

Article

Mercury, Arsenic and Lead Removal by Air Gap Membrane Distillation: Experimental Study

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Abstract: Synthetic industrial wastewater samples containing mercury (Hg), arsenic (As), and lead (Pb) ions in various concentrations were prepared and treated by air gap membrane distillation (AGMD), a promising method for heavy metals removal. Three different membrane pore sizes (0.2, 0.45, and 1 μm) which are commercially available (TF200, TF450, and TF1000) were tested to assess their effectiveness in combination with various heavy metal concentrations and operating parameters (flow rate 1–5 L/min, feed temperature 40–70 °C, and pH 2–11). The results indicated that a high removal efficiency of the heavy metals was achieved by AGMD. TF200 and TF450 showed excellent membrane removal efficiency, which was above 96% for heavy metal ions in a wide range of concentrations. In addition, there was no significant influence of the pH value on the metal removal efficiency. Energy consumption was monitored at different membrane pore sizes and was found to be almost independent of membrane pore size and metal type.

Keywords: air gap membrane distillation; wastewater treatment; heavy metal removal; industrial wastewater

1. Introduction

Industrial wastewater is one of the most serious pollutants, contributing significantly to the current load constraints of conventional wastewater treatment plants. Several industrial sectors such as petroleum, petrochemicals, tanning and electroplating are generating large amounts of toxic heavy metal wastewater, which needs to be extensively treated prior to its release to the ecosystem. The type of contaminants and physicochemical properties of industrial wastewater effluent such as temperature, viscosity, salinity, and turbidity vary with each stream. Nevertheless, most industrial wastewater streams contain heavy metals such as zinc, copper, mercury, lead, and arsenic in amounts that if left untreated will exceed the limit allowable by the national public health and safety regulations for their safe disposal [1].

Heavy metals have different properties based on their atomic number and chemical structure that contribute to their effects and toxicity to the environment and human health. There are 17 elements that are considered to be very toxic, including mercury (Hg), lead (Pb), and arsenic (As). Toxicity levels depend on the type of metal and the type of organism that is exposed to it [2].

1.1. Membrane Technology

Membrane technologies can be used to effectively treat wastewater that includes heavy metals due to their flexibility, scalability, and easiness to operate and maintain. Heavy metal concentrations, type of contaminants, and the level of filtration required determines the treatment process type. Moreover, the membrane performance (rejection) can be affected by several parameters such as the material used, the membrane pore size, and the membrane composition; for example, metallic, ceramic, and composite materials are utilized to remove heavy metals. In addition, the influence of heavy metal concentration and type of contaminants on the rejection factor were reported.

Kurniawan et al. [3] conducted a comparative study of heavy metal removal (Cd, Cr, Cu, Ni, and Zn) via ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO). They concluded that UF could remove about 90% of heavy metal concentration at a pH of 5 to 9.5. In addition, they noticed that the heavy metal concentration is essential in the case of UF and NF; however, the rejection factor for RO was not affected by the metal concentration. Moreover, Seidel et al. [4] investigated the rejection factor of As(III) and As(V) using NF and RO. They found that As(V) can be effectively removed by RO and NF. However, RO can be used to achieve a high removal rate in As(III) only. In addition, they observed that the removal of As(III) declined from 28% to 5% as the metal concentration increased from 0.01 to 0.316 ppm.

A hybrid process has been implemented to treat wastewater that contains heavy metals. For example, Blocher et al. [5] developed a flotation/microfiltration process to remove copper, nickel, and zinc from aqueous solutions. Moreover, synthetic waste water was treated by electrocoagulation followed by a microfiltration process [6].

1.2. Membrane Distillation (MD)

Membrane distillation (MD) is a promising technology for treating saline water and wastewater with high rejection rates, which cannot be accomplished by conventional technologies. MD is a thermally driven separation process in which only the vapor molecules pass through a microporous hydrophobic membrane. The vapor pressure difference which is caused by the temperature difference across the membrane surface is considered to be the driving force for MD. In order to avoid a wetting incidence of the membrane pores, the membrane pore size must be as small as possible. However, MD permeate flux will decline. Therefore, an optimum membrane pore size needs to be determined for each MD application and the feed type to be treated [7,8].

Liquid entry pressure (LEP) is an important membrane characteristic. LEP is defined as the minimum transmembrane pressure that is required for a feed solution to penetrate a large pore size (r_{max}). Therefore, the hydrostatic pressure should be lower than LEP to avoid membrane wetting [9,10]. LEP can be estimated from the following equation:

$$\Delta P = P_F - P_P = \frac{-2B\gamma_l \cos\theta}{r_{max}} \quad (1)$$

where P_F and P_P are the hydraulic pressures on the feed and permeate side, B is the geometric pore coefficient (equal to 1 for cylindrical pores), γ_l is the liquid surface tension, θ is the contact angle, and r_{max} is the maximum pore size.

Operating conditions such as the temperature, flow rate, membrane type, as well as membrane pore structure play a significant role in the system's efficiency [10].

The mass flux (J) in MD is assumed to be proportional to the vapor pressure difference across the membrane, and is given by [10,11]

$$J = C(P_f - P_p) \quad (2)$$

where C is the membrane coefficient and P_f and P_p is the difference of vapor pressure at the membrane feed and permeate surfaces.

Membrane distillation (MD) has not been widely applied for heavy metal removal; for example, contaminated groundwater with arsenic (40–2000 ppm) has been treated by direct contact MD (DCMD) to 10 ppm [12]. Almost 100% arsenic removal was achieved without wetting the membrane pore. Moreover, arsenite (As(III)) and arsenate (As(V)) removals were examined by DCMD. It was pointed out that the rejection of As was independent of the solution pH and the temperature [13]. Treatment of heavy metal wastewater by vacuum membrane distillation (VMD) was achieved by Zhongguang Ji [14]. The effect of pH on VMD performance was studied, and the author specified that the VMD process showed good acid resistance. A modified PVDF/TiO₂ electrospun membrane was prepared to remove heavy metal traces from water via VMD [15]. Improvements in the rejection factor and permeate flux were noticed.

To the authors' current knowledge, there are no studies available which deal with the removal of heavy metal from industrial wastewater via AGMD. Therefore, this study investigates the application of suitable membrane technologies over a wide range of industrial heavily polluted wastewater. Air gap membrane distillation (AGMD) is a promising method for heavy metals removal (Pb, As and Hg) from industrial wastewaters. AGMD was chosen to treat industrial wastewater due to the high quality of permeate flux and low risk of membrane wetting. Three different membrane pore sizes (0.2, 0.45 and 1 µm) which are commercially available (TF200, TF450 and TF1000) were tested to assess their effectiveness in combination with various heavy metal concentrations and operating parameters (flow rate –5 L/min, feed temperature 40–70 °C, pH 2–11). The AGMD process could be used for localized, low-cost deployment treatment in the industry.

2. Experimental Procedure and Material

The impact of a wide range of concentrations for Pb, As, and Hg on permeate flux and on the rejection factor was examined as shown in Table 1. Metal ion solutions of Pb²⁺, As³⁺, and Hg²⁺ were prepared from Pb(NO₃)₂, AsO₃, and HgCl₂ compounds, respectively. The influence of pH on the rejection factor at room temperature was explored at three different pH values: 2, 7, and 11. In addition the influence of pore size was studied using three commercial membranes (0.2, 0.45 and 1.0 µm). The experiments were conducted over a wide range of heavy metal concentrations at a constant feed flow rate (1.5 L/min), feed temperature (50 °C) and coolant temperature (10 °C). Moreover, a mixed metal ion solution of Pb²⁺, As³⁺, Hg²⁺, and NaCl (synthetic industrial wastewater) was prepared and treated by different membrane pore sizes for 5 h. The experimental tests were achieved by the AGMD module in a horizontal position with a membrane effective area of 0.006 m², as shown in Figure 1. The air gap width was about 5 mm. Three types of flat sheet polytetrafluoroethylenes (PTFE) microporous hydrophobic membranes were employed in this study, as shown in Table 2.

Table 1. Range of concentration of heavy metals used in the filtration process.

Heavy Metal	Concentration (ppm)				
Lead Pb ²⁺	50	100	200	1000	1500
Arsenic As ³⁺	2	5	10	25	100
Mercury Hg ²⁺	5	10	20	-	-
Synthetic wastewater	Pb ²⁺ (1000 ppm); As ³⁺ (5 ppm); Hg ²⁺ (5 ppm) and NaCl (2000 ppm).				

The effect of the flow rate, feed temperature, and condensing temperature on the rejection factor was examined. The influence of contaminated water flow rate at 1, 3 and 5 L/min was explored. The flow rate could be manipulated by adjusting the pump speed to achieve the desired flow rate. The contaminated water was heated and pumped to the top part of the membrane cell at a constant temperature (50 °C). Moreover, cooling fluid was also pumped at a constant flow rate (2.5 L/min) to the bottom cell compartment at a constant temperature of 10 °C.

With regard to the feed temperature influence, contaminated water temperature at 40 °C, 50 °C, 60 °C and 70 °C was analysed. The temperature was adjusted to the desired point and controlled as

well throughout the process. The contaminated water was heated and pumped at 1.5 L/min to the top part of the membrane cell. The cooling fluid temperature was kept at a constant temperature of 10 °C and pumped to the bottom part of membrane cell.

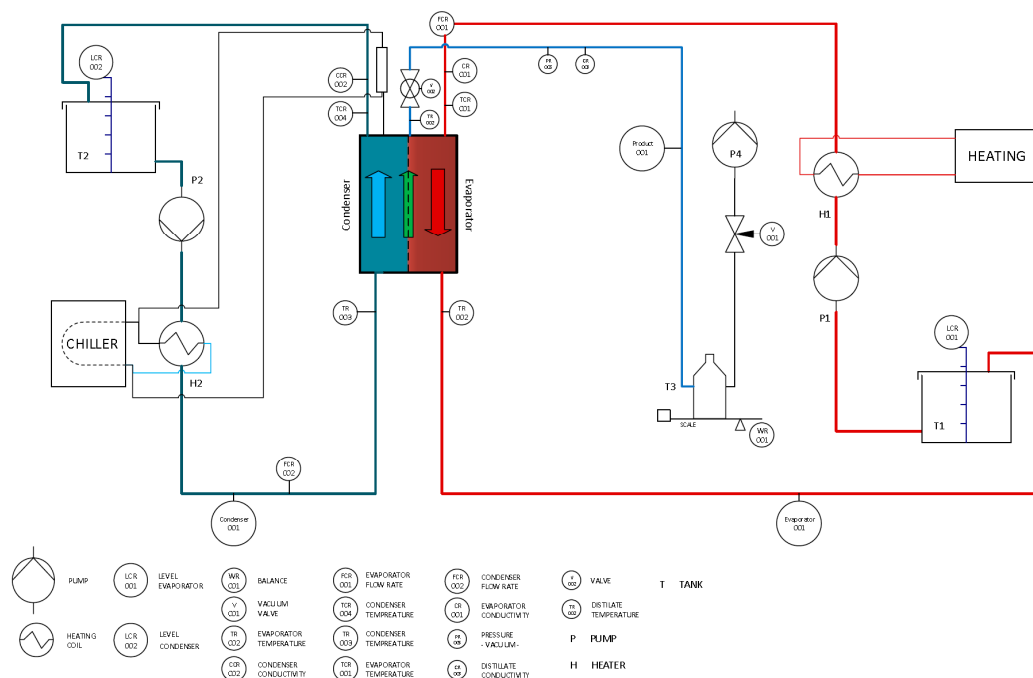


Figure 1. Schematic diagram of the air gap membrane distillation (AGMD) experimental apparatus used in this study.

Table 2. Properties of membranes that were used in this work as specified by the manufacturer. PTFE: polytetrafluoroethylenes.

Specification	Description
Trade name	TF200 TF450 TF1000
Manufacturer	Sterlitech corporation
Material	PTFE
Membrane support	Polypropylene
Thickness	175 µm
Mean pore size and liquid entry pressure (LEP)	0.2 µm (2.55 bar) 0.45 µm (0.76 bar) 1.00 µm (0.28 bar)

The permeate flux (J) was measured by weighing the obtained permeate for a predetermined time using an electronic balance which was connected to a computer:

$$J = \frac{W}{A \Delta t} \quad (3)$$

where W is the obtained permeate weight and A is the effective membrane area.

Furthermore, the concentration of heavy metals in the feed and permeate was measured by atomic absorption spectrometry (AAS) and inductive couple plasma (ICP). Standard solutions for Pb(II),

As(III) and Hg(II) were used to ensure the readings of AAS and ICP were correct. Therefore, the rejection factor can be estimated as

$$\text{Rejection Factor} = \left(1 - \frac{C_p}{C_f}\right) \times 100 \quad (4)$$

where (C_f) and (C_p) are the feed and permeate concentrations (in ppm), respectively.

3. Results and Discussion

The effect of various metal ion concentrations of Pb^{2+} , As^{3+} , and Hg^{2+} in different operating conditions on the membrane flux and the rejection factor were tested. Furthermore, the impact of the pH solution on the rejection factor was investigated.

3.1. Membrane Pore Size and Metal Concentration Effect

The impact of the membrane pore size on the heavy metal rejection by AGMD was investigated. The experiments were conducted over a wide range of heavy metal concentrations at a constant feed flow rate, feed temperature, and coolant temperature.

The effect of membrane pore size on the heavy metal rejection is shown in Figure 2. It should be noted that the TF200 membrane has a pore size of 0.2 μm , the TF450 membrane has a pore size of 0.45 μm and the TF1000 membrane has a pore size of 1.0 μm . TF200 shows excellent membrane removal for heavy metal ions $\text{Pb}(\text{II})$, $\text{As}(\text{III})$, and $\text{Hg}(\text{II})$ with a wide range of concentrations. The rejection was almost 100% due to the small membrane pore size. For instance, the permeate flux of Pb (1000 ppm), As (25 ppm), and Hg (10 ppm) was 0.271, 0.293, and 0.302 $\text{g}/\text{m}^2\cdot\text{s}$, respectively. As is evident from Figure 2, the efficiency of metal ion removal by the TF200 membrane was not affected by the metal concentration. For TF450, the rejection factor for $\text{Hg}(\text{II})$ was almost 100% and was not affected by the metal concentration due to its ionic size. On the other hand, the rejection factor for $\text{Pb}(\text{II})$ at 1000 ppm and 1500 ppm was 98% and 96%, respectively. In contrast to TF200, the TF1000 membrane showed less membrane efficiency for metal removal due to its increased membrane pore size. This result can be attributed to the decrease of the membrane hydrophobicity due to the wetting incidence in the membrane pores. Few hydrophobic membrane pores were wetted, which allowed the feed solution to penetrate through the permeate side due to the lower value of the liquid entry pressure (LEP) for TF1000. It is worth noting that the LEP value depends on several factors such as the maximum membrane pore size and the membrane hydrophobicity. Nonetheless, it was noticed that the permeate flux increased compared to TF200 and TF450. As a consequence, TF1000 was not further used.

Moreover, the rejection factor of $\text{Pb}(\text{II})$, $\text{As}(\text{III})$ and $\text{Hg}(\text{II})$ for high concentrations was lower than the rejection factor for low concentrations, which implies that a concentration polarization phenomenon occurred. Concentration polarization is defined as an increase of feed concentration near the membrane surface. Therefore, permeate flux slightly increased and was accompanied with a decrease in the permeate quality due to the wetting incidence of the PTFE membrane. Eykens et al [16] indicated that the membrane defects and the maximum pore size strongly affects LEP and is a possible cause of membrane wetting. For instance, the rejection factor for $\text{Pb}(\text{II})$ 1500 ppm, $\text{As}(\text{III})$ 100 ppm and $\text{Hg}(\text{II})$ 20 ppm was 93%, 97% and 98%, respectively. As a result, the TF200 and TF450 were selected to study the impact of feed flow rate, feed temperature, and pH.

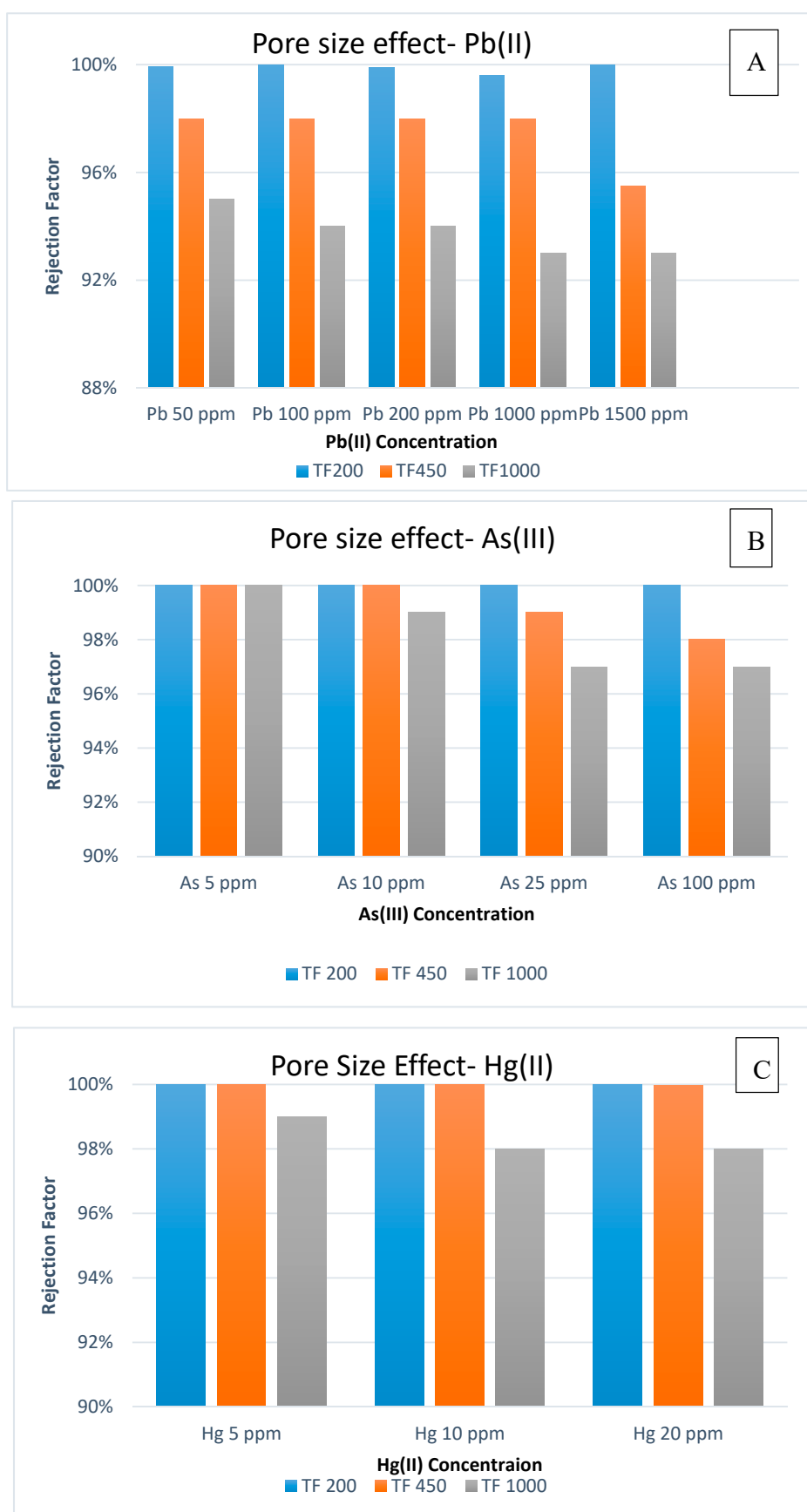


Figure 2. Pore size effect for different heavy metals using TF200, TF450 and TF1000: (A) Pb(II), (B) As(III), and (C) Hg(II).

3.2. Effect of Feed Flow Rate

In order to study the impact of feed flow rate on the rejection of heavy metal removal, several experiments were conducted by changing the initial feed concentration and feed flow rate from 1 to 5 L/min at a constant feed and cooling temperatures.

Figures 3–5 reveal that Pb(II), As(III) and Hg(II) were almost rejected completely (99–100%) by the TF200 membrane due to the high LEP value. Similarly, the TF450 membrane as shown in Figure 5 could reject Hg(II) completely at different flow rates. The high removal of Hg(II) by the TF450 membrane might be due to the membrane hydrophobicity and the size of the mercury ions. Olatunji and Camacho [17] pointed out that the mass transfer coefficient at the feed boundary layer increases due to the increase in feed flow rate which reduces the concentration polarization impact and can lead to membrane wetting.

It was also noticed that Pb(II) rejection increases when the feed flow rate increases regardless of the concentration (Figure 6). For example, the rejection factor of the membrane TF450 for 1000 ppm Pb(II) was 97%, 98% and 100% at 1, 3 and 5 L/min, respectively. This can be explained by the decrease of the concentration polarization phenomenon resulting in the increase of the feed flow rate [11,18].

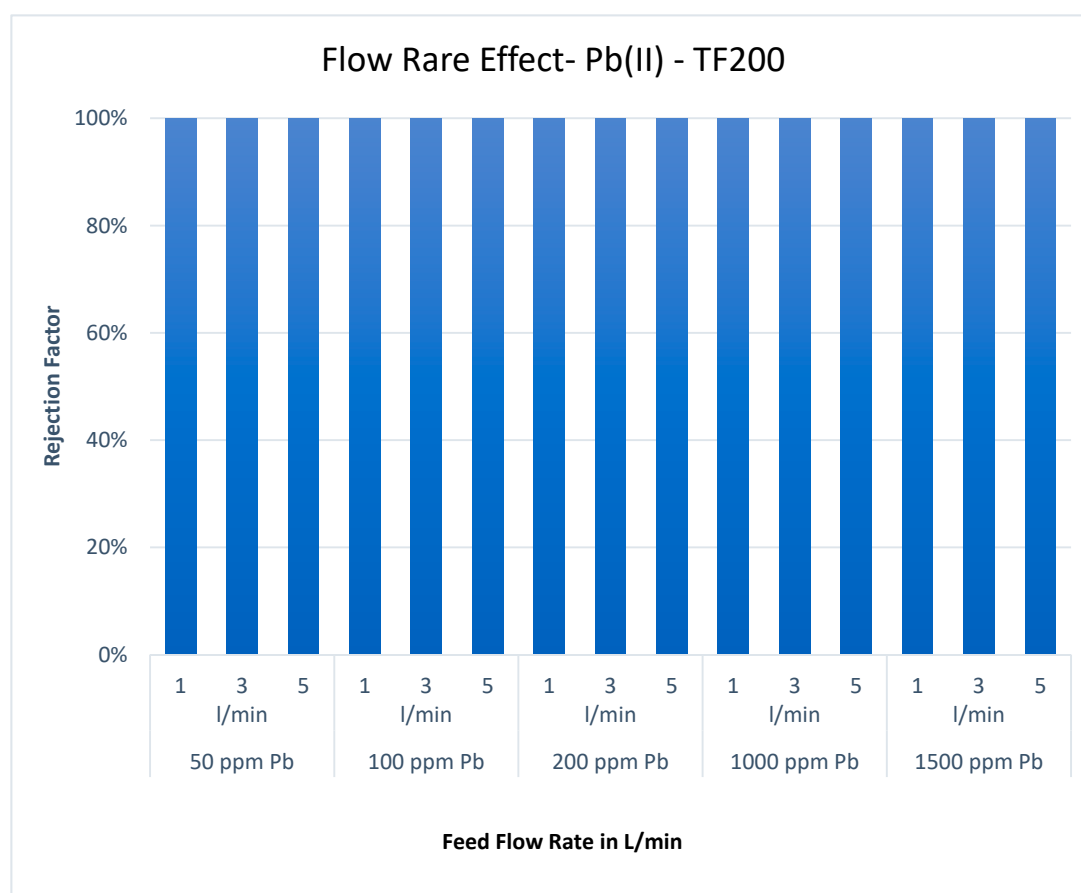


Figure 3. Effect of feed flow rate on the rejection factor using the TF200 membrane for Pb(II) (feed temperature = 50 °C, condensing temperature = 10 °C).

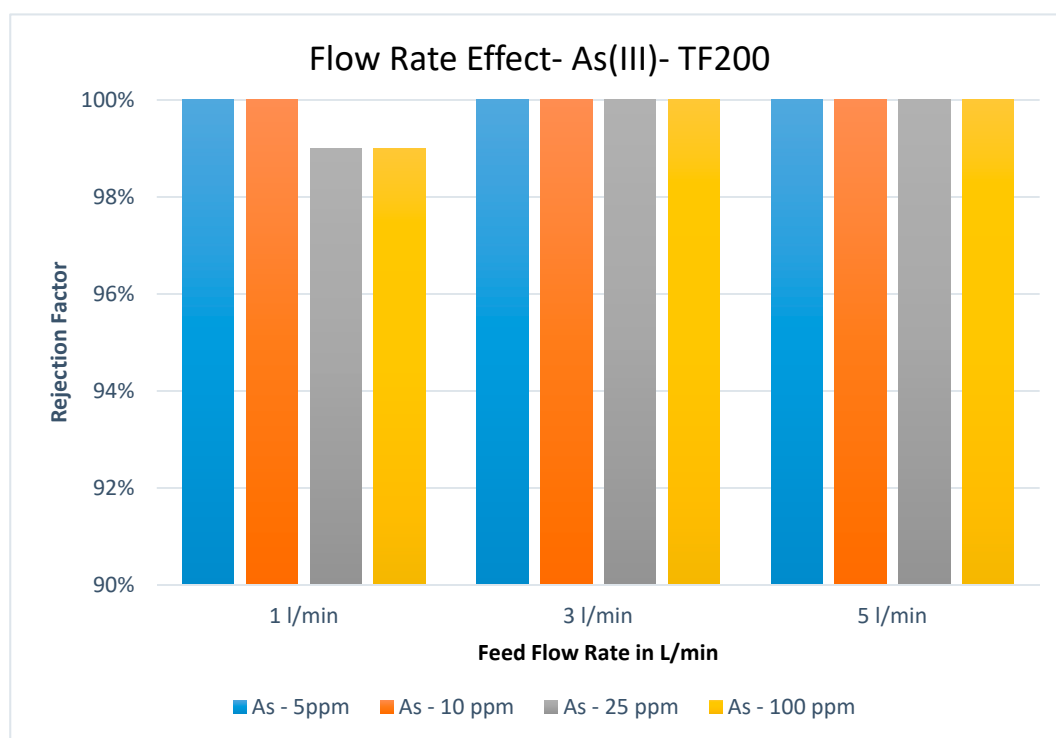


Figure 4. Effect of feed flow rate on the rejection factor using TF200 membrane for As(III) (feed temperature = 50 °C, condensing temperature = 10 °C).

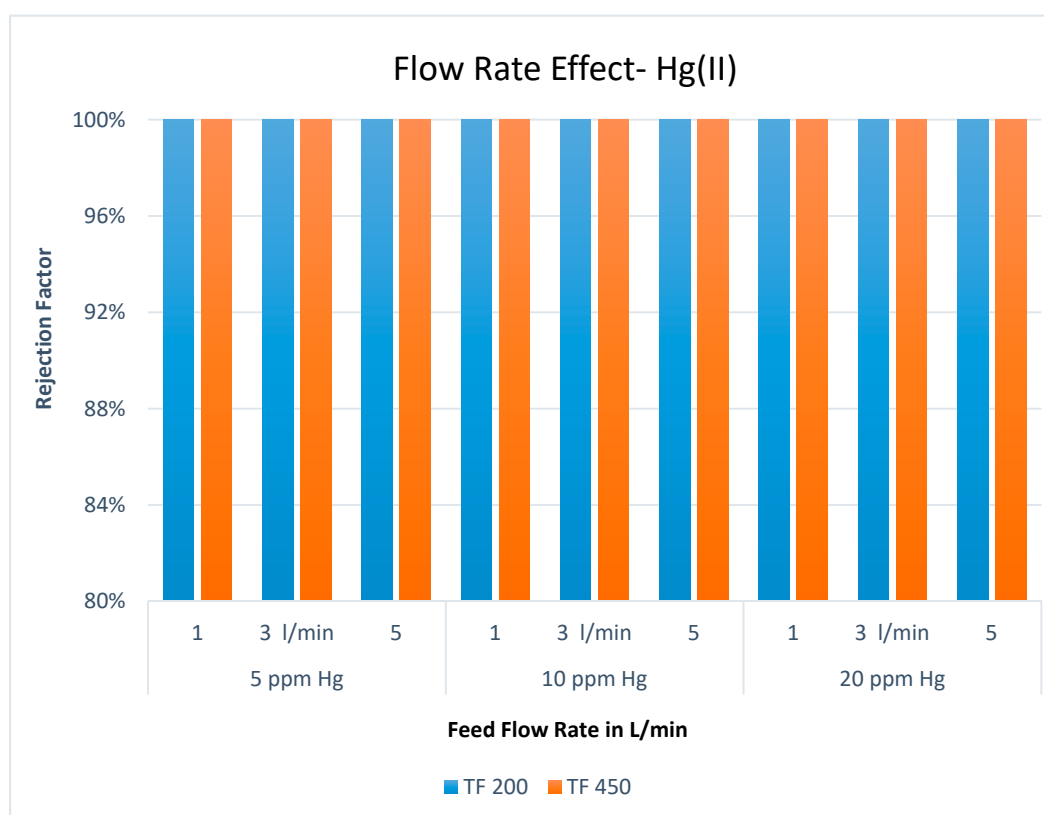


Figure 5. Effect of feed flow rate on the rejection factor using TF200 and TF450 membranes for Hg(II) (feed temperature = 50 °C, condensing temperature = 10 °C).

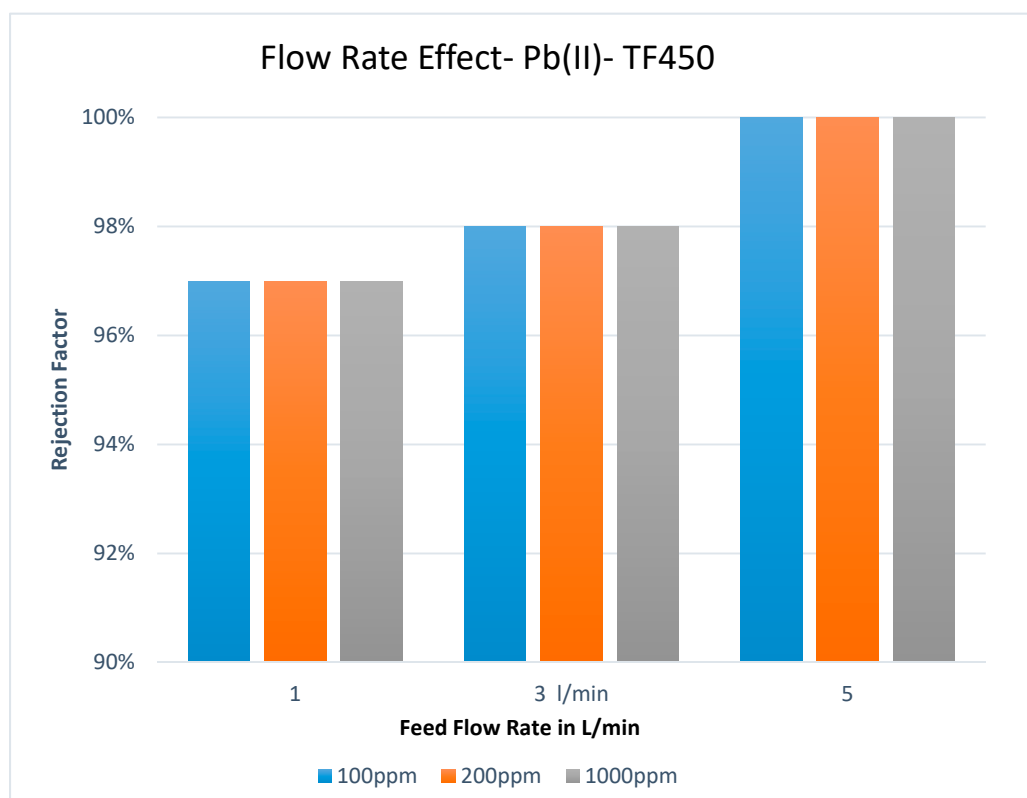


Figure 6. Effect of feed flow rate on the rejection factor using TF450 membrane for Pb(II) (feed temperature = 50 °C, condensing temperature = 10 °C).

3.3. Effect of Feed Temperatures

The impact of feed temperature on heavy metal rejection by the AGMD was investigated. The experiments were performed with a wide feed temperature range—i.e., 40–70 °C—and a constant feed flow rate and coolant temperature.

The impact of feed temperature on heavy metal removal by the AGMD process with a wide temperature range is shown in Figures 7 and 8. The heavy metal removal efficiency for TF200 membrane was perfect and stable over a wide concentration range of heavy metals and temperature. The removal was almost 100%, indicating the minimum influence of feed temperature on the membrane's performance. Likewise, the TF450 membrane showed excellent membrane efficiency for the metal removal of Hg(II) and As(III), which might be attributed to the membrane hydrophobicity. Additionally, a higher flux occurs at higher feed temperatures, which improves the rejection factor due to a “dilution” of the leakage, if it occurred. For example, the Hg(II) removal at 5, 10 and 20 ppm was almost 100% over a wide temperature range. However, feed temperature plays a role in Pb(II) removal, as shown in Figure 8C. This decrease in the Pb(II) removal at high concentration (1500 ppm) might be attributed to the decrease of the feed surface tension with the increase of the feed concentration and temperature [19,20] which negatively affects the LEP value.

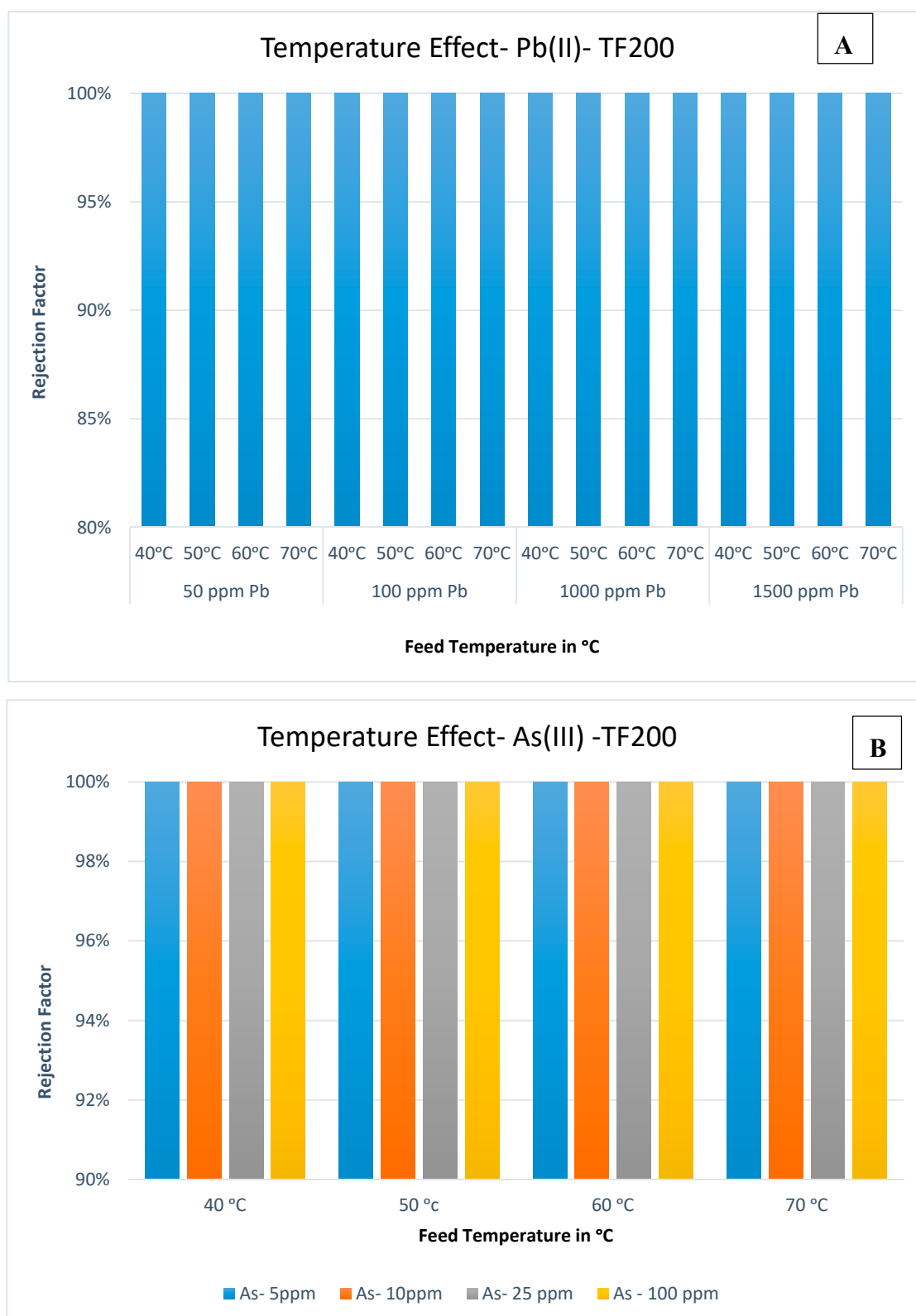


Figure 7. Cont.

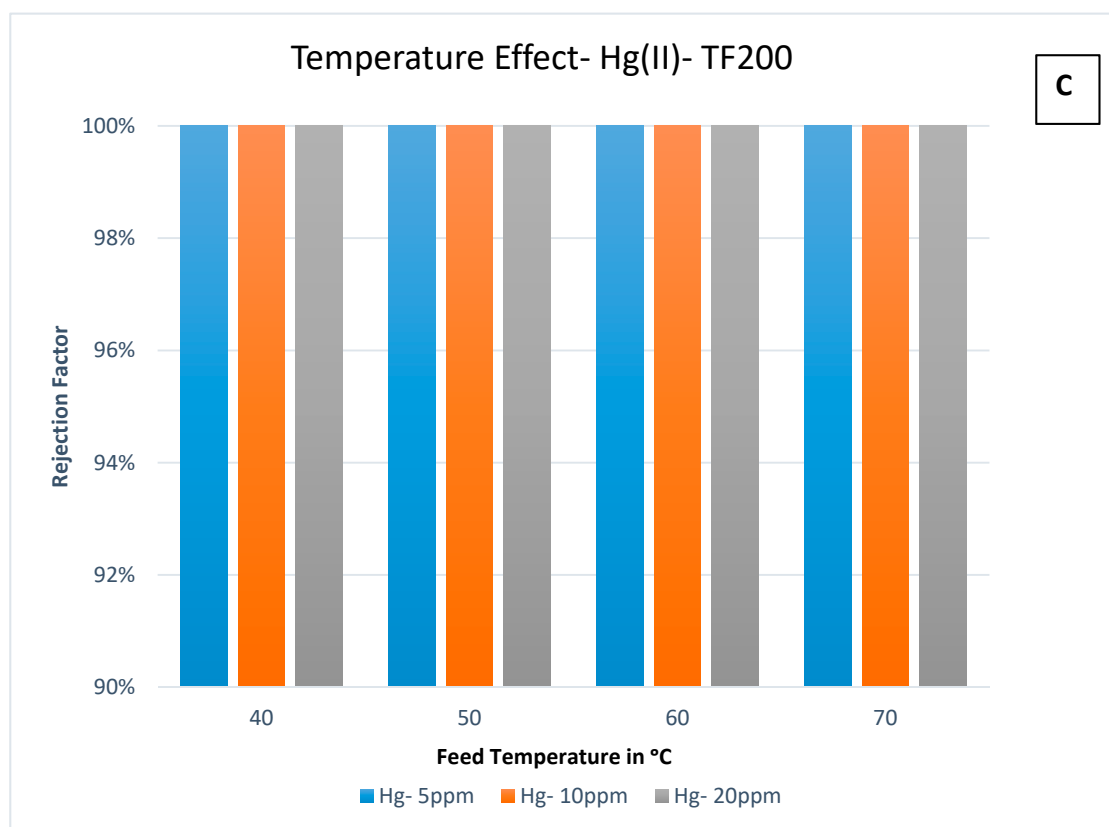


Figure 7. Effect of feed temperature on the rejection factor for TF200 membrane: (A) Pb(II), (B) As(III) and (C) Hg(II) (coolant temperature = 10 °C, flow rate = 3 L/min).

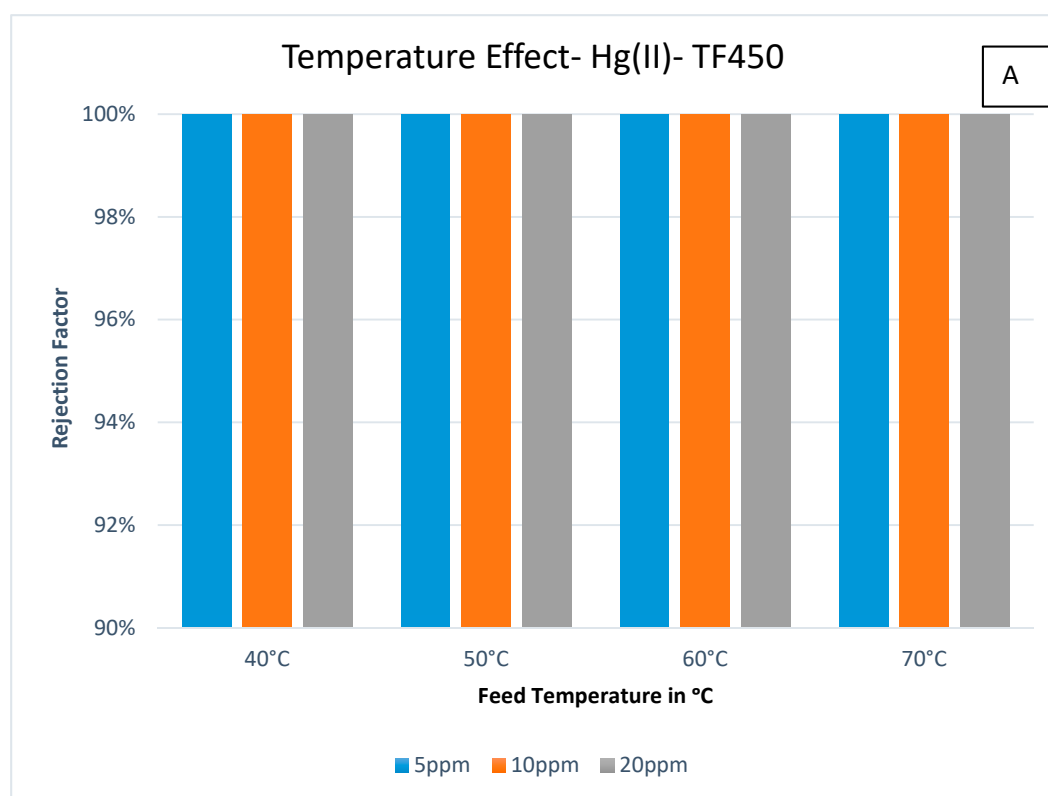


Figure 8. *Cont.*

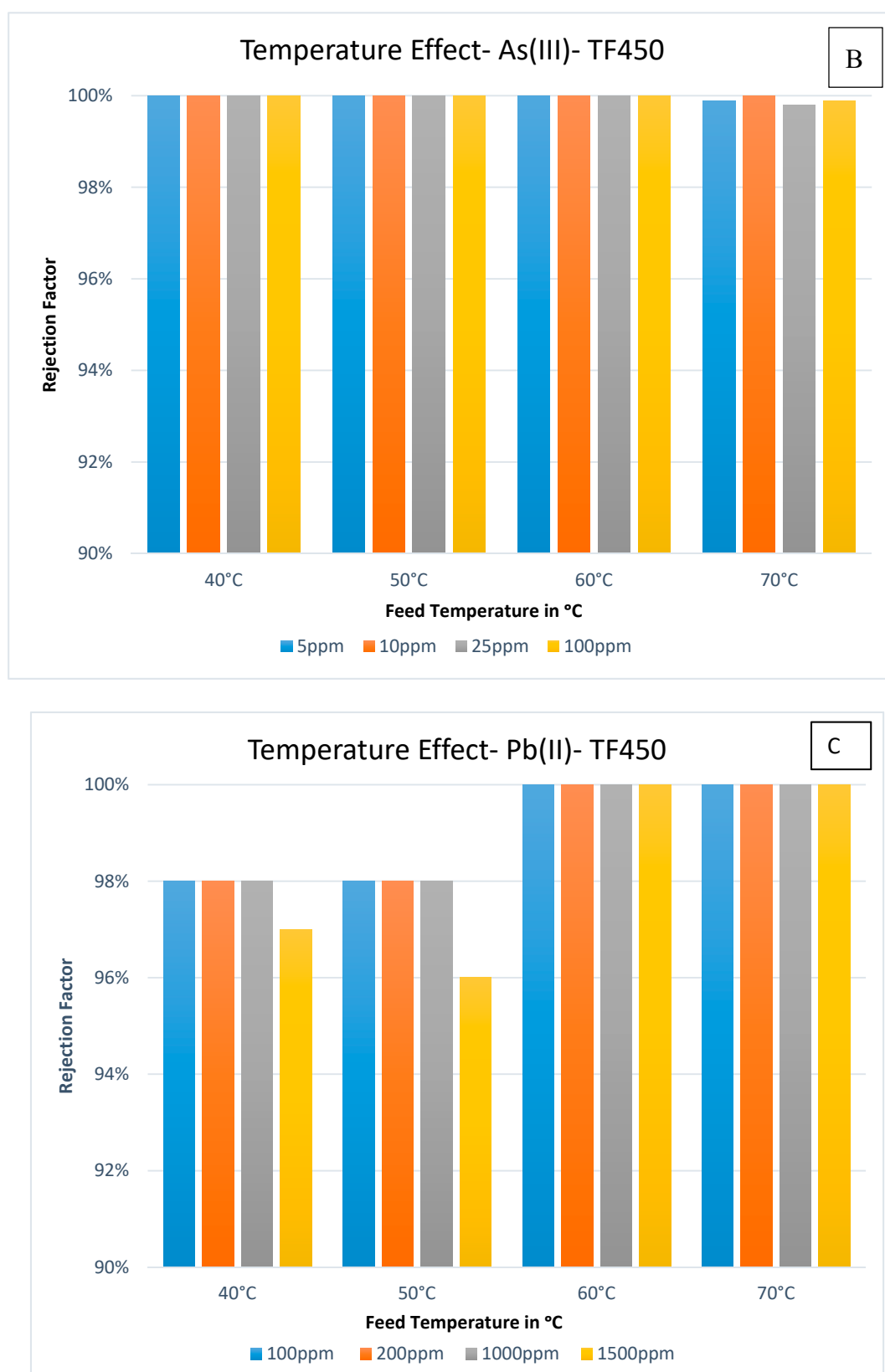


Figure 8. Effect of feed temperature on the rejection factor for TF450 membrane: (A) Hg(II), (B) As(III) and (C) Pb(II) (coolant temperature = 10 °C, flow rate = 3 L/min).

3.4. Effect of pH

Conventional treatment methods—especially membrane processes—are highly dependent on the pH value of the feed solution. The effect of the pH on heavy metal retention was studied at different pH values and flow rates to assess the effectiveness of MD.

The influence of pH on the heavy metal removal from wastewater by AGMD was investigated using TF200 and TF450. Experiments were performed with pH values ranging from 2–11. Metal ion removal was measured to study the impact of solution acidity. As evident from Figure 9, the removal of heavy metals is stable and stayed at an excellent level for the entire treatment process, which indicates that high acidity has no significant impact on the membrane's performance. A similar finding was reported by Zhongguang [14] and Qu et al. [13]. It is important to point out that the rejection factor for TF450 slightly decreased with a low pH value and flow rate, resulting in a rejection factor varying from 96% to 100%. For instance, the concentrations of the heavy metals in the permeate side at a pH of 2 and (3 L/min) for Pb (1500 ppm initial concentration), As (100 ppm initial concentration) and Hg (20 ppm initial concentration) were 36 ppm, 2 ppm, and 0.6 ppm, respectively. It is important to note that the PTFE membrane is an excellent chemical resistor.

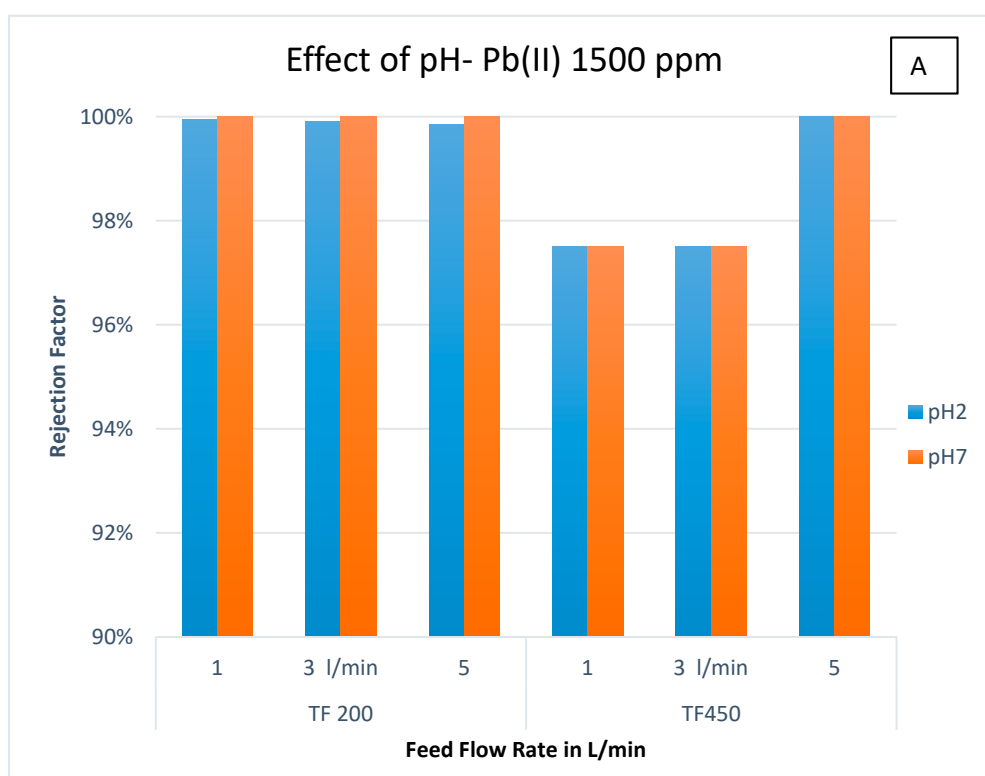


Figure 9. Cont.

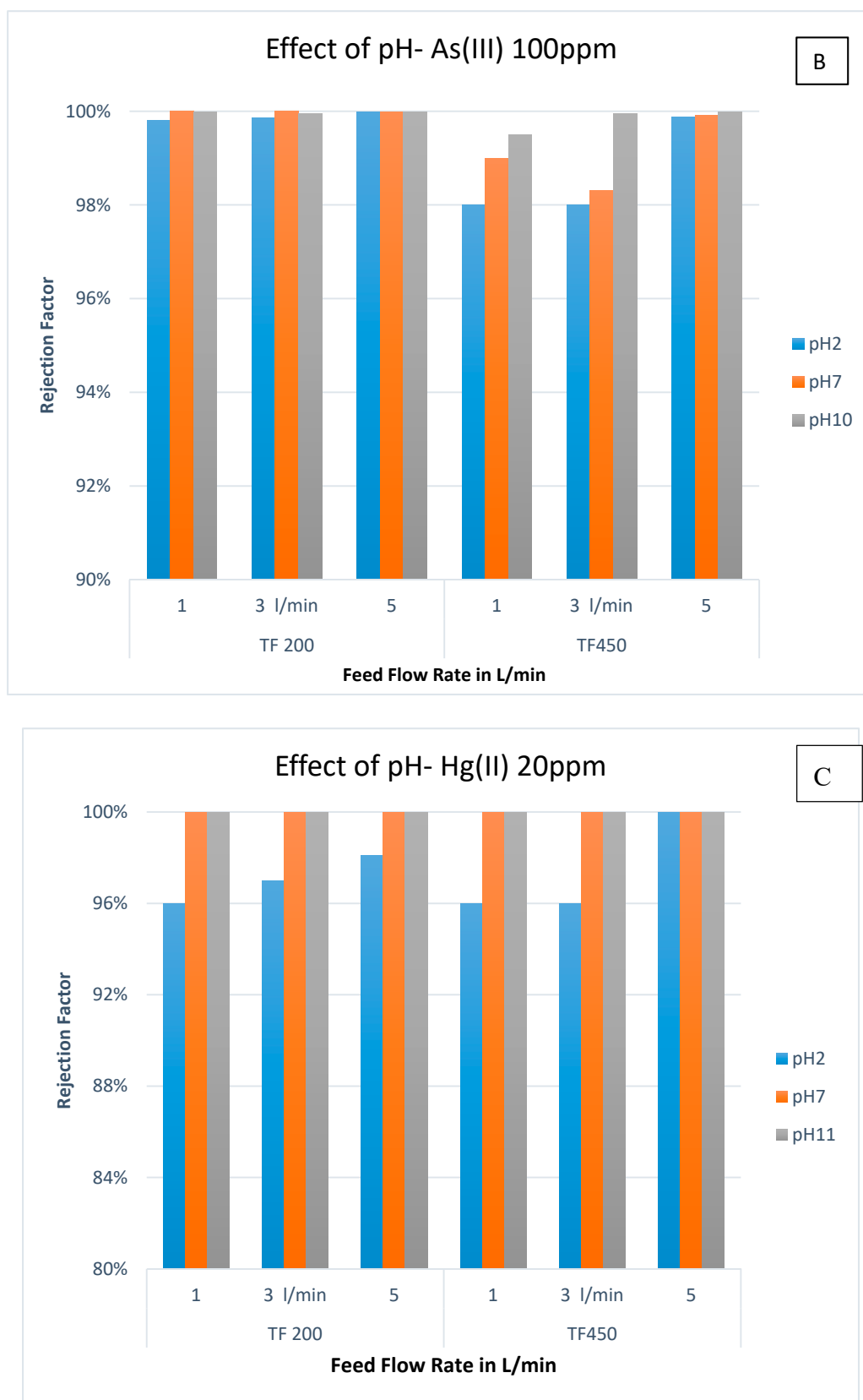


Figure 9. Effect of pH on the rejection factor using TF200/TF450 membranes for (A) Pb(II), (B) As(III) and (C) Hg(II) (feed temperature = 50 °C, flow rate = 3 L/min).

3.5. Energy Consumption

It was observed in this work that the operating parameters of a 5 L/min feed flow rate and a 60 °C feed temperature are the optimum operating parameters due to an excellent rejection factor and a high permeate flux for both TF200 and TF450. Therefore, synthetic wastewater was treated via TF200 and TF450. The permeate flux, energy consumption, and rejection factor were measured. The energy consumption in this study referred to the amount of electrical energy consumed (in kWh) for the heating, cooling, and pump systems. It was inferred that the energy consumption was almost independent of the membrane pore size and metal type. This result can be attributed to the fact that the stagnant air gap represents the predominant resistance to the mass and heat transfer. The energy consumption per gram of treated water was about 0.02 kWh/g for TF200 and about 0.016 for TF450. It is worth noting that the permeate flux for TF200 and TF450 was 0.242, and 0.323 g/m² s, respectively. The results of the permeate flux, energy consumption, and the rejection factor for both membranes are shown in Table 3. It was observed that the rejection factor of the TF200 membrane was almost stable; however, the rejection factor slightly decreased in the case of the TF450 membrane for Pb(II) and As(III).

Table 3. Permeate flux, energy consumption and rejection factor for synthetic wastewater via TF200 and TF450.

Membrane	Permeate Flux (g/m ² s)	Energy Consumption (kWh/g)	Heavy Metal Removal Percentage
TF200	0.242	0.022	Pb(II) 100% As(III) 100% Hg(II) 100%
TF450	0.323	0.016	Pb(II) 99.4% As(III) 99.7% Hg(II) 100%

4. Conclusions

Air gap membrane distillation (AGMD) has been implemented at different operating parameters to treat industrial wastewater. Three different membrane pore sizes of 0.2, 0.45, and 1 µm which are commercially available (TF200, TF450 and TF1000) were tested to assess their effectiveness. Moreover, AGMD was employed to treat synthetic industrial wastewater and assess the effectiveness of the heavy metal removal. The major findings can be summarized as follows:

- AGMD is a promising technology for wastewater treatment and is expected to be a cost-effective process.
- TF200 and TF450 membranes presented a better metal removal efficiency over a wide concentration range, temperature and flow rate.
- The heavy metal rejection increases when the feed flow rate increases, regardless of its concentration.
- The TF1000 membrane showed less membrane efficiency for metal removal due to the increase in the membrane pore size.
- A high acidity of metal solution (PH value) has no significant impact on the membrane's performance.
- The energy consumption is almost independent of the membrane pore size and metal type.

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