

Reduction of Iron (Fe) in Environmental Engineering Chemistry Laboratory Liquid Waste Using Membrane Distillation

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Abstract

Laboratory wastewater generated from chemical analysis activities contains hazardous pollutants that require proper treatment before discharge. Preliminary analysis of wastewater from the Environmental Engineering Chemistry Laboratory, Universitas PGRI Adi Buana Surabaya, indicated that the iron (Fe) concentration exceeded the maximum permissible limit specified in the Indonesian Minister of Health Regulation No. 2 of 2023 concerning Environmental Health. This study aimed to evaluate the effectiveness of vacuum membrane distillation technology in reducing Fe concentration using different membrane materials and pore sizes. Three membrane configurations were investigated: (A) Polytetrafluoroethylene (PTFE) with a pore size of 0.22 μm , (B) PTFE with a pore size of 0.45 μm , and (C) Polyvinylidene fluoride (PVDF) with a pore size of 0.45 μm . The experimental results demonstrated that both PTFE membranes exhibited superior performance, achieving Fe removal efficiencies of 75.4% and 74.6% for the 0.22 μm and 0.45 μm membranes, respectively. These treatments reduced the initial Fe concentration of approximately 0.4 ppm to 0.10–0.12 ppm, meeting the maximum allowable concentration of 0.2 ppm established by the Indonesian Minister of Health Regulation No. 2 of 2023. In comparison, the PVDF membrane showed a lower removal efficiency of 50.5%, producing a final Fe concentration of 0.215 ppm, slightly above the regulatory limit. These findings indicate that PTFE membranes, particularly the 0.22 μm membrane, are more effective than PVDF membranes for Fe removal and demonstrate the potential of vacuum membrane distillation as an environmentally friendly technology for treating laboratory wastewater.

Keywords: Membrane Distillation, Fe, PTFE, PVDF.

Abstrak

Air limbah laboratorium yang dihasilkan dari kegiatan analisis kimia mengandung polutan berbahaya yang memerlukan pengolahan sebelum dibuang ke lingkungan. Hasil analisis awal terhadap air limbah Laboratorium Kimia Teknik Lingkungan Universitas PGRI Adi Buana Surabaya menunjukkan bahwa konsentrasi besi (Fe) melebihi baku mutu maksimum yang ditetapkan dalam Peraturan Menteri Kesehatan Republik Indonesia Nomor 2 Tahun 2023 tentang Kesehatan Lingkungan. Penelitian ini bertujuan untuk mengevaluasi efektivitas teknologi Vacuum Membrane Distillation (VMD) dalam menurunkan konsentrasi Fe menggunakan variasi material membran dan ukuran pori yang berbeda. Tiga konfigurasi membran yang diuji meliputi: (A) membran Polytetrafluoroethylene (PTFE) dengan ukuran pori 0,22 μm , (B) membran PTFE dengan ukuran pori 0,45 μm , dan (C) membran Polyvinylidene Fluoride (PVDF) dengan ukuran pori 0,45 μm . Hasil penelitian menunjukkan bahwa kedua membran PTFE memiliki kinerja yang lebih baik, dengan efisiensi penyisihan Fe masing-masing sebesar 75,4% dan 74,6% untuk membran berukuran pori 0,22 μm dan 0,45 μm . Perlakuan tersebut berhasil menurunkan konsentrasi awal Fe sekitar 0,4 ppm menjadi 0,10–0,12 ppm, sehingga memenuhi baku mutu maksimum sebesar 0,2 ppm sesuai Peraturan Menteri Kesehatan Republik Indonesia Nomor 2 Tahun 2023. Sebaliknya, membran PVDF menunjukkan efisiensi penyisihan yang lebih rendah, yaitu sebesar 50,5%, dengan konsentrasi Fe akhir sebesar 0,215 ppm, yang masih sedikit di atas baku mutu yang dipersyaratkan. Hasil penelitian ini menunjukkan bahwa membran PTFE, khususnya dengan ukuran pori 0,22 μm , lebih efektif dibandingkan membran PVDF dalam menghilangkan Fe, serta menunjukkan potensi teknologi Vacuum Membrane Distillation (VMD) sebagai metode yang ramah lingkungan untuk pengolahan air limbah laboratorium.

Keywords: Membran Distilasi, Fe, PTFE, PVDF.

1. Introduction

In many Environmental Engineering laboratories, the management of chemical residues from experiments remains inadequate. Typically, chemicals used during laboratory activities are directly discharged as liquid waste without prior treatment. Although the volume of such waste is relatively small, it is categorized as hazardous and toxic (B3) waste [1]. A study conducted at the Environmental Engineering Chemistry Laboratory of PGRI Adi Buana University, Surabaya, revealed that the laboratory's liquid waste contained 0.4 ppm of Fe. These concentrations exceed the permissible limits set by the Indonesian Minister of Health Regulation No. 2 of 2023 on Environmental Health. Without proper treatment, this liquid waste has the potential to contaminate the surrounding environment.

The findings highlight the urgency of implementing an appropriate and practical treatment system to reduce iron content in laboratory wastewater. Considering the diverse educational backgrounds of community members and laboratory users, it is essential to apply a simple technology transfer approach that is easy to understand and operate. Such an approach should not only address environmental concerns but also produce useful and applicable outcomes that can support daily needs, fostering awareness and participation in sustainable environmental practices (Febrianti et al., 2025).

In recent years, membrane technology has emerged as a promising solution for wastewater treatment, particularly for domestic effluents. This method is capable of effectively reducing pollutant concentrations in wastewater with high efficiency. Its key advantages include low energy consumption, the absence of additional chemicals during operation—thus avoiding secondary waste generation—simple operation and maintenance, and minimal land requirements. Owing to these benefits, membrane-based systems are increasingly viewed as a viable alternative for large-scale wastewater treatment [3].

One of the most advanced applications of membrane technology is membrane distillation (MD), a hybrid thermal process that utilizes the vapor pressure difference between hot and cold streams separated by a hydrophobic membrane. In this process, water vapor passes through the membrane and condenses on the cooler side, producing purified or distilled water [4]. The hydrophobic membranes typically employed in MD systems are made from materials such as polytetrafluoroethylene (PTFE) and polyvinylidene fluoride (PVDF).

Membrane distillation (MD) has emerged as a promising technology for treating industrial wastewater at elevated temperatures due to its ability to utilize thermal gradients as the driving force for separation. Previous studies have demonstrated that PTFE membranes exhibit superior contaminant removal performance compared to PVDF membranes when treating industrial wastewater at feed temperatures ranging from 50 to 80 °C, as PTFE maintains stable filtration efficiency while PVDF membranes experience a notable decline in performance at higher operating temperatures [5].

The efficiency of a membrane distillation system is commonly evaluated based on its permeate flux, which represents the volume of purified water produced per unit membrane area over a specific period. Several factors influence this flux, including the intrinsic properties of the membrane—such as material composition, thickness, porosity, and pore size—as well as operational parameters like the temperatures and flow rates of both feed and permeate streams.

2. Method

This study focuses on transforming chemical laboratory liquid waste into water that meets clean water standards. The raw water samples were collected from the Environmental Engineering Chemistry Laboratory at PGRI Adi Buana University, Surabaya. Before treatment, the samples were analyzed to determine the initial concentrations of Iron (Fe). The analysis showed that the wastewater contained 0.4 ppm of Fe. These results indicate that the concentrations of Fe exceed the permissible limits set by the Regulation of the Minister of Health of the Republic of Indonesia No. 2 of 2023 on Environmental Health.

Following the preliminary assessment, the study proceeded with the design of a treatment system intended to convert the laboratory's liquid waste into clean water. The treatment system was developed using membrane distillation technology, employing PTFE membranes with pore sizes of 0.22 μm and 0.45 μm , as well as PVDF membranes with a 0.45 μm pore size. Once the system design was completed, simulations were carried out to evaluate its potential in producing clean water that complies with national quality standards. The final stage of the research involved testing the treated water to assess the effectiveness and performance of the designed system in reducing heavy metal content and improving overall water quality.

2.1 Characteristics of Liquid Waste Prior to Treatment

The preliminary examination of liquid waste generated by the Chemistry Laboratory, Faculty of Engineering, Universitas PGRI Adi Buana Surabaya, revealed the presence of heavy metals, particularly a high concentration of iron (Fe). Given these findings, it is essential to treat the wastewater before it is released into the environment to avoid potential pollution of surrounding water bodies. The results of this initial analysis are presented in Table 1 below.

Table 1. Initial Test Results of Liquid Waste of Environmental Engineering Chemical Laboratory Liquid Waste

Parameter	Initial Test Result (ppm)	Quality Standards (ppm)
Iron (Fe)	0.4	0.2

Source: Laboratory Test Results of Sidoarjo DLHK

2.2 Treating Liquid Waste using Distillation Membranes

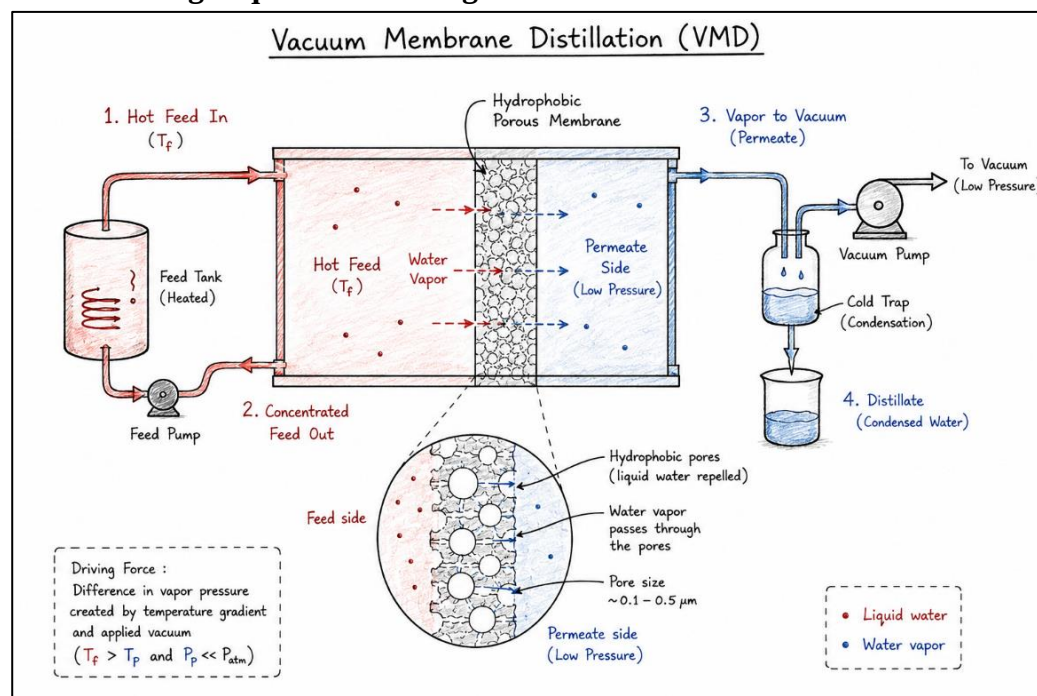


Figure 1. Diagram of the Vacuum Distillation Membrane Processing Flow for Laboratory Liquid Waste

A 250 ml distillation flask was filled with liquid waste from the chemistry laboratory. The wastewater was then preheated to 100 $^{\circ}\text{C}$ using a heater, passed through a condenser to

turn steam into water, and then passed through a Bucher funnel to be filtered through 0.22 μm PTFE, 0.45 μm PTFE, and 0.45 μm PVDF membranes with the help of a vacuum. Permeate is created throughout the membrane's processing procedure and is examined to ascertain the removal percentage [6]. Figure 1 illustrates the use of a distillation membrane for processing.

Vacuum Membrane Distillation (VMD) was performed by maintaining a temperature gradient and a pressure gradient across a hydrophobic microporous membrane. The feed stream was heated to a feed temperature (T_f) higher than the permeate temperature (T_p) to increase the vapor pressure of water at the membrane surface. Simultaneously, the permeate side was maintained under vacuum using a vacuum pump, resulting in a permeate pressure (P_p) substantially lower than the atmospheric pressure (P_{atm}). The resulting transmembrane vapor pressure difference ($T_f > T_p$ and $P_p \ll P_{atm}$) provided the driving force for selective water vapor transport through the membrane pores. Because the membrane is hydrophobic, liquid water and dissolved contaminants were prevented from penetrating the membrane, allowing only water vapor to permeate and subsequently condense into high-purity distillate.

3. Result and Discussion

Figure 2 shows the impact of various PTFE and PVDF membrane types on lowering iron levels using different membrane materials and pore sizes.

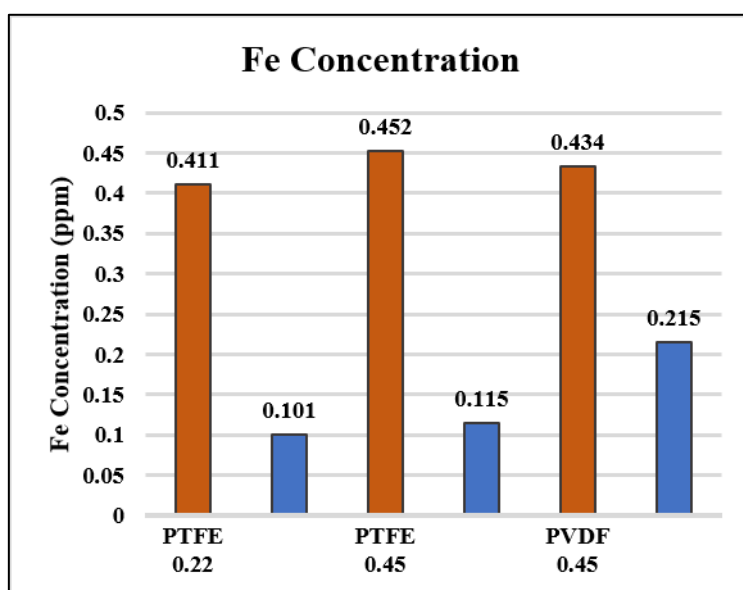


Figure 2. Fe Concentration (Orange = Before VMD, Blue = After VMD)

The initial Fe concentrations (orange bars) were 0.411 ppm for the PTFE 0.22 μm membrane, 0.452 ppm for the PTFE 0.45 μm membrane, and 0.434 ppm for the PVDF 0.45 μm membrane. After filtration (blue bars), the Fe concentrations decreased to 0.101 ppm, 0.115 ppm, and 0.215 ppm, respectively. Among the three membranes, the PTFE 0.22 μm membrane exhibited the highest Fe removal efficiency, reducing the Fe concentration by approximately 75.4%, followed by the PTFE 0.45 μm membrane with a reduction of approximately 74.6%. In

contrast, the PVDF 0.45 μm membrane showed the lowest removal performance, reducing the Fe concentration by approximately 50.5%. These results indicate that the PTFE membranes were more effective than the PVDF membrane in removing dissolved Fe, with the smaller pore size (0.22 μm) providing slightly better removal efficiency.

The reduction in Fe concentration can be influenced by several factors, with temperature being one of the most significant. Research conducted by [7] demonstrated that the flux performance of PTFE microporous membranes tends to rise as the feed temperature increases within the range of 80°C to 130°C. Their findings also revealed that PTFE membranes exhibit superior permeability and contaminant rejection at higher temperatures, attributed to their enhanced hydrophobic characteristics and strong resistance to wetting.

Temperature plays a significant role in influencing this process. Research conducted by [8] explored how a temperature of 100 °C, combined with applied pressure, affects the removal efficiency of metal ions from aqueous solutions using membrane distillation [8].

Supporting this, [9], [10] reported that the permeate flux of PTFE and PVDF microporous membranes increased as the feed temperature rise from 80°C to 130°C. Comparative thermal analyses have consistently demonstrated that PTFE possesses a higher degradation temperature and lower susceptibility to thermal oxidation than PVDF, even under extreme conditions such as gamma-ray irradiation.

Shim et al. [11] reported that polytetrafluoroethylene (PTFE) exhibits higher thermal stability than polyvinylidene fluoride (PVDF) when subjected to gamma-ray irradiation. Based on TGA and DSC analyses, PTFE showed a higher degradation temperature and lower extent of oxidative damage compared to PVDF, indicating superior resistance to thermal and radiation-induced degradation.

Thickness and Porosity Trade-offs in PTFE is able to compensate for smaller apertures, 0.22 μm membranes are often engineered to be thinner or more porous than 0.45 μm variants. This balances mass transfer resistance, leading to comparable net efficiency [12].

In membrane distillation applications treating industrial wastewater at elevated feed temperatures (50–80 °C), PTFE membranes consistently achieved higher contaminant removal efficiencies than PVDF membranes, which exhibited significant performance decline at higher temperatures [5]

To stop heat loss in the membrane, a PTFE membrane material with low mass transfer resistance and low thermal conductivity is utilized. Furthermore, membrane materials with good chemical and thermal stability are needed. According to Minister of Health Regulation No. 2 of 2023, these three factors can lower iron levels to the maximum allowable level of 0.2 ppm, which is the threshold for hygiene and sanitation water quality regulations.

Figure 3 depicts the Fe removal percentages achieved by the different membrane types. The PTFE 0.22 μm membrane exhibited the highest removal efficiency, achieving 75.426%, followed closely by the PTFE 0.45 μm membrane with 74.558% removal. In contrast, the PVDF 0.45 μm membrane demonstrated a considerably lower removal efficiency of 50.461%. The difference in removal performance suggests that the PTFE membranes were more effective than the PVDF membrane for Fe removal under the tested conditions. Furthermore, the slightly

higher efficiency of the PTFE 0.22 μm membrane compared with the PTFE 0.45 μm membrane indicates that a smaller pore size may enhance Fe retention, although the improvement was relatively small. Overall, the results demonstrate that both membrane material and pore size influence Fe removal efficiency, with PTFE membranes providing superior performance.

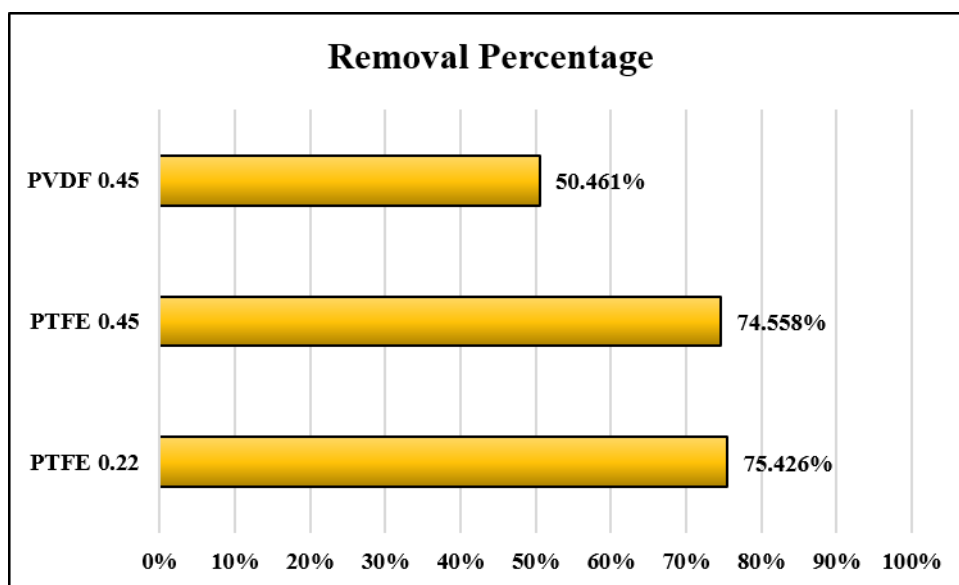


Figure 3. Percentage of Decrease in Iron Concentration

The comparative performance of PTFE and PVDF membranes is frequently documented, though findings on their relative efficiency are nuanced and depend on material structure. One study notes that PVDF hollow fiber membranes may exhibit higher water flux than PTFE equivalents, potentially due to having a thinner membrane wall and higher porosity [13]. Conversely, other research highlights the superior performance of PTFE in specific applications, noting that PTFE membranes can achieve higher permeate flux compared to PVDF membranes [14].

Regarding the rejection of contaminants, PTFE membranes have been shown to outperform PVDF in chemically intensive environments, such as the treatment of anodic oxidation wastewaters [15]. This superiority is attributed to the PTFE membrane's morphology, structure, and inherently slippery, hydrophobic surface, which imparts anti-adhesion properties that prevent fouling and maintain water quality [15]. While both materials are hydrophobic and effective at removing contaminants like conductivity, COD, and sulfate, the PTFE structure remains less influenced by high feed water temperatures and chemical exposure compared to PVDF [15], [16].

Furthermore, the morphological of PTFE features a highly uniform "node-and-fibril" structure. In contrast, PVDF membranes—typically produced via phase inversion—often possess sponge-like or finger-like structures with broader pore size distributions. These irregular large pores in PVDF act as focal points for early wetting and leakage [17].

At elevated operating temperatures, PTFE membranes exhibit more stable pollutant removal performance than PVDF membranes due to their superior thermal stability, whereas

PVDF membranes tend to suffer from structural relaxation and performance degradation near their thermal limit.

4. Conclusion

The study concludes that membrane distillation is a highly effective technology for treating laboratory liquid waste, specifically in reducing iron (Fe) concentrations to meet Indonesian environmental health standards. Experimental results demonstrate that Polytetrafluoroethylene (PTFE) membranes with pore sizes of 0.22 μm and 0.45 μm are the most efficient, achieving reduction efficiencies of approximately 75.4% and 74.6%, respectively. This process successfully lowered the initial iron concentration of 0.4 ppm to as low as 0.1 ppm, which is well below the maximum permissible threshold of 0.2 ppm established by the Minister of Health Regulation No. 2 of 2023. In contrast, the Polyvinylidene fluoride (PVDF) membrane exhibited significantly lower performance, with a removal efficiency of only 50.5%.

The superior performance of the PTFE membranes is primarily attributed to their exceptional thermal stability and strong hydrophobic properties compared to PVDF. When operating at a high temperature of 100 °C, the PTFE membranes maintained their structural integrity and consistent filtration performance, whereas PVDF membranes tend to suffer from performance degradation near their thermal limits. Furthermore, the low thermal conductivity and high resistance to wetting of the PTFE material helped prevent heat loss and ensured reliable contaminant rejection. These findings indicate that membrane distillation using PTFE membranes provides a promising and practical alternative for managing hazardous laboratory waste and protecting the environment from heavy metal contamination.

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