



Fabrication and Performance Evaluation of PVDF/Nano-TiO₂ Membrane via NIPS Method for Tetracycline Removal Using Vacuum Filtration.

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ABSTRACT

Water pollution caused by tetracycline contamination is a serious issue due to its poor biodegradability and potential to trigger antimicrobial resistance. This study aims to evaluate the filtration performance (permeate flux and rejection efficiency) of a PVDF membrane modified with 10% nano-TiO₂ fabricated via non-solvent induced phase separation (NIPS) under vacuum filtration for tetracycline removal. Polyvinylidene fluoride (PVDF) membranes modified with 10% (wt) nano-TiO₂ were cast using the NIPS method and processed using a vacuum filtration system at 200 mbar. The incorporation of nano-TiO₂ may improve membrane hydrophilicity and pore formation, as indicated by SEM morphological changes and supported by previous studies. The morphology exhibits a distinct asymmetric structure, consisting of a thin, dense upper layer and a lower layer dominated by sponge-like pore structures. The modified membrane demonstrated a very high permeate flux ranging from 312.5 to 8,375.2 L·m⁻²·h⁻¹·bar⁻¹, while tetracycline rejection ranged from 29.31% to 55.81%. The high flow rate indicates that the system can manage wastewater contaminants on a large scale. However, further optimization of the molecular sieve separation system is required to improve rejection. Overall, PVDF modification with nano-TiO₂ has proven effective in boosting filtration productivity and holds potential for treating tetracycline-contaminated wastewater.

1. INTRODUCTION

The frequent direct discharge of wastewater into water bodies has led to a drastic decline in water quality in various regions around the world; this is largely due to industrial activities and the lack of adequate wastewater treatment (Jalloul et al., 2024).

One of the contaminants is tetracycline; tetracycline is a potent and persistent antibiotic, which has become a global concern due to its effects on the environment and its potential to trigger microbial resistance (Z. Lu et al., 2024). Unfortunately, conventional wastewater treatment plants are not very effective at removing pharmaceutical contaminants such as tetracycline, so residues from this wastewater will persist in aquatic environments (Anggryanti et al., 2025). This situation is further exacerbated by the continuous discharge of

untreated industrial wastewater, leading to rising pollution levels that can cause substantial damage to aquatic ecosystems. (Kurniawan & Mariadi, 2019). Exposure to tetracycline not only pollutes the environment; from an ecological perspective, this contaminant can also disrupt soil microflora, which may lead to ecosystem imbalances and ultimately cause harm in many areas, including human health (Manoharan et al., 2023).

One solution to the limitations of these conventional methods is membrane technology, which offers greater efficiency and selectivity while consuming less energy than standard conventional methods (Aprianti et al., 2025). However, the use of polymer membranes such as polyvinylidene fluoride (PVDF) often still faces challenges in the form of fouling, which can lead to a decline in filtration performance and shorten the membrane's service life (J.

Zhang et al., 2022). The addition of TiO₂ nanoparticles may offer a solution, as they have been shown to enhance the hydrophilicity of the membrane, thereby improving its resistance to fouling and effectively refining the pore structure of the PVDF membrane (Rahman & Sulistyowati, 2023). To achieve effective results from the PVDF-based membrane technology, it can be combined with vacuum filtration, where the efficiency of the separation process can be observed through the increase in flow rate during the process (Prastyo & Sri Rahayoe, 2018).

Controlling the pressure variable significantly affects the performance of the vacuum filtration system, as pressure is a critical factor that can influence the flow rate of water passing through the membrane and the membrane's ability to retain contaminant molecules (Kurniawan et al., 2023). Therefore, evaluating the balance between membrane flux and rejection performance is important for assessing the suitability of PVDF/TiO₂ membranes for tetracycline removal at different feed concentrations. (Bhernama et al., 2023)

2. METHOD

2.1 Materials and Membrane Preparation

The membrane synthesis protocol used in this study was developed based on previously reported procedures by (S. Lu & Zou, 2025), with several modifications to accommodate the objectives and experimental conditions of the present study. The process began with the preparation of the membrane-forming materials, including 0.4 g of PVDF (Base Polymer), 4 mL of NMP (Solvent), 2 mL of acetone (Co-solvent), and 0.50 mL of glycerol Pore former. TiO₂ (Additive) nanoparticles were incorporated at a fixed concentration of 10% (w/w) relative to the total weight of PVDF to prepare the PVDF/TiO₂ composite membrane. The dope solution was prepared through a two-step thermal process, where PVDF was initially dissolved with NMP, acetone, and glycerol in a sealed glass bottle at 60 °C for 30 minutes under constant magnetic stirring until a homogeneous solution was formed. Subsequently, TiO₂ nanoparticles were added to the mixture and stirred again at 60 °C for 30 minutes, followed by additional stirring without heating for 30 minutes at room temperature to ensure suspension stability and to obtain a homogeneous dope solution.

2.2 Vacuum Filtration Process

In the vacuum filtration process, a tetracycline solution was prepared with an initial concentration of 500 ppm and then diluted to obtain feed solution concentrations of 2.5 ppm, 5 ppm, 7.5 ppm, and 10 ppm. Prior to filtration, a pre-filtration step is performed using 100 ml of distilled water to ensure the membrane is of good quality and suitable for vacuum filtration. Subsequently, the filtration process was conducted using 100 mL of tetracycline feed solution at a vacuum pressure of 200 mbar (pressure difference). The flat-sheet membrane with an effective area of 0.001194 m² was installed in the filtration cell and pre-filtered using distilled water before testing. The filtration was performed at room temperature for five sequential cycles using the same membrane, with 5 mL of permeate collected from each cycle for UV-Vis analysis.

After vacuum filtration, 5 ml of permeate from each of the 5 experimental cycles was collected to determine the maximum wavelength of tetracycline, which was

subsequently used as the basis for calculating the membrane rejection percentage.

This vacuum technique is commonly used in laboratories to improve liquid-solid separation by utilizing pressure differences to accelerate the filtration rate at the feed inlet, thereby producing a clean filtrate through a more efficient process (F. Zhang et al., 2016).

The flux and rejection values are calculated using the following equations:

$$J_w = \frac{V}{A \cdot t \cdot \Delta P} \quad (1)$$

$$\%R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (2)$$

In Equation (1), (J_w) represents the pure water permeability, (V) is the volume of water in liters (L), (A) is the membrane surface area, (t) is the filtration time in hours, and (ΔP) is the applied pressure in millibar (mbar). In Equation (2), (C_p) represents the concentration of the substance in the permeate, while (C_f) denotes the concentration of the substance in the feed solution.

2.3 Performance Evaluation

Performance evaluation in this study utilized scanning electron microscopy (SEM), which is an analytical technique that allows for detailed analysis of the membrane's cross-section, which is crucial for identifying potential structural defects during the membrane manufacturing process. The assessment was based on the morphological properties of the membrane, including its surface topography and internal pore structure. This characterization is essential for visually identifying modifications and understanding how pore distribution affects filtration rate and overall efficiency (Sun et al., 2020).

The evaluation of membrane selectivity was based on the membrane's efficiency in rejecting tetracycline contaminants, through a combination of physicochemical mechanisms involving the membrane surface and tetracycline molecules (J. Zhang et al., 2023). At this stage, a trade-off often occurs, whereby increasing the flow rate frequently results in a decrease in the membrane's efficiency in rejecting contaminants (Indriyani et al., 2017). The tetracycline concentration was analyzed using a UV-Vis spectrophotometer. The maximum absorption wavelength (λ_{max}) was determined at 357 nm based on tetracycline solutions with different concentrations (2.5, 5, 7.5, and 10 ppm). A calibration curve was established to correlate absorbance values with tetracycline concentrations. The obtained calibration equation was then used to calculate the remaining tetracycline concentration in each permeate sample after filtration at different initial feed concentration. This method was chosen due to its effectiveness in achieving high accuracy for real-time monitoring of antibiotics in wastewater, and the sensitivity of UV-Vis spectroscopy in providing highly sensitive detection limits (Li et al., 2022).

Membrane performance is analyzed based on the permeate flow rate and tetracycline rejection efficiency, where the permeate flow rate is the volume of fluid or contaminants that can pass through the membrane surface per unit of time, which is influenced by the membrane characteristics and the applied

pressure. Membrane rejection efficiency is the membrane's ability to reject contaminants, a decrease in flow rate during the filtration process is often caused by fouling and concentration polarization (Quezada et al., 2021). High flow rate is a key efficiency parameter; a high value indicates the membrane's ability to produce permeate optimally (As'adah & Munasir, 2023)

3. RESULTS AND DISCUSSION

3.1 Membrane Morphological Characterization

Scanning electron microscopy (SEM) results (Figure 1) of PVDF membranes doped with 10% nano-TiO₂ and fabricated using the Non-solvent Induced Phase Separation (NIPS) method show that the cross-sectional morphology exhibits a distinct asymmetric structure, consisting of a thin, dense upper layer and a lower layer dominated by sponge-like pore structures

The addition of 10% nano-TiO₂ to the PVDF membrane significantly alters the membrane's internal structure. The hydrophilic nature of TiO₂ accelerates the exchange rate between the solvent and non-solvent during the membrane casting process, resulting in a more elongated pore structure compared to PVDF membranes without added nano-TiO₂. On the membrane surface, SEM images confirm that TiO₂ nanoparticles are successfully incorporated and embedded within the PVDF matrix, contributing to increased surface roughness and enhanced hydrophilicity which improves membrane performance. TiO₂ evenly dispersed at this concentration provides a layer density for optimal contaminant rejection and a balance between pore structure to enhance permeability.

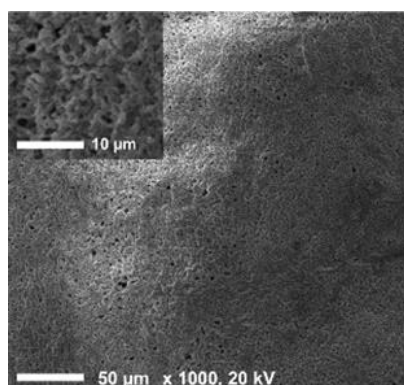


Figure 1. Scanning electron microscopy (SEM) images of the 10% PVDF-TiO₂ membrane, reproduced from (Kurniawan et al., 2026) with permission: the upper section shows the membrane surface morphology, while the lower section reveals a cross-sectional structure dominated by interconnected sponge-like pore structures.

3.2 UV-Vis Spectrophotometric Analysis

The assessment of tetracycline contamination in this study utilized UV-Vis spectrophotometry within the wavelength range of 200–800 nm. The measurement results indicate that tetracycline has a maximum absorption wavelength (λ_{max}) of approximately 357 nm, which is consistent with the findings of the study by (González-Garrido et al., 2025), which found a maximum absorption wavelength of 359 nm under acidic conditions and a wavelength of 362 nm corresponding to an increase in the pH level of the contaminant. During the

filtration stage, the contaminant had four concentration levels: 2.5 ppm, 5 ppm, 7.5 ppm, and 10 ppm. This was done to determine the relationship between solution concentration and the absorbance value of tetracycline when analyzed by UV-Vis spectrophotometry; this aligns with Beer-Lambert's law, with a correlation coefficient (R^2) approaching 1.

The UV-Vis absorption results for each concentration are clearly presented in Figures 2A–2D. At the lowest concentration of 2.5 ppm (Figure 2A), the absorption intensity was the lowest, yet it still exhibited a long, sharp peak at 357 nm. At increased concentrations of 5 ppm (Figure 2B) and 7.5 ppm (Figure 2C), the peak intensity consistently increased, indicating higher tetracycline concentrations in the contaminated solutions; at a concentration of 10 ppm (Figure 2D), the highest peak was produced where absorbance reaches its maximum within the tested standard range, without any bathochromic wavelength shifts or hypochromic intensity changes. Figures 2A–2D show consistent λ_{max} positions across all concentrations, indicating spectral consistency and compliance with Beer-Lambert behavior without detectable concentration-dependent spectral shifts under the measurement conditions in the tested tetracycline-containing solutions.

These results indicate that any change in the concentration of tetracycline contaminants that have undergone vacuum filtration can be detected with high precision, consistent with the findings by (Kurniawan et al., 2026), who observed a significant decrease in the peak absorbance intensity at 357 nm following vacuum filtration using a PVDF-TiO₂ membrane, indicating the effectiveness of the active membrane layer in removing tetracycline molecules; this decrease in intensity served as the basis for calculating the rejection rate, which yielded moderate values ranging from 29.31% to 55.81%.

3.3 Permeate Flux and Rejection Efficiency

The selection of a PVDF membrane modified with 10% TiO₂ was based on the results of a study by (Kurniawan et al., 2026). In membranes with this concentration, the nanoparticles are evenly distributed throughout the membrane matrix and are effective at filling the microporous structure of the membrane without causing mechanical failure due to agglomeration.

The PVDF-TiO₂ membrane with a 10% TiO₂ content exhibited high permeate flux values that varied depending on the feed concentration and the vacuum filtration cycle; at a concentration of 2.5 ppm (Figure 3A), the lowest permeate flux was recorded in cycle 1 at $312.5 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$, and the highest in cycle 2 at $1,107.83 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$. At a concentration of 5 ppm (Figure 3B), an increase in results was observed in cycle 1, reaching 2,684.36, followed by a decrease in cycle 5 to 802.22. At a concentration of 7.5 ppm (Figure 3C), the permeate flux in cycle 1 reached 8,375.2 $\text{L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$. This value was obtained from the calculated flux based on the measured permeate volume, effective membrane area, and filtration time under the applied vacuum pressure condition. In cycles 4 and 5, a decrease occurred to 6754.2, and at a concentration of 10 ppm (Figure 3D), the highest permeate rate was in cycle 1 at $3172.42 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$ and the lowest was in cycle 5 at $1046.9 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$.

The flux values obtained in this study were very high, supporting the theory that the addition of TiO₂ in the PVDF membrane fabrication process using the NSIP method can

accelerate the exchange between the solvent and non-solvent, thereby significantly improving the membrane's hydrophilicity and porosity, resulting in a membrane with open and well-connected pores. Additionally, the relatively small membrane surface area (0.001194 m^2) contributes to the high increase in flux values, consistent with the findings of (Kurniawan et al., 2026)

As the number of filtration cycles increased, a continuous decline in permeate flux was observed. This flux decline indicates potential performance loss, which may be attributed to membrane fouling, concentration polarization, or structural membrane compaction, this was due to the accumulation of tetracycline contaminant molecules on the membrane surface, while the rejection rate remained moderate and decreased as the solution concentration increased. At 2.5 ppm (Figure 3A), rejection ranged from 51.76% to 55.81%; at 5 ppm (Figure 3B), it ranged from 35.69% to 40.15%. At a concentration of 7.5 ppm (Figure 3C), the rejection rate fluctuated between

29.31% and 46.07%, while at a concentration of 10 ppm (Figure 3D), the rejection rate remained relatively stable in the range of 36.63%-43.83%. This decrease in rejection may be associated with the higher tetracycline concentration, which can affect membrane interactions through mechanisms such as adsorption, fouling, and concentration polarization. In this study, moderate tetracycline rejection rates ranging from 29.31% to 55.81% were observed; and these moderate rejection results are consistent with findings in a study conducted by (Duarte et al., 2022), where the efficiency of removing antibiotics (such as oxytetracycline) using membranes without external activation generally remained below 60%. This indicates that adsorption interactions on the membrane surface and rejection mechanisms are predominantly governed by physical barrier effects; thus, the data from this study provide a basis for characterizing the intrinsic performance of PVDF-TiO₂ membranes against micro-organic pollutants.

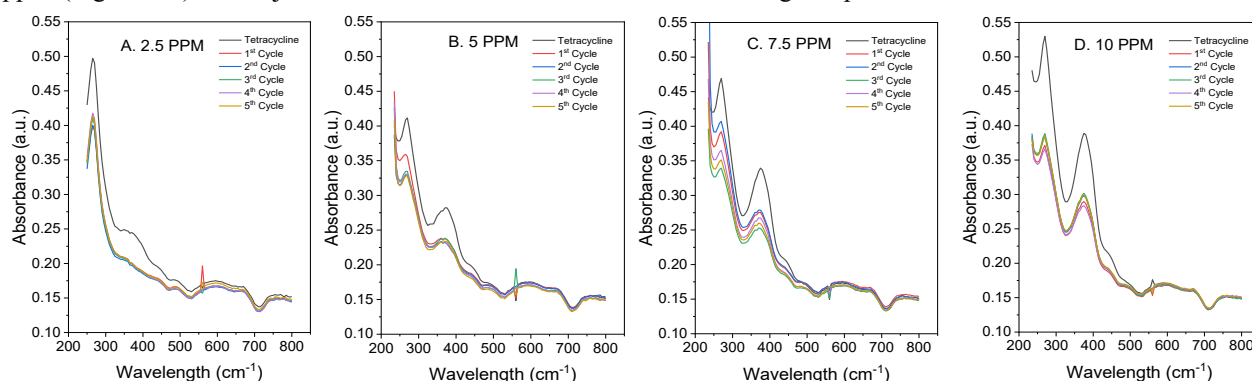


Figure 2. UV-Vis absorbance spectra of tetracycline permeate samples after the vacuum filtration process using PVDF-TiO₂ (10%) membrane. The spectra were obtained from permeate samples collected at different tetracycline feed concentrations: (A) 2.5 ppm, (B) 5 ppm, (C) 7.5 ppm, and (D) 10 ppm. The x-axis represents wavelength (nm), and the y-axis represents absorbance (a.u.). The spectra represent raw UV-Vis measurements without baseline correction.

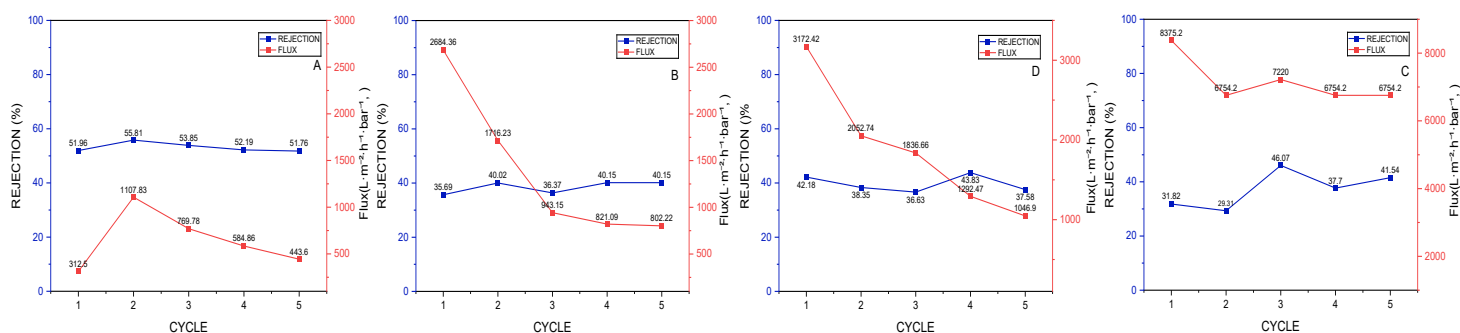


Figure 3 Rejection and permeability performance of PVDF-TiO₂ (10%) membrane at different tetracycline feed concentrations: (A) 2.5 ppm, (B) 5 ppm, (C) 7.5 ppm, and (D) 10 ppm. Rejection and permeability values are presented in %, and $\text{L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$, respectively. The plotted values correspond to the numerical data reported in the manuscript.

4. CONCLUSION

Main Findings

In this study, PVDF membranes modified with 10% nano-TiO₂, were evaluated using vacuum filtration to remove soluble tetracycline contaminants. SEM analysis showed that the membrane has a cross-sectional morphology with a distinct asymmetric structure consisting of a thin, dense top layer and a porous bottom layer characterized by finger-shaped

macroporosity. In UV-Vis spectrophotometry, the maximum wavelength of tetracycline was at 357 nm. The flux results showed high values ranging from $312.5 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$ to $8,375.2 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$; conversely, the tetracycline rejection efficiency was in the moderate range, varying between 29.31% and 55.81%.

Significance and Relevance

The strength of this study lies in the ability of the PVDF-TiO₂ membrane to achieve high filtration performance under

laboratory-scale conditions. The incorporation of TiO₂ nanoparticles into the PVDF membrane fabricated using the NIPS method may contribute to the formation of an open-pore structure, which can enhance membrane performance. However, further studies involving real wastewater, long-term operation, membrane stability, cleaning procedures, scale-up evaluation, and economic analysis are required to assess its practical application.

Limitations of the Study

The main limitation identified in this study is that the tetracycline rejection performance under synthetic feed conditions remained moderate. The rejection values suggest that further optimization of membrane properties and operating conditions is required to improve tetracycline retention.

Recommendations for Further Research

For future research, it is recommended to conduct experiments using membranes with varying surface areas to assess the stability of flow rates, as well as to optimize membrane layer thickness for rejection efficiency and to employ additional treatment methods necessary to balance high flow rates with improved membrane rejection efficiency, thereby achieving optimal filtrate quality.

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