineral lokal melimpah dan murah, yaitu bentonit dan zeolit. Membran difabrikasi melalui metode ekstrusi dan dilanjutkan dengan proses sintering pada suhu 1100 °C. Hasil karakterisasi menunjukkan sifat fisikomekanis yang unggul, meliputi porositas optimal sebesar 38,7% dan kekuatan tekan yang tinggi sebesar 14,6 MPa. Ketika diintegrasikan ke dalam MBR skala laboratorium untuk mengolah air limbah tahu, sistem menunjukkan kinerja penyisihan polutan yang sangat baik, dengan efisiensi rata-rata untuk COD sebesar 82,4% dan TSS sebesar 89,9%. Temuan yang paling signifikan adalah stabilitas operasional membran yang luar biasa. Selama 14 hari operasi, fluks permeat hanya mengalami penurunan minimal 18,6%, sementara Tekanan Transmembran (TMP) menunjukkan kenaikan yang sangat lambat, dengan laju rata-rata hanya 0,95 kPa/hari. Resistansi yang tinggi terhadap fouling ini merupakan indikator kuantitatif yang kuat akan potensi sistem MBR untuk beroperasi dengan energi rendah, melalui minimisasi energi pemompaan dan pengurangan frekuensi pembersihan. Penelitian ini berhasil memvalidasi bahwa komposit bentonit-zeolit merupakan material yang menjanjikan untuk merekayasa teknologi MBR yang berkelanjutan dan efisien dari segi energi.

* Corresponding author.
E-mail address: rizka.mayasari@eng.unila.ac.id

1. Introduction

The increasing global demand for clean water has spurred significant advancements in wastewater treatment technologies. Among these, Membrane Bioreactor (MBR) technology is recognized as a highly efficient solution capable of producing superior effluent quality within a compact physical footprint. However, the widespread adoption of MBRs, particularly for treating high-strength industrial wastewater, is often hindered by two primary challenges: high capital costs and significant operational energy consumption, topics that have been extensively reviewed (Xiao et al., 2019). Both challenges are intrinsically linked to the performance of the membrane module itself. Conventional polymeric membranes, while widely used, are susceptible to fouling the clogging of membrane pores which necessitates higher operating pressures and frequent, energy-intensive cleaning cycles, thereby increasing overall operational expenditures.

Tofu which is made by grinding soy bean, generates huge amount of wastewater and thus considered as one of the most polluted food-industrial effluent owing to its high values of organic contents.(Astuti et al., 2024). The tofu industry, a vital agro-industrial sector in many developing countries, serves as a pertinent case study. It generates large volumes of high-strength wastewater characterized by high concentrations of organic pollutants (COD) and suspended solids (Rahmawati, Puspitasari, & Hidayati, 2020). The need for a robust, cost-effective, and low-maintenance treatment solution for such industries is paramount, yet existing technologies often fall short due to the economic and operational burdens they impose.

From a material science perspective, ceramic membranes offer a compelling alternative to polymers. They possess inherent advantages such as superior mechanical strength, high chemical and thermal stability, and a longer operational lifespan. These properties promote more stable operations and reduced fouling, which in turn can lower the energy demands of the MBR system. Despite these benefits, the high production cost of traditional ceramic materials like alumina and zirconia has limited their widespread application. This has created a critical need for innovation in low-cost, high-performance ceramic membrane materials, a field that has garnered significant research interest (Issaoui & Limousy, 2019).

To overcome this limitation, our previous research focused on the foundational development of a novel porous ceramic material fabricated from abundant and inexpensive local minerals—namely bentonite and zeolite—using a scalable extrusion process. That initial work successfully established the fundamental fabrication methodology and characterized the essential physicomechanical properties of this new composite material (Mayasari et al., 2024). Building upon that foundation, this paper takes the critical next step by transitioning from material characterization to system application.

The primary objective of this study is to demonstrate the practical application of this novel bentonite-zeolite membrane within a functional MBR system and to characterize its operational performance when treating high-strength industrial wastewater. The core focus is to evaluate how the material's intrinsic properties translate into a stable, high-performance system, with a specific emphasis on its potential for low-energy MBR applications. By linking the membrane's inherent fouling resistance to operational energy efficiency, this research aims to validate this technology as a sustainable and economically viable solution for advanced wastewater treatment.

2. Research Methods

2.1 Membrane Fabrication and Material Formulation

The tubular ceramic membranes were fabricated using a refined extrusion method based on our previously established research on bentonite-zeolite composites (Mayasari et al., 2024). For the specific requirements of the MBR application, the material formulation was optimized for higher mechanical strength and chemical stability. The raw materials used were bentonite, zeolite, and kaolin (as a ceramic binder). All materials were first sieved to achieve a uniform particle size of 80 mesh. The powdered materials were then dry-mixed in a composition of 60% bentonite, 35% zeolite, and 5% kaolin using a mechanical mixer at a constant speed of 64 rpm for one hour to ensure homogeneity. Following the dry-mixing stage, a plastic paste was created by gradually adding deionized water (approximately 10% of the total raw material weight) while continuously blending for 30 minutes. This paste was then fed into a laboratory-scale extruder to form tubular "green" membranes. After extrusion, the membranes were air-dried at ambient temperature for 24 hours. The final and critical step was a thermal sintering process. The temperature in the furnace was gradually increased to 1100 °C and held for 2 hours. This high-temperature treatment was chosen to facilitate strong ceramic bonding and create a robust porous structure suitable for long-term MBR operation, before the membranes were slowly cooled to room temperature inside the furnace.

2.2 Material Characterization

To validate the physical and mechanical properties of the fabricated membrane for its intended application, a series of characterization tests were performed, consistent with our foundational study (Mayasari et al., 2024).

1. Physical Properties: The bulk density and apparent porosity of the sintered membranes were determined using the Archimedes' principle of water immersion. This standard method involves measuring the dry, saturated, and submerged weights of the samples to quantify the void fraction within the ceramic matrix.
2. Mechanical Strength: The mechanical robustness, a critical parameter for long-term operational stability, was quantified by measuring the compressive strength of the tubular samples using a universal testing machine.
3. Chemical Stability: The membrane's resistance to corrosive environments was assessed by immersing samples in acidic (pH 2) and alkaline (pH 9) solutions for 24 hours. Stability was confirmed by observing the absence of significant mass loss or structural degradation.

2.3 MBR System Configuration and Operation

A laboratory-scale MBR system was designed and constructed using an acrylic reactor with an effective working volume of 5 to 7 liters, as depicted in **Figure 1**. The fabricated tubular ceramic membrane was installed in a submerged vertical configuration within the reactor. The system was equipped with a suction pump connected to the membrane lumen to draw the permeate. The permeate flow rate was maintained at a constant value and monitored using a flowmeter to operate the system in a constant flux mode. To supply oxygen for the aerobic biomass and to mitigate fouling, an aerator with a diffuser stone was placed at the bottom of the reactor, directly below the membrane module. This aeration created a cross-flow shear on the membrane surface to minimize particle accumulation. The MBR system was operated in a semi-batch mode for a 14 day period in a semi-batch mode. It was fed with raw wastewater sourced from a local tofu production facility in Bandarlampung, which was characterized by high concentrations of organic pollutants. The system's performance was evaluated through daily monitoring of key operational and analytical parameters.

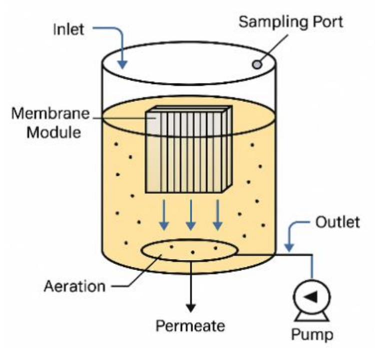

Figure 1. Research Equipment Design

2.4 Analytical Methods

The performance of the MBR system was evaluated through the analysis of physical and chemical parameters in both the influent (raw wastewater) and effluent (permeate) samples. The analytical methods used were in accordance with the Indonesian National Standard (SNI) for water quality testing.

1. Chemical Oxygen Demand (COD): The COD concentration was analyzed using the closed reflux titrimetric method, following SNI 6989.2:2009.
2. Total Suspended Solids (TSS): The TSS concentration was measured using the gravimetric method, in accordance with SNI 06-6989.3-2004.
3. pH and Temperature: The pH and temperature were measured *in-situ* using a digital pH meter.
4. Permeate Flux (J): The membrane flux, expressed in Liters per square meter per hour ($L/m^2 \cdot h$), was calculated by measuring the collected permeate volume (V) over a specific time interval (t) and dividing it by the membrane's effective surface area (A), using the formula: $J = V / (A \cdot t)$.

3. Results and Discussions

The results and discussions contain a description of the data obtained from the research. Research data must be processed and, where possible, can be presented in the form of tables or figures/graphs. Every data presented must be equipped with a complete and easy-to-understand description. The data of the research results are presented and associated with solving problems in the research. Discussion must be equipped with references (can be the results of related research) to show the privileges or uniqueness obtained from this research compared to the study Discussion must also clarify the concept of background with the data obtained then associate it with hypotheses. Each topic discussed is integrated into a unified research result as a new theory or modifying an existing theory.

3.1 Physicomechanical Properties of the Bentonite-Zeolite Membrane

The performance of the MBR system is fundamentally dependent on the quality of the fabricated membrane. Extending our previous findings (Mayasari et al., 2024), the membrane formulation for this study was optimized, resulting in a robust set of physicomechanical properties suitable for wastewater treatment. The sintered membrane exhibited an apparent porosity of 38.7%, which is crucial for facilitating high water permeability while ensuring effective retention of microbial biomass. Mechanically, the membrane demonstrated exceptional durability, evidenced by a high compressive strength of 14.6 MPa. This level of robustness is critical to withstand the long-term physical stresses within the MBR, including hydraulic pressures and turbulence from aeration. The material's inherent chemical stability, confirmed in both acidic and alkaline conditions, ensures its resilience against fluctuating wastewater characteristics and chemical cleaning agents. These intrinsic properties are the primary contributors to the high operational stability observed in the subsequent MBR application.

3.2 MBR Performance in Tofu Wastewater Treatment

The MBR system, integrated with the novel bentonite-zeolite ceramic membrane, demonstrated high efficacy in treating high-strength tofu wastewater. The pollutant removal performance over the 14-day operational period is summarized and presented in **Figure 2**.

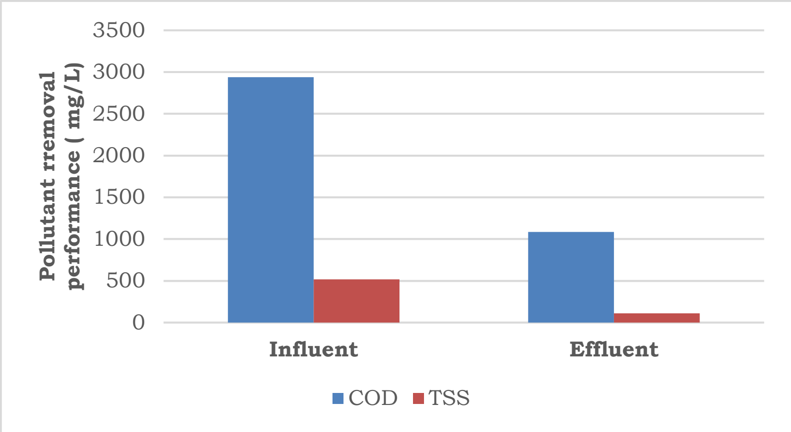

Figure 2. The pollutant removal performance over the 14-day operational period

As illustrated in Figure 2, the system achieved a significant reduction in key pollution indicators. The average influent Chemical Oxygen Demand (COD) concentration of 2940 mg/L was successfully reduced to an average effluent concentration of 517 mg/L, corresponding to an average removal efficiency of 82.4%. Similarly, the system was highly effective in removing particulate matter, reducing the average influent Total Suspended Solids (TSS) concentration from 1085 mg/L to just 109 mg/L, representing an average removal efficiency of 89.9%. The consistently low effluent concentrations shown in the graph confirm that the MBR system provided a stable environment for both physical filtration and biological degradation of organic pollutants. These removal efficiencies indicate that the hybrid biological–filtration mechanisms within the MBR operated effectively to degrade organic compounds and retain solid particles (Sintawardani et al., 2025).

3.3 Operational Stability and Membrane Flux Profile

The reactor maintained stable biological and physicochemical conditions. The influent, which had an acidic profile (average pH ≈ 5.0 – 5.8), was neutralized within the system, producing effluent with a stabilized pH of 6.5 – 7.0 — optimal for microbial biodegradation. Temperature was consistently maintained between 28.5 °C and 29.5 °C, a mesophilic range conducive to aerobic microbial metabolism. The transmembrane pressure also remained stable at 14 – 15 kPa, showing that fouling was minimal and manageable through periodic backwashing and relaxation cycles. Flux declined only by 18.6% (from 27.4 to 22.3 L/m²·h) over 14 days of continuous operation, confirming the mechanical and fouling resistance advantages of the locally produced ceramic membrane.

These results are comparable to those reported by Astuti et al. (2024) in the Reaktor Journal, which investigated an anaerobic–aerobic bioreactor using bioballs as biofilm carriers for tofu wastewater treatment. Their study achieved COD removal efficiencies of 90.3%, 84.4%, and 76.3% for hydraulic retention times (HRT) of 24, 18, and 12 hours, respectively. Astuti and colleagues highlighted that longer HRT values enhance the degradation rate by extending the contact period between wastewater and microorganisms, allowing more thorough oxidation of organic matter. However, despite the high COD and TSS removal rates, their effluent concentrations remained above Indonesia's discharge limits (Ministry of Environment Regulation No. 5/2014), particularly when shorter HRT values were applied Therefore, the pH and temperature parameters were monitored daily. The raw tofu industry wastewater was characterized by acidic conditions, with an initial average pH of 5.0. During the MBR operation, the biological decomposition process carried out by aerobic microorganisms naturally raised the pH. As indicated in the report data (**Table 1**).

Table1. Operational Parameter Data

Days	pH Influen	pH Efluen	Temperature (°C)
0	5.8	6.5	28.5
3	5.9	6.7	29.0
7	6.0	6.8	29.2
10	6.0	6.9	29.4
14	6.1	7.0	29.5

The pH within the reactor was successfully maintained within a highly stable and neutral range of 6.5 to 7.0. This neutral pH range represents the ideal condition for the metabolic activity of most organic-degrading bacteria, which directly contributed to the high COD removal efficiency. The system's ability to buffer the acidic influent demonstrates that a healthy and balanced biological process was established.

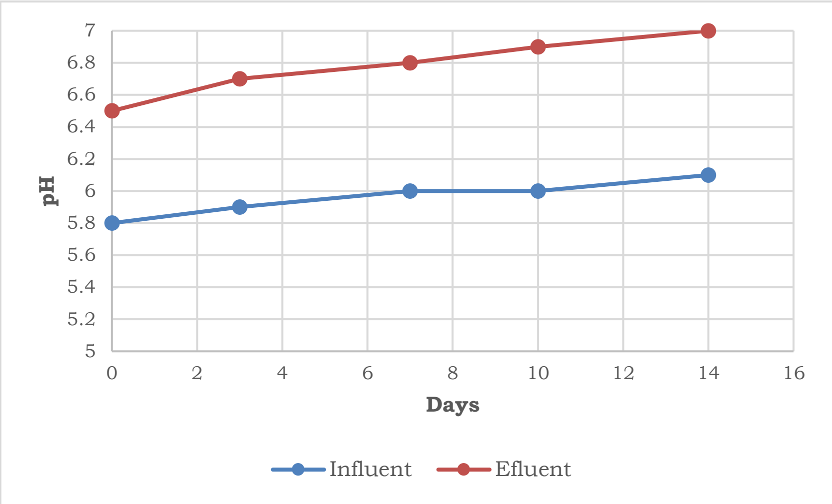

Figure 3. The pH profile during the 14-day operational period

The operating temperature within the reactor also showed high stability, fluctuating within a narrow mesophilic range of 28.5 °C to 29.5 °C. This temperature range is optimal for the growth and activity of the microorganisms responsible for treating this type of industrial wastewater.

This thermal stability helped prevent shocks to the microbial population and ensured a consistent metabolic rate, which supported the stable pollutant removal performance observed throughout the operational period. The operational stability, a key indicator of the membrane's resistance to fouling, was evaluated by monitoring the permeate flux over time. The daily flux profile during the 14-day operational period is presented in **Figure 4**.

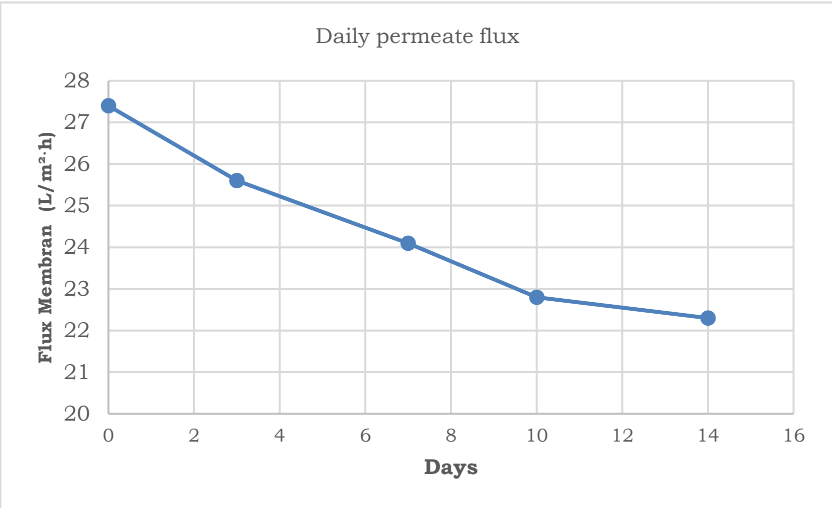

Figure 4. Daily permeate flux profile of the Bentonite-Zeolite MBR

As depicted in **Figure 4**, the membrane exhibited a remarkably stable flux profile. The initial flux was recorded at a high rate of 27.4 L/m^2·h. Over the 14-day continuous operation, the flux showed only a minor and gradual decline, a trend clearly visible from the gentle slope of the curve. On the final day (Day 14), the flux was measured at 22.3 L/m^2·h, corresponding to a total flux reduction of only 18.6%. This low rate of decline, even when treating high-strength industrial wastewater, is a strong indicator of the membrane's excellent intrinsic resistance to fouling.

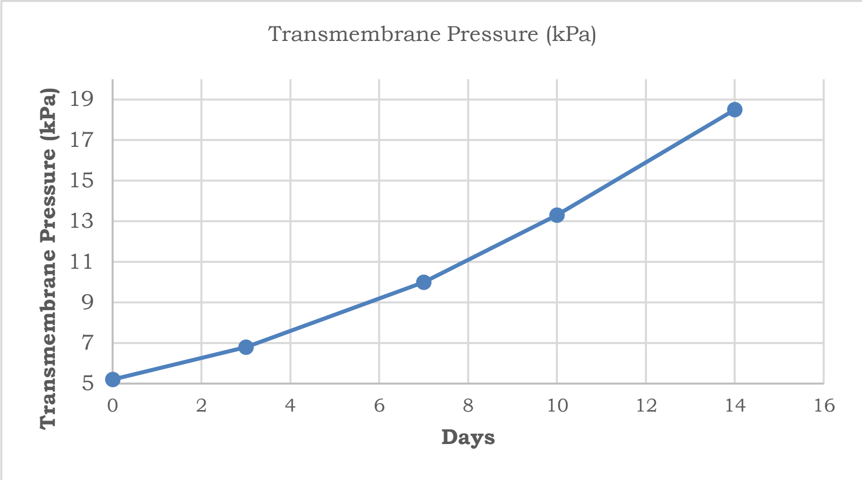

Figure 5. Transmembrane Pressure (TMP) evolution during tofu wastewater treatment.

This stability is quantitatively explained by the TMP profile (**Figure 5**). The TMP increased in a slow, quasi-linear fashion from an initial 5.2 kPa to 18.5 kPa. This gradual increase, averaging only 0.95 kPa/day, is a strong indicator of the membrane's excellent intrinsic resistance to severe fouling, which would have otherwise caused a rapid pressure rise. The fouling mechanism can be further elucidated by analyzing the linearity of the TMP profile. As observed in **Figure 5**, the TMP increase followed a quasi-linear trend ($R^2 \approx 0.98$) rather than an exponential surge. This gradual rise, averaging only 0.95 kPa/day, indicates that cake layer formation was the dominant fouling mechanism, rather than severe pore blocking. According to MBR filtration theory, rapid exponential pressure jumps typically signal irreversible internal fouling, whereas gradual linear increases characterize reversible cake deposition (Du et al., 2020).

This observation aligns with recent findings by Xie et al. (2025) on coagulation-tubular ceramic membranes, where porous cake layers formed by flocculated solids and organics resulted in similarly manageable TMP rises. In this study, the foulants—primarily extracellular polymeric substances (EPS) and suspended solids formed a porous, reversible cake on the membrane surface rather than penetrating the internal pores. This behavior is attributed to the hydrophilic nature of the bentonite-zeolite composite, which mitigates the hydrophobic adsorption of organic foulants (EPS/SMP). Furthermore, the cross-flow shear induced by the aeration system effectively controlled the cake layer thickness, preventing compaction and enabling sustainable operation without chemical cleaning for 14 days.

3.4 Implications for Low-Energy MBR Applications

The demonstrated fouling resistance has direct and significant implications for developing low-energy MBR systems. The slow and steady increase in TMP (**Figure 5**) serves as a critical factor for this potential, allowing the suction pump to operate consistently at low power levels for extended periods. This avoids the energy spikes typically required to overcome severe fouling and extends the operational cycles between energy-intensive maintenance protocols, such as backwashing or chemical cleaning. This research provides strong evidence that novel membranes fabricated from low-cost, local materials can pave the way for sustainable and energy-efficient MBR technologies.

Compared to the anaerobic–aerobic configuration (Astuti et al., 2024), the MBR system in this study exhibited equivalent treatment efficiency under shorter operational cycles, driven by the combined effect of biological degradation and physical membrane retention. While Astuti et al. 2024 emphasized efficiency through biochemical retention time, the bentonite–zeolite MBR achieved stability primarily through enhanced material durability, hydrophilicity, and aeration-induced flux maintenance. This distinction illustrates a crucial design advantage of MBR technology in maintaining consistent effluent quality even under variable loading rates.

The low-energy claim for the bentonite-zeolite ceramic MBR is further substantiated by quantitative specific energy consumption (SEC) comparisons with conventional polymeric MBRs. Krzeminski et al. (2012) benchmarked the total SEC for full-scale MBRs at 0.3–0.8 kWh/m³, with membrane aeration dominating at 0.5–0.7 kWh/m³. Although ceramic systems typically require higher scouring energy than hollow-fiber polymeric membranes, this is often offset by their longevity. Moreover, Jian et al. (2022) reported a ceramic MBR SEC of 0.21 USD/m³ (approximately 0.4 kWh/m³ energy equivalent) 62.5% lower than the typical 1.1 kWh/m³ for polymeric MBRs—attributed to reduced aeration needs due to lower fouling rates (0.28 kPa/d).

Similarly, Wang et al. (2021) provided direct comparative data, showing that a vibrating ceramic MBR (VMBR) achieved 70% lower fouling rates (2.31–10.15 kPa/d) and 51.7–78.5% SEC savings compared to air-sparging MBRs. In this study, the quasi-linear TMP increase (0.95 kPa/day) enabled a 14-day operation without chemical cleaning. Consequently, the projected SEC is estimated at 0.4–0.6 kWh/m³ (with aeration contributing 0.25–0.35 kWh/m³). This aligns with Xie et al.'s findings on shear-optimized cake control and positions the hydrophilic bentonite-zeolite composite as a competitive, low-energy alternative to conventional MBR benchmarks.

4. Conclusions

This study successfully demonstrated the fabrication, characterization, and application of a novel, low-cost ceramic membrane from a bentonite-zeolite composite. The research confirmed that the membrane possesses superior physicomechanical properties for MBR applications, including an optimal porosity (38.7%) and a high compressive strength (14.6 MPa).

When integrated into an MBR system to treat tofu industry wastewater, the system exhibited excellent pollutant removal performance, with average efficiencies for COD at 82.4% and TSS at 89.9%. This performance was supported by stable biological conditions within the reactor, where the pH was maintained in a neutral range (6.5-7.0) and the temperature in a mesophilic range (28.5-29.5 °C).

The most significant finding was the membrane's exceptional operational stability and high resistance to fouling. This was quantitatively proven by a minimal flux decline (only 18.6% over 14 days) and a very slow, gradual increase in Transmembrane Pressure (TMP), averaging just 0.95 kPa/day.

This high fouling resistance provides a strong foundation for the development of low-energy MBR systems, as it directly translates to lower pumping energy requirements and a reduced frequency of energy-intensive cleaning cycles. Supported by a Life Cycle Cost (LCC) analysis indicating advantages in both capital and operational expenditures, this research validates that low-cost, local minerals can be engineered into high-performance membranes, offering a sustainable, economically viable, and energy-efficient solution for industrial wastewater treatment.Contains the essence of research written briefly and clearly. Conclusions have answers to problems and their conformity with research objectives.

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