

Effect of TiO₂ Nanoparticle Incorporation on the Hydrophilicity and Stability of NIPS-Fabricated PVDF Membranes for Methylene Blue Removal

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Abstrak

Water pollution resulting from the presence of synthetic dyes such as Methylene Blue has become a serious environmental concern due to its high stability, toxicity, and resistance to natural degradation. Membrane-based separation technology is considered a promising approach for treating colored wastewater; however, the application of Polyvinylidene Fluoride (PVDF) membranes is still limited by their inherent hydrophobic nature, which leads to fouling and a decline in filtration performance during operation. This study aims to investigate the effect of varying concentrations of titanium dioxide (TiO₂) nanoparticles on the hydrophilicity and operational stability of PVDF membranes fabricated via the Non-Solvent Induced Phase Separation (NIPS) method. PVDF-TiO₂ composite membranes were prepared with TiO₂ loadings of 0%, 5%, 10%, and 15% relative to the mass of PVDF. Membrane hydrophilicity was characterized using contact angle measurements, while membrane stability was evaluated through repeated filtration tests using a Methylene Blue solution. The results demonstrate that the incorporation of TiO₂ significantly enhances membrane hydrophilicity, as indicated by a reduction in contact angle compared to pristine PVDF membranes. The membrane containing 5% TiO₂ exhibited the highest hydrophilicity, reflected by the lowest contact angle value. Improved hydrophilicity contributed to enhanced operational stability by reducing fouling tendency during the filtration process. However, excessive TiO₂ loading led to nanoparticle agglomeration, which diminished the effectiveness of hydrophilicity enhancement. Overall, the findings indicate that PVDF membrane modification with TiO₂ is an effective strategy to improve hydrophilicity and maintain membrane performance stability in Methylene Blue removal applications.

Keywords: PVDF, TiO₂, hidrofilisitas, stabilitas membran, fouling, Methylene Blue.

INTRODUCTION

The availability of clean water has become one of the major challenges in sustainable development, driven by increasing industrial activities and global population growth (Eddy et al., 2023). Various industrial sectors, particularly textiles, printing, paper, and cosmetics, generate large volumes of wastewater containing synthetic dyes (Zou & Lee, 2022). One of the most commonly found dyes in industrial effluents is Methylene

Blue, a cationic dye characterized by high chemical stability and strong resistance to natural degradation processes (Crini & Lichtfouse, 2019). The presence of Methylene Blue in aquatic environments can reduce light penetration, disrupt the photosynthetic processes of aquatic organisms, and induce toxic effects on aquatic biota (Benalia et al., 2024).

Various wastewater treatment methods have been developed to address dye pollution, including coagulation-

flocculation, adsorption, chemical oxidation, biodegradation, and electrochemical processes. However, most of these methods still exhibit limitations such as high chemical demand, the generation of secondary sludge, high energy consumption, or suboptimal removal efficiency (Khan et al., 2022). Therefore, membrane-based separation technology has emerged as a promising alternative due to its high efficiency, relatively low energy consumption, and ability to produce high-quality permeate (Field & Wu, 2022).

Polyvinylidene Fluoride (PVDF) is one of the most widely used membrane materials due to its excellent thermal stability, chemical resistance, and high mechanical strength (Kim et al., 2020). However, PVDF inherently exhibits hydrophobic characteristics, which promote strong interactions between the membrane surface and organic pollutants. This condition leads to membrane fouling, resulting in flux decline, increased flow resistance, and reduced operational lifespan of the membrane (Zhang et al., 2022).

One widely adopted approach to overcome these limitations is membrane modification using inorganic nanoparticles. Titanium dioxide (TiO₂) is among the most commonly used nanoparticles due to its hydrophilic properties, chemical stability, non-toxic nature, and strong photocatalytic activity (Benhabiles et al., 2019). The incorporation of TiO₂ into the PVDF matrix has been reported to increase the membrane surface energy, thereby enhancing its wettability and reducing fouling propensity (Chamam et al., 2025).

Several previous studies have demonstrated the beneficial effects of TiO₂ incorporation into PVDF membranes. (Benhabiles et al., 2019) reported that TiO₂ nanoparticles significantly improved membrane

hydrophilicity and antifouling performance. Likewise, (Zhang et al., 2022) observed that TiO₂ enhanced water permeability and reduced membrane fouling through improved surface wettability. Furthermore, (Chamam et al., 2025) found that the addition of TiO₂ promoted more uniform membrane morphology and enhanced filtration performance. These findings indicate that TiO₂ is an effective modifier for improving PVDF membrane characteristics.

In addition to enhancing hydrophilicity, the incorporation of TiO₂ also has the potential to improve membrane operational stability during the filtration process (Zhang et al., 2022). A more hydrophilic membrane surface tends to form a hydration layer that can inhibit the adsorption of organic molecules onto the membrane surface. Consequently, flux decline due to fouling can be minimized, resulting in more stable membrane performance over a prolonged operational period (AlSawaftah et al., 2021).

Although various studies have reported improvements in membrane hydrophilicity resulting from the incorporation of TiO₂, investigations into the relationship between different TiO₂ concentrations and the hydrophilicity as well as operational stability of PVDF membranes fabricated via the Non-Solvent Induced Phase Separation (NIPS) method remain limited (Zulkefli et al., 2021).

Most previous studies have primarily focused on improving membrane permeability, antifouling behavior, or photocatalytic performance. However, comprehensive investigations that systematically evaluate the influence of different TiO₂ concentrations on both membrane hydrophilicity and operational stability under repeated filtration cycles remain scarce. In addition, limited studies have specifically examined these characteristics in PVDF membranes

fabricated using the NIPS method for Methylene Blue removal.

Therefore, this study was conducted to evaluate the effect of varying TiO₂ nanoparticle concentrations on the hydrophilicity and operational stability of PVDF membranes in the removal of Methylene Blue (Benhabiles et al., 2019).

The novelty of this study lies in the systematic evaluation of TiO₂ nanoparticle concentration on the hydrophilicity and operational stability of PVDF membranes fabricated using the NIPS method. Unlike previous studies that mainly emphasized permeability or antifouling performance, this work correlates variations in TiO₂ concentration with membrane wettability and flux stability during repeated filtration cycles for Methylene Blue removal, thereby providing a more comprehensive understanding of membrane performance optimization.

MATERIAL AND METHODS

This study employed Polyvinylidene Fluoride (PVDF) as the primary membrane polymer and titanium dioxide (TiO₂) nanoparticles as an inorganic additive to enhance membrane hydrophilicity. PVDF, TiO₂ nanoparticles, and glycerol were obtained from Sigma-Aldrich. In addition, N-Methyl-2-Pyrrolidone (NMP) and acetone with a purity of 99% were used as solvents, Methylene Blue as a model synthetic dye solution, and deionized water as the coagulation medium. All materials were used without further purification (Li et al., 2025).

All chemicals used in this study were of analytical grade. The TiO₂ nanoparticles and PVDF polymer were selected due to their excellent compatibility for membrane fabrication and modification, while identical material compositions were maintained

throughout all experiments to ensure reproducibility.

PVDF TiO₂ composite membrane were fabricated using the Non-Solvent Induced Phase Separation (NIPS) method (Benhabiles et al., 2019). A total of 0.4 g of PVDF was dissolved in a solvent mixture consisting of 4 mL NMP, 2 mL acetone, and 0.5 mL glycerol, followed by stirring using a magnetic stirrer at 60 °C for 30 minutes until a homogeneous solution was obtained (Liu et al., 2022). Subsequently, TiO₂ nanoparticles were added to the solution at varying concentrations of 0%, 5%, 10%, and 15% relative to the mass of PVDF. The mixture was further stirred at 60 °C for 30 minutes and then stirred without heating for an additional 30 minutes to ensure uniform nanoparticle dispersion and to minimize the formation of air bubbles in the solution (Qin et al., 2025).

To maintain consistent fabrication conditions, each membrane dope solution was prepared following the same stirring time, temperature, and solvent composition. The prepared solution was allowed to stand briefly before casting to reduce the presence of residual air bubbles that could affect membrane morphology.

The resulting homogeneous dope solution was cast onto a glass substrate using the casting method and immediately immersed in a deionized water coagulation bath (Ilyas et al., 2020). The solvent-nonsolvent exchange during coagulation induced phase separation, leading to the formation of a porous membrane structure. The formed membrane was allowed to undergo coagulation for 10 minutes, after which it was washed and stored in deionized water prior to characterization (Salahshoori et al., 2024).

All membranes were fabricated under identical laboratory conditions to minimize experimental variation during the phase inversion process and to ensure consistent membrane formation.

Membrane hydrophilicity was characterized by contact angle measurements using a contact angle analyzer. The test was performed by placing a droplet of deionized water onto the membrane surface using a micropipette, after which the angle formed between the water droplet and the membrane surface was measured. The contact angle value was used as an indicator of membrane hydrophilicity, where a lower contact angle corresponds to higher surface wettability (Du et al., 2020). Conversely, a higher contact angle indicates a more hydrophobic surface. The effect of TiO₂ incorporation on membrane hydrophilicity was evaluated based on changes in contact angle values across different nanoparticle concentrations (Chamam et al., 2025). Contact angle measurements were performed at several locations on each membrane surface, and the average value was used to represent the hydrophilicity of each membrane composition.

Membrane stability was evaluated through repeated filtration tests using a vacuum filtration system with a Methylene Blue feed solution at a concentration of 2.5 ppm. Each membrane was used to filter 50 mL of feed solution for three consecutive filtration cycles without membrane replacement, simulating repeated operational conditions (Zhang et al., 2022). For each cycle, the permeate volume and filtration time were recorded to determine the membrane flux. Changes in flux during the experiments were used as an indicator of membrane performance stability and fouling behavior. A membrane with high stability

is characterized by a relatively small decline in flux over repeated filtration cycles (Qin et al., 2025).

All experimental data were analyzed descriptively by comparing the hydrophilicity and operational stability of membranes containing different TiO₂ concentrations. The results were presented in the form of graphs to illustrate the influence of TiO₂ incorporation on membrane performance.

Furthermore, the relationship between hydrophilicity and membrane stability was analyzed by correlating contact angle values with the ability of the membrane to maintain its filtration performance during operation. Accordingly, the effect of TiO₂ nanoparticle incorporation on enhancing hydrophilicity and improving membrane resistance to fouling was comprehensively evaluated (Goh et al., 2019).

RESULTS AND DISCUSSION

Hydrophilicity and stability are two interrelated parameters that determine membrane performance during the filtration process. In this study, the effect of TiO₂ nanoparticle incorporation on both parameters was evaluated through contact angle measurements and stability testing using repeated filtration of a Methylene Blue solution. The contact angle value was used to represent membrane hydrophilicity, whereas stability was assessed based on relative efficiency values, which indicate the membrane's ability to maintain its filtration performance over multiple operational cycles.

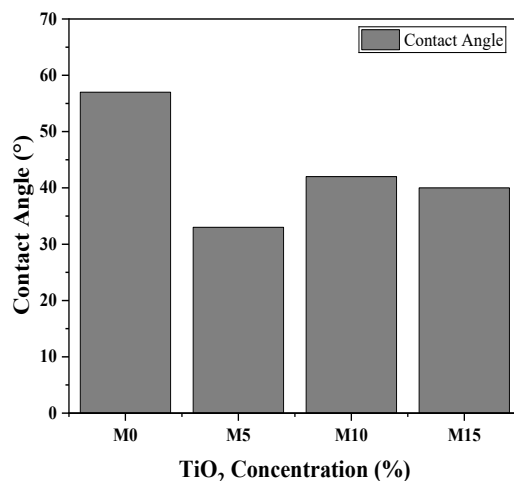


Figure 1. Water contact angles of PVDF-TiO₂ membranes with different TiO₂ concentrations: (M0) 0% TiO₂, (M5) 5% TiO₂, (M10) 10% TiO₂, and (M15) 15% TiO₂ (Kurniawan et al., 2026)

The data presented in Figure 1 were reproduced from our previous study (Kurniawan et al., 2026), Figure 1 illustrates the effect of TiO₂ concentration on the contact angle of PVDF membranes. As shown in the figure, the incorporation of TiO₂ significantly influenced membrane hydrophilicity. The pristine PVDF membrane (M0) exhibited a contact angle of 57°, whereas membranes containing 5%, 10%, and 15% TiO₂ showed contact angles of 33°, 42°, and 40°, respectively (Abba et al., 2021). The reduction in contact angle observed for all TiO₂-incorporated membranes indicates that the presence of TiO₂ nanoparticles effectively enhanced membrane hydrophilicity compared with the pristine PVDF membrane.

The most pronounced reduction in contact angle was observed for the M5 membrane, which exhibited a value of 33°. This result indicates that the incorporation of 5% TiO₂ represents the optimum condition for enhancing membrane surface wettability. The improvement in hydrophilicity can be attributed to the presence of hydroxyl (–OH) groups on the surface of TiO₂ nanoparticles, which are polar in nature and capable of forming hydrogen bonds

with water molecules. When TiO₂ nanoparticles are uniformly dispersed within the PVDF matrix, the membrane surface energy increases, allowing water molecules to spread more readily across the membrane surface and consequently producing a lower contact angle. These findings are consistent with those reported by (Zhang et al., 2022), who demonstrated that the incorporation of TiO₂ into PVDF membranes enhances hydrophilicity by increasing surface energy and providing a greater number of hydrophilic sites on the membrane surface. Nevertheless, increasing the TiO₂ concentration to 10% and 15% resulted in a rise in contact angle to 42° and 40°, respectively (Abba et al., 2021).

The results further indicate that membrane hydrophilicity is not solely determined by the intrinsic hydrophilic nature of TiO₂ nanoparticles but also by their dispersion within the PVDF matrix. Uniform dispersion provides a larger number of accessible hydrophilic sites on the membrane surface, facilitating stronger interactions with water molecules. Consequently, the membrane surface exhibits improved wettability, as reflected by the lower contact angle observed for the M5 membrane. These findings suggest that both nanoparticle

concentration and dispersion quality play essential roles in determining membrane hydrophilicity.

This phenomenon indicates that the enhancement of hydrophilicity does not increase linearly with TiO₂ loading. The observed behavior is likely attributed to nanoparticle agglomeration at higher concentrations (Gayatri et al., 2023). Such agglomeration reduces the effective exposure of hydrophilic TiO₂ groups on the membrane surface, thereby diminishing their contribution to hydrophilicity enhancement (Zeng et al., 2024). Furthermore, non-uniform nanoparticle distribution may lead to surface structural irregularities that adversely affect membrane wettability. Similar findings were reported by (Aldana et al., 2024), who observed that TiO₂ concentrations exceeding the optimum level can promote nanoparticle aggregation, consequently reducing the effectiveness of membrane surface modification. Numerous studies have investigated the enhancement of PVDF membrane hydrophilicity through the incorporation of TiO₂ nanoparticles. One study demonstrated that surface modification of PVDF membranes via

TiO₂ coating significantly reduced the water contact angle from 127.1° to 114.3°, indicating an improvement in membrane surface hydrophilicity due to the presence of hydroxyl groups on TiO₂ that interact favorably with water molecules (Penboon et al., 2019). These findings suggest that TiO₂-based surface modification is an effective approach for improving membrane wettability.

Another study reported that the incorporation of TiO₂ into PVDF membranes via the phase inversion method significantly enhanced membrane hydrophilicity, as evidenced by a reduction in the water contact angle to as low as 43.8° at a specific TiO₂ concentration. This improvement was accompanied by a substantial increase in water flux, indicating that membrane hydrophilicity is directly correlated with water transport performance (Lee et al., 2016). Furthermore, the increased abundance of surface hydroxyl (-OH) groups resulting from the presence of TiO₂ contributes to stronger interactions between water molecules and the membrane surface.

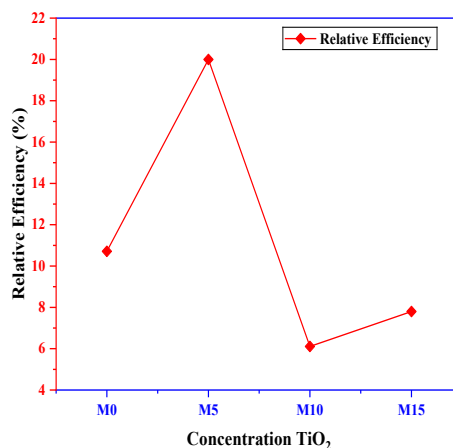


Figure. 2 Relative efficiency of PVDF–TiO₂ membranes with different TiO₂ concentrations: (M0) 0% TiO₂, (M5) 5% TiO₂, (M10) 10% TiO₂, and (M15) 15% TiO₂.

The incorporation of TiO₂ significantly affected membrane stability during the filtration process. Figure 2 presents the relative efficiency values of the

fabricated membranes following repeated filtration tests using a Methylene Blue solution. The results demonstrate that TiO₂-modified membranes exhibited

superior operational stability compared with the pristine PVDF membrane. Among all samples, the M5 membrane achieved the highest relative efficiency of 20.00%, indicating the greatest ability to maintain filtration performance during repeated operation. In contrast, the pristine PVDF membrane (M0) exhibited a relative efficiency of only 10.71%. Meanwhile, the M10 and M15 membranes showed relative efficiency values of 6.11% and 7.80%, respectively.

The improvement in the stability of TiO₂-modified membranes is closely associated with the enhanced hydrophilicity of the membrane surface. A more hydrophilic surface has a greater ability to attract water molecules, resulting in the formation of a stable hydration layer on the membrane surface. This hydration layer acts as a physical barrier that reduces direct contact between Methylene Blue molecules and the membrane surface. Consequently, dye adsorption and pore blockage can be minimized, leading to a lower fouling formation rate. With reduced fouling, flux decline during the filtration process can be suppressed, allowing the membrane to maintain its performance over a longer operational period. These findings are consistent with the study reported by (Liu et al., 2022), which demonstrated that the increase in hydrophilicity resulting from TiO₂ incorporation enhanced membrane fouling resistance through the formation of a more stable water layer on the membrane surface.

Interestingly, although the M10 and M15 membranes still exhibited better hydrophilic properties than the membrane without TiO₂, their stability was lower than that of the M5 membrane. This phenomenon indicates that enhanced hydrophilicity alone is insufficient to guarantee improved stability when the concentration of incorporated TiO₂ is excessively high. At higher concentrations, nanoparticle

agglomeration may lead to a less homogeneous pore distribution and increase the likelihood of defect formation within the membrane structure. Such conditions make certain pores more susceptible to blockage during the filtration process, resulting in a greater rate of flux decline. These findings are consistent with the study reported by (Jang et al., 2024), which demonstrated that excessively high TiO₂ concentrations can reduce membrane structural homogeneity and negatively affect filtration performance stability.

These observations indicate that increasing TiO₂ concentration beyond the optimum level does not necessarily improve membrane performance. Instead, excessive nanoparticle incorporation may offset the beneficial effects of enhanced hydrophilicity due to structural heterogeneity caused by nanoparticle agglomeration. Therefore, optimization of TiO₂ concentration is essential to achieve the best compromise between membrane hydrophilicity and operational stability.

Overall, the results demonstrate a strong relationship between membrane hydrophilicity and stability. Membranes with lower contact angle values tended to exhibit better stability during the filtration process. This relationship was clearly observed in the M5 membrane, which possessed the lowest contact angle and simultaneously achieved the highest relative efficiency value. The incorporation of TiO₂ was proven to enhance the performance of PVDF membranes through improved hydrophilicity, the formation of a more stable hydration layer, and a reduced tendency for fouling during filtration. However, increasing the TiO₂ concentration beyond the optimum level led to nanoparticle agglomeration, which diminished the effectiveness of membrane modification. Therefore, a TiO₂ concentration of 5% can be considered the optimum condition,

providing the best balance between enhanced hydrophilicity and operational stability of the membrane in the removal of Methylene Blue. In addition to improving hydrophilicity, the incorporation of TiO₂ was also found to enhance the structural and operational stability of PVDF membranes.

Compared with previous studies that mainly focused on improving membrane permeability or antifouling performance, this study demonstrates that the optimum TiO₂ concentration should be determined by simultaneously considering membrane hydrophilicity and operational stability. The present findings reveal that the incorporation of 5% TiO₂ provides the most favorable balance between surface wettability and filtration stability. This information provides a useful reference for the design and optimization of PVDF–TiO₂ composite membranes for dye wastewater treatment applications.

The interaction between TiO₂ nanoparticles and the PVDF polymer matrix results in stronger interfacial bonding, making the membrane more resistant to degradation during repeated filtration processes (Gayatri et al., 2023). This finding indicates that the presence of TiO₂ not only enhances the initial membrane performance but also contributes to maintaining its long-term stability.

CONCLUSION

The incorporation of TiO₂ nanoparticles had a significant effect on stability of PVDF membranes fabricated using the Non-Solvent Induced Phase Separation (NIPS) method. Membrane modification with TiO₂ successfully enhanced the hydrophilic properties of the membrane surface, as evidenced by the reduction in contact angle values compared with the pristine PVDF

membrane. The membrane containing 5% TiO₂ exhibited the highest hydrophilicity, with the lowest contact angle value of 33°, indicating improved surface wettability.

The enhancement in hydrophilicity contributed to improved membrane stability during the filtration of Methylene Blue by reducing the tendency for fouling. The membrane containing 5% TiO₂ achieved the highest relative efficiency of 20.00%, demonstrating the greatest ability to maintain filtration performance over repeated operational cycles. In contrast, increasing the TiO₂ concentration to 15% did not result in a proportional improvement in performance due to nanoparticle agglomeration, which reduced the effectiveness of hydrophilic sites and compromised the structural homogeneity of the membrane.

Overall, the results demonstrate that a TiO₂ concentration of 5% represents the optimum condition for enhancing both the hydrophilicity and stability of PVDF membranes. These findings confirm that the modification of PVDF membranes with TiO₂ nanoparticles is an effective strategy for improving membrane resistance to fouling and extending operational stability, thereby highlighting its potential application in the treatment of dye-contaminated wastewater.

This study contributes to the development of PVDF–TiO₂ composite membranes by demonstrating that the optimum TiO₂ concentration should be determined not only based on enhanced membrane hydrophilicity but also by considering its influence on operational stability during repeated filtration. The findings provide a more comprehensive understanding of the relationship between TiO₂ concentration, membrane surface properties, and filtration performance, thereby serving as a useful reference for the optimization of

polymeric membranes for wastewater treatment applications.

From a practical perspective, the optimized PVDF–TiO₂ membrane developed in this study has the potential to improve the efficiency and durability of membrane-based dye wastewater treatment systems by minimizing membrane fouling and maintaining stable filtration performance.

Future studies are recommended to investigate the long-term operational performance of PVDF–TiO₂ membranes under continuous filtration conditions and to evaluate their effectiveness in treating real industrial wastewater containing more complex contaminant compositions. Furthermore, additional characterization techniques, such as membrane porosity, mechanical strength, and surface morphology analyses, could provide a more comprehensive understanding of the influence of TiO₂ incorporation on membrane performance.

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