International Journal of Nanoelectronics and Materials
IJNeaM
ISSN 1985-5761 | E-ISSN 2232-1535

A novel conductive polymeric hollow fiber membrane as anode for enhanced electrocatalytic dye degradation and separation

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Received 18 May 2025, Revised 24 June 2025, Accepted 2 July 2025

ABSTRACT

Methylene blue, a widely used synthetic dye, poses significant environmental and health risks due to its non-biodegradable nature, toxicity, and persistence in aquatic systems. Addressing its removal has become a priority, with electrocatalytic systems gaining attention for their high efficiency in degrading such pollutants. These systems utilize reactive oxygen species generated at the electrodes to break down dyes. In this study, for the first time, conductive polymeric hollow fiber membranes were developed as anodes in electrocatalytic systems to enhance methylene blue degradation. The membranes were prepared using a phase inversion technique, incorporating polysulfone (PSf) and varying polyaniline (PANI) contents (0 wt.%, 1 wt.%, 3 wt.%, and 5 wt.%). Results demonstrated that increased PANI content significantly improved conductivity, with the highest conductivity of 0.0006 S/cm observed for 5 wt.% PANI, correlating with a maximum methylene blue degradation efficiency of nearly 70%. This study contributes by introducing an innovative membrane design, combining conductivity and electrocatalytic performance for sustainable wastewater treatment.

Keywords: *Electrocatalytic; Polymeric hollow fiber membrane; Dye Separation; Conductive Membrane*

1. INTRODUCTION

Dye wastewater from the textile industry poses severe environmental challenges due to its stability, toxicity, and resistance to degradation, with approximately 280,000 tons of dyes annually released into water bodies, threatening ecosystems and biodiversity. Conventional treatments like coagulation-flocculation, adsorption, and biological methods face limitations such as high costs, sludge generation, and inefficiency against synthetic dyes. Advanced methods like photocatalysis are promising but are hindered by slow reaction rates and scalability issues, highlighting the urgent need for innovative, efficient, and sustainable wastewater treatment technologies [1]. It's interesting to note that electrocatalytic systems have become known as a potential new method for wastewater dye removal, with notable benefits including high efficiency and versatility for a variety of dyes. Direct or indirect dye degradation is made possible by electrocatalytic systems, which use electrical energy to induce oxidation and reduction reactions at electrodes.

The anode of these novel systems, which is usually composed of boron-doped diamond (BDD) or titanium coated with ruthenium oxide (Ti/RuO_2), is essential for producing reactive oxygen species (ROS), including hydroxyl radicals (•OH). Strong oxidizing agents, these ROS target and degrade dye molecules into smaller, less dangerous derivatives. The dye degradation process is completed when the cathode, which is usually composed of materials like graphite or stainless steel, aids in the reduction reaction of the intermediate compounds [2]. However, electrocatalytic processes also face similar challenges to photocatalytic methods. One of the main issues is the separation of degradation products and remaining electrode particles from the treated effluent, which can complicate subsequent treatment processes and increase operating costs. In addition, electrodes made from Ti/RuO_2 are relatively costly and may exhibit a decline in performance over time due to fouling or surface passivation. In contrast, although BDD electrodes are known for their superior oxidative strength, their application is often limited by high manufacturing expenses, physical fragility, and susceptibility to damage under extreme operational conditions [3].

To address practical application challenges, a novel conductive polymeric hollow fiber membrane composed of PSf and PANI was developed using the phase inversion method. Notably, polymer membranes provide advantages over traditional anode topologies by fusing flexibility and high surface area with improved separation capabilities,

enabling selective transport of ions or molecules and electron transfer to happen simultaneously in a small system. Because of these benefits, polymer membranes are ideal for small systems that need both procedures. PSf was chosen as the base material due to its high mechanical strength, good permeability, ease of modification, and simple fabrication process. In addition, polyaniline (PANI), as a conductive polymer, was incorporated into the membrane matrix to enhance its electrical conductivity and catalytic properties for use in electrocatalytic systems for the first time [4]. Furthermore, the developed membrane was systematically characterized to evaluate its morphological properties, electrical conductivity, and degradation performance in dye removal applications. By investigating the membrane composition and reaction conditions, this study aims to provide valuable insights into the development of conductive membranes for water treatment. Through comprehensive analysis, this study contributes to the ongoing pursuit of innovative, scalable, and eco-friendly solutions for water treatment challenges.

2. MATERIALS

The P-1700 Polysulfone (PSf) pellets were obtained from the Solvay company. Polyaniline (PANI) was purchased from Merck (Germany). Polyvinylpyrrolidone (PVP) as a pore agent and N-Dimethylacetamide (DMAc) as a solvent were procured from Sigma-Aldrich (USA).

2.1. Preparation of novel conductive polymeric hollow fiber membrane

In this study, PSf pellets were dried at 60 °C for 24 hours to eliminate moisture, ensuring proper dissolution in the DMAc solution. The weighed DMAc solution was mixed with polyaniline (PANI) and stirred for 24 hours using a mechanical stirrer, followed by 30 minutes of sonication to improve particle dispersion. Subsequently, the pore former and additional polymer, as detailed in Table 1, were added and stirred for another 24 hours to ensure uniform mixing, with the temperature maintained at 75 °C for efficient polymer dissolution. The pore former played a critical role in forming a porous structure suitable for ultrafiltration. After blending, the dope solution was sonicated for 45 minutes to remove trapped gases before being transferred into reservoirs. Conductive polymeric hollow fiber membranes were then fabricated by co-extruding the dope solutions through a triple-orifice spinneret into a coagulation bath of tap water, following the spinning parameters in Table 2. The membranes were collected on a rotating drum, submerged in water for two days to remove residual solvents, and finally dried at room temperature.

Table 1. Dope solution for conductive polymeric hollow fiber membrane

Membrane	Dope composition (%wt)			
	PSf	PANI	PVP	DMAc
M0	18	-	3	79
M1	18	1	3	78
M3	18	3	3	76
M5	18	5	3	74

Table 2. Spinning Parameter of Conductive Polymeric Hollow Fiber Membrane

Parameter	Specification
Bore fluid flow rate (ml/min)	8
Inner layer flow rate (ml/min)	26
Bore fluid	Distilled water
Air gap (cm)	10
Coagulant temperature	Room temperature
Spinner rate dimension	1.2 mm
Take up speed	Free fall
Coagulant	Tap water

2.2. Characterization of conductive membrane at different PANI content

The conductive polymeric hollow fiber membranes were characterized using several analytical methods to evaluate their performance. A Hitachi TM3000 SEM was used for morphological analysis to observe surface and structural features. A Contact Angle Goniometer (OCA 15EC) measured static contact angles to assess hydrophilicity and wettability. A Zwick/Roell universal testing machine evaluated tensile strength and elongation at break to determine mechanical properties. An Autolab potentiostat measured conductivity to assess electrical performance. In this study, a four-probe method was employed to accurately measure the electrical conductivity of the hollow fiber membrane. The experimental setup included a multimeter and an AutoLab potentiostat configured with a three-electrode system consisting of a working electrode (WE), a reference electrode (RE), and a counter electrode (CE). Silver wires were tightly coiled at both ends of the membrane to serve as electrical contacts. A known resistor (Rknown) was placed in series with the membrane, and the potentiostat applied a controlled current through the circuit. The voltage drops across the known resistor was

used to calculate the circuit current (Icircuit), while the multimeter simultaneously measured the potential difference across the membrane (Vmeasured). Using Ohm's law, the resistance of the fiber was determined, and subsequently, its conductivity was calculated by incorporating the physical dimensions of the membrane. This four-probe technique minimized contact resistance errors, thereby enabling precise evaluation of the membrane's intrinsic electrical properties. Figure 1 shows the experimental setup used for performing the conductivity measurements.

$$I_{circuit} = \frac{V_R}{R_{known}} \quad (1)$$

$$R_{fiber} = \frac{V_{measured}}{I_{circuit}} \quad (2)$$

$$Conductivity, \sigma = \frac{L}{R_{fiber} \bullet A} \quad (3)$$

where *R* is resistance (obtained from the slope of current vs. potential), L is the length of the membrane, and A is the cross-sectional area.

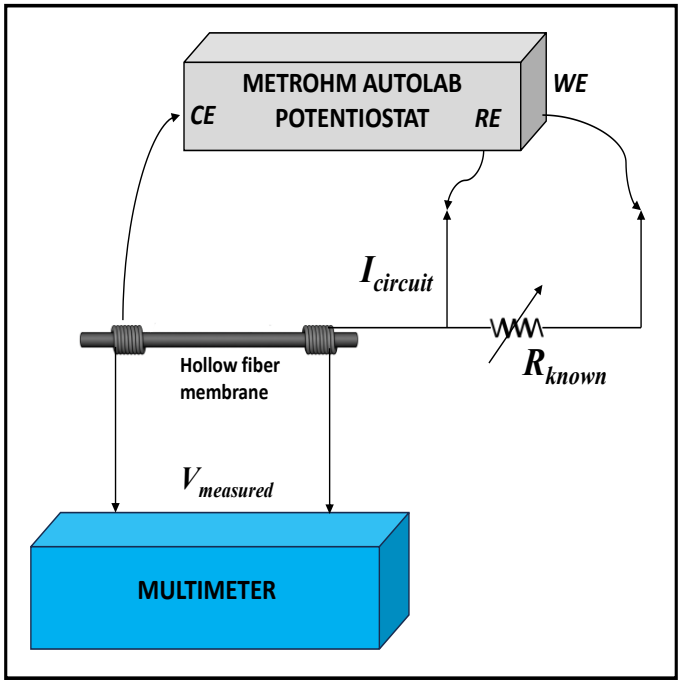

Figure 1. The experimental setup used for performing the conductivity measurements

2.3. Performance of the electrocatalytic membrane at different PANI content

Figure 1 shows a novel electrocatalytic system designed for an advanced oxidation process (AOP), utilizing a conductive polymeric hollow fiber membrane (PSf and PANI) as the anode and a graphite electrode as the cathode. In this system, a 100-ppm methylene blue solution was used as the feed solution and delivered into the reactor using a peristaltic pump, where the electrocatalytic reaction occurred. A DC power supply provided a current of 500 µA to drive the electrochemical reactions. The test was conducted for 180 minutes, with samples collected every 20 minutes and analyzed for color degradation using a UV-Vis spectrophotometer (Shimadzu UV-1800, Japan). The color degradation was evaluated using the equation:

$$Color\ Degradation\ (\%) = \frac{C_o - C_t}{C_o}\ x\ 100\% \quad (4)$$

Where C_o is the initial absorbance of the solution and C_t is the absorbance at time (t). This setup highlights the potential of the system for efficient water treatment and pollutant decomposition.

2.4. Electrocatalytic membrane system

The electrocatalytic membrane reactor operated by integrating both filtration and electrochemical oxidation mechanisms in order to treat colored wastewater. In this setup, a conductive hollow fiber membrane embedded with polyaniline (PANI) functioned as the anode, while graphite electrodes served as the cathode. A DC power supply was connected to the system to apply a controlled voltage and current, thereby creating an electric field across the electrodes submerged in the polluted water.

Once the system was activated, electrons were withdrawn from the anode and supplied to the cathode. At the same time, the oxidation reaction occurred, primarily generating reactive oxygen species (ROS) such as hydroxyl radicals (•OH), superoxide radicals ($O_2\bullet^-$), and other oxidants. These highly reactive species exhibit strong oxidative potential and play a pivotal role in degrading complex organic pollutants. They effectively facilitate dye ring cleavage and promote the mineralization of compounds that are typically resistant to conventional treatment methods. Moreover, the PANI layer facilitated electron transport and enhanced conductivity on the membrane surface, ensuring that the oxidation reactions occurred efficiently along the membrane walls. Simultaneously, the hollow fiber membrane provided physical separation, allowing treated water to permeate while retaining larger or partially oxidized molecules. This dual function improved the overall removal efficiency. At the cathode (graphite electrode), reduction reactions occurred.

The pollutants underwent electrocatalytic degradation, which involved multiple steps, including electron transfer, radical attack, and oxidative ring cleavage. Methylene blue, being a smaller molecule, degraded more quickly into smaller, colorless molecules or CO_2. Over time, the combined effect of continuous ROS generation, applied voltage, membrane filtration, and catalytic surface reactions led to a significant reduction in color and organic content. By optimizing parameters such as voltage, current, electrode spacing, and treatment duration, the reactor demonstrated the capability to degrade persistent pollutants in a controlled and scalable process. Figure 2 shows the setup and mechanism of the electrocatalytic system.

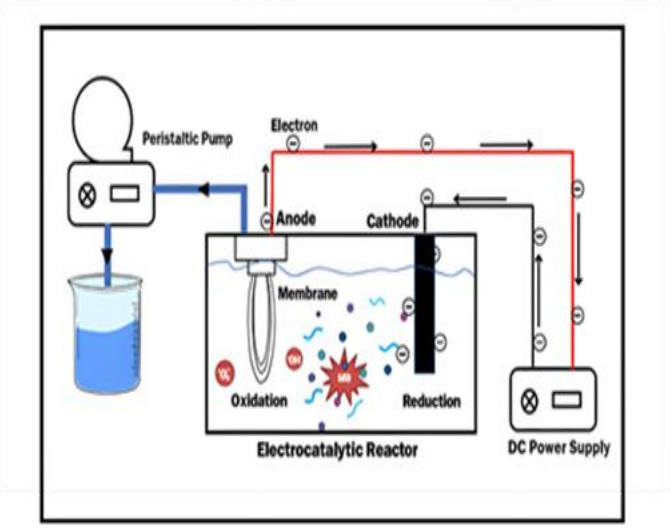

Figure 2. Novel Electrocatalytic System with Conductive Polymeric Hollow Fiber Membrane as Anode and Graphite Electrode as Cathode

3. RESULTS AND DISCUSSION

The cross-sectional SEM images of conductive polymeric hollow fiber membranes (M0, M1, M2, and M3), as shown in Figure 2, reveal structural differences that significantly affect their performance in color degradation. M0, the membrane without PANI, has a less compact structure with larger pores. In contrast, membranes with PANI (M1-M3) exhibit increasingly dense structures as the PANI content rises to 1 wt%, 3 wt%, and 5 wt%. This increasing compactness improves mechanical strength and electron transfer properties but can reduce porosity and hinder fluid flow at higher PANI content, as seen in the 5 wt% membrane (M3). In this regard, the 3 wt% PANI membrane (M2) achieves a more balanced and uniform porous structure with well-defined channels, enhancing pollutant transport and interaction with the membrane surface, which are essential for effective color degradation. At higher magnifications, the 3 wt% PANI membrane demonstrates an optimal balance between porosity and structural integrity, allowing better interaction between dye molecules and the membrane surface. This result was in agreement with Fahmy *et al.* (2023) [5] and Butt *et al.* (2022) [6] support that conductive polymers like PANI improve electrocatalytic activity by enhancing electron transfer and pollutant breakdown.

Table 3 shows the water contact angle, porosity, and tensile strength of conductive PSf/PVP/PANI hollow fiber membranes at different PANI contents. As can be observed, the water contact angle of PSf/PVP/PANI hollow fiber membranes decreases as the PANI content increases. The pristine membrane (M0) has the highest water contact angle of 86.0°, reflecting a more hydrophobic surface. It is interesting to note that when PANI is incorporated into the membrane, the water contact angle decreases to 78.8° for M1, 75.2° for M3, and 69.9° for M5, indicating increased hydrophilicity. This improvement is due to PANI's inherent hydrophilic properties and its ability to introduce polar groups into the membrane structure. It has been established that hydrophilicity is critical for enhanced water permeability, as reported by Al-Qasas [7], which demonstrated that conductive polymers like PANI improve water affinity, facilitating better wettability and enhanced performance in water treatment applications.

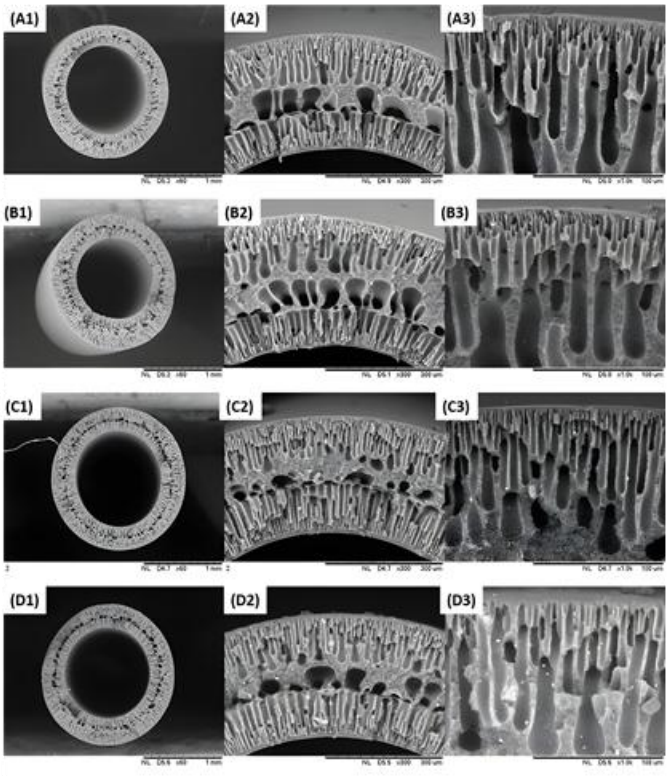

Figure 3. Cross-sectional SEM images of the conductive polymeric hollow fiber membrane at different PANI contents: (A) M0 (B) M1, (C) M3, and (D) M5; (1) the overall cross section, (2) x300 magnification, and (3) x1000 magnification

Meanwhile, the porosity of the membranes increases significantly with higher PANI content (Table 3). The M0 membrane has a porosity of 40.9%, while the porosity reaches 56.4%, 73.2%, and 80.2% for M1, M3, and M5, respectively. The SEM images (Figure 2) support these observations, showing that the incorporation of PANI modifies the membrane's internal structure, creating a denser yet more interconnected porous network at moderate PANI levels (e.g., M3). This interconnected porosity facilitates pollutant transport and interaction with the membrane surface, crucial for effective dye removal. This is explained, as previously highlighted by David *et al.* (2020), by the fact that an optimal pore structure enhances water and ion transport, improving performance in electrocatalytic systems [8].

Accordingly, the tensile strength of the membranes decreases as PANI content increases, dropping from 4.85 MPa for M0 to 4.35 MPa, 3.6 MPa, and 4.0 MPa for M1, M3, and M5, respectively (Table 3). This decline in mechanical strength is attributed to the disruption of the polymer matrix as PANI content rises. While PANI enhances conductivity and porosity, it reduces the overall cohesion within the PSf/PVP matrix. Interestingly, the slight recovery in tensile strength at M5 (4.0 MPa) may be due to enhanced compaction of the matrix, as indicated in the SEM images. Maintaining mechanical integrity is essential for practical applications, and previous studies, such as Kabanova *et al.* (2024), emphasized the importance of balancing

mechanical strength with functional properties for sustainable membrane performance [9]

Table 3. Water Contact Angle, Porosity and Tensile Strength of Conductive Polymeric Hollow Fiber Membrane

Conductive Membranes	Water Contact Angle (°)	Porosity (%)	Tensile Strength (MPa)
M0	86.0 ± 0.34	40.9 ± 0.45	4.85 ± 0.33
M1	78.8 ± 0.60	56.4 ± 0.34	4.35 ± 0.33
M3	75.2 ± 0.30	73.2 ± 0.36	3.6 ± 0.36
M5	69.9 ± 0.18	80.2 ± 0.49	4.0 ± 0.09

(Number of samples, n = 3)

The conductivity results presented in Table 4 demonstrate a strong correlation between the PANI content in the membranes and their electron transfer capabilities. Herein, M5, with the highest PANI loading, exhibited the highest conductivity (0.0006 S/cm), attributed to the formation of a dense yet interconnected conductive network. This result is consistent with the SEM images (Figure 1), which reveal a compact structure that facilitates efficient electron transport pathways. However, the excessive compaction observed in M5 compromises tensile strength, reducing it to 4.0 MPa, thereby highlighting the trade-off between conductivity and mechanical stability. Furthermore, the increased hydrophilicity, as evidenced by the reduced water contact angle (69.9°), enhances dye interaction and promotes the generation of reactive oxygen species (ROS), essential for electrocatalytic processes.

In contrast, M3 showed a more balanced performance with moderate conductivity (0.0002 S/cm) and better structural stability, making it a potential candidate for large-scale applications. SEM analysis showed that M3 maintained an optimal porous structure, supporting efficient ion transport and effective dye degradation. This finding was further supported by a lower water contact angle (75.2°) and a tensile strength of 3.6 MPa, indicating a balance between hydrophilicity and mechanical integrity. Interestingly, previous studies, as reported by Guillen *et al.* (2010), also emphasized the role of the dual functions of PANI in enhancing conductivity and hydrophilicity, thereby improving electrocatalytic performance [10]. Therefore, although M5 recorded the highest conductivity, M3 offered the best combination of conductivity, porosity, and mechanical properties, ensuring long-term operational stability in wastewater treatment systems.

Table 4. Conductivity Result for Conductive Polymeric Hollow Fiber Membrane

Membrane	Voltage, V	Current, A	Resistance, R (Ω)	Conductivity, σ (S/cm)
M0	5	1.44348×10^{-9}	3.46383×10^{9}	$(1.60388 \pm 0.05) \times 10^{-8}$
M1	5	1.77917×10^{-9}	2.81031×10^{9}	$(1.97684 \pm 0.04) \times 10^{-8}$
M3	5	1.61194×10^{-5}	3.10185×10^{5}	0.0002 ± 0.00001
M5	5	5.25208×10^{-5}	9.5200×10^{4}	0.0006 ± 0.00002

In order to study the effect of the conductivity of the conductive polymeric hollow fiber membrane as an anode in a novel electrocatalytic system, the methylene blue degradation was performed for 180 minutes, as shown in Figure 3. As expected, M0 displays negligible degradation, reaching less than 5% after 180 minutes. Remarkably, M1 achieves approximately 10% degradation, while M3 shows a substantial improvement, reaching about 30%. Interestingly, M5 demonstrates the highest efficiency, with degradation peaking at nearly 70%. This result showed that the main contribution to the trend can be attributed to the enhanced generation of reactive oxygen species (ROS) facilitated by the incorporation of PANI. The conductive pathways formed by PANI improve electron transfer, enabling efficient production of ROS like hydroxyl radicals (•OH) and superoxide radicals (O_2^-), which actively degrade methylene blue. This mechanism aligns with findings by Sajith *et al.* (2024), who reported that PANI-based composites effectively degrade textile dyes through ROS generation under light irradiation [11].

To further understand the electrochemical behavior of the electrocatalytic system, it is essential to consider the reaction kinetics governing pollutant degradation. The process follows a pseudo-first-order reaction model with respect to the dye concentration. When a constant voltage is applied, hydroxyl radicals (•OH), superoxide radicals ($O_2•^-$), and other reactive oxygen species (ROS) are generated at the surface of the conductive membrane. These radicals initiate oxidation of dye molecules by abstracting hydrogen atoms or attacking the chromophoric groups, leading to decolorization and breakdown into simpler organic acids or CO_2 and water.

Although PANI in this system does not directly donate electrons, its conductive properties enable efficient electron transport across the membrane surface. This facilitates the movement of externally supplied electrons from the DC power source to active reaction sites, supporting continuous ROS generation. These conditions accelerate degradation efficiency by supporting reactive species activity and maintaining electrical connectivity. Similar findings have been reported by Ramohlola *et al.* (2024), who demonstrated enhanced current density through PANI-assisted electron mobility in electrochemical membranes [12].

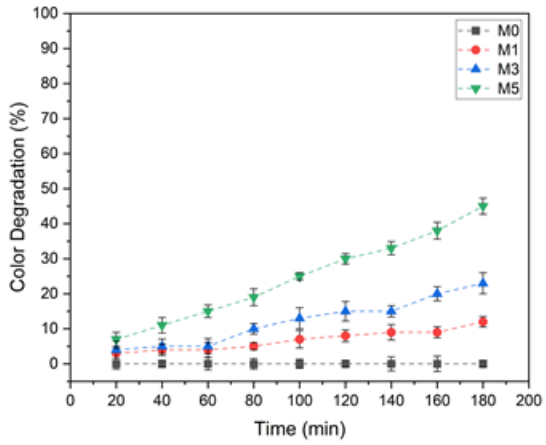

Figure 4. Methylene blue degradation via conductive membrane as anode at different PANI content via electrocatalytic

4. CONCLUSION

Among the membranes developed, M5 (5% PANI) exhibited the highest performance, with a conductivity of 0.0006 S/cm and nearly 70% methylene blue degradation due to enhanced reactive oxygen species (ROS) generation, increased hydrophilicity (water contact angle of 69.9°), and high porosity (80.2%). However, its dense SEM morphology and reduced tensile strength (4.0 MPa) suggest limited mechanical stability. In contrast, M3 (3% PANI) demonstrated a well-balanced performance with a conductivity of 0.0002 S/cm, methylene blue degradation of 30%, moderate hydrophilicity (75.2°), optimal porosity (73.2%), and improved tensile strength (3.6 MPa). The SEM analysis confirmed that M3's well-defined porous structure facilitated efficient pollutant transport while maintaining mechanical integrity. Therefore, M3 emerged as the most suitable membrane for long-term applications, balancing conductivity, stability, and electrocatalytic efficiency. To further enhance the membrane's catalytic performance, incorporating electrocatalysts such as manganese dioxide (MnO_2) is recommended, as this could promote ROS production and improve dye degradation efficiency in electrocatalytic wastewater treatment systems.

5. ACKNOWLEDGEMENT

The authors gratefully acknowledge the financial support from the Ministry of Higher Education Malaysia under the Higher Institution Centre of Excellence (R.J130000.7809.4J663) and Universiti Teknologi Malaysia under UTM Fundamental Research (Project number: Q.J130000.3809.23H89) and UTM-UNSRI Matching Grant (Project number: R.J130000.7301.1U055).

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