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Comparative study of the removal of nickel(II) and chromium(VI) heavy metals from metal plating wastewater by two nanofiltration membranes

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ABSTRACT

The treatment of aviation industry metal plating wastewater containing Ni^{2+} and Cr^{6+} by nanofiltration was investigated in this study. Two commercial membranes (NF90 and NF270) and two membrane filtration systems (dead end and cross flow) were used. The effects of both the transmembrane pressure (10, 20, and 30 bar) and the feed pH (3.5, 7, and 10) on the membrane performance were analyzed. The rejection of both nickel and chromium ions increased with increasing pH but did not considerably change by the pressure difference for both membranes. The optimum conditions were found to be at 30 bar with a pH of 10 for both the NF90 and NF270 membranes. Under optimum conditions for the NF90 membrane, the rejection values of Ni^{2+} and Cr^{6+} were found to be 99.2 and 96.5%, respectively. For the NF270 membrane, the rejection values of Ni^{2+} and Cr^{6+} were 98.7 and 95.7%, respectively.

Keywords: Nanofiltration; Nickel; Chromium; Metal plating wastewater; Dead-end system; Cross-flow system

1. Introduction

Heavy metals are among the most important environmental pollutants due to their toxic nature and their accumulation in living organisms [1]. The main sources of heavy metals in wastewaters result from the automotive, metal, steel, tanning, petroleum, electroplating, fertilizer, photographic, battery, mining and textile industries [2,3]. Two of the most common heavy metals in industrial wastewater streams are chromium and nickel. These two metals cause a large amount of water pollution, and even low levels of them might be harmful for human health [3]. Therefore, the removal of these contaminants is crucial for wastewater treatment. To date, the removal of heavy metals has been achieved through a variety of methods, including chemical precipitation [4,5], coagulation–flocculation [6], ion exchange [7,8], adsorption [9,10] and flotation [11,12]. However, there are several disadvantages associated with the use of these

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methods. For example, large amounts of chemical consumption occur when using both chemical precipitation and coagulation–flocculation methods. In addition, storing the sludge formed during these processes is expensive. In the case of adsorption method, the selectivity and the regeneration are poor. Furthermore, a suitable ion exchange resin is hard to find and is expensive for ion exchange method [13,14].

In recent years, membrane processes have shown promise based on their high efficiency and their convenience for the treatment of wastewater containing heavy metals [3,13,15–24]. Nanofiltration (NF) is a new pressure-driven membrane process. Other new technologies for membranes are reverse osmosis and ultrafiltration [25]. Compared to reverse osmosis, NF has some advantages, including high permeate flux, low operating pressure and low energy consumption. NF membranes also have lower molecular weight cut-off (MWCO) values than ultrafiltration [13].

Heavy metal removal from wastewaters with the NF process has been discussed in the relevant research; however, there is a lack of reports in the literature using this technology for the removal of heavy metals from specifically aviation industry metal plating facilities. In the present work, NF process was studied for the treatment of the wastewater directly taken from a metal plating facility from aviation industry. Two kinds of commercially available NF membranes (NF90 and NF270) were used in the experiments. These two membranes were preferred due to their high rejection capacities and hydrophilic features. The influence of the pressure (10, 20 and 30 bar) and feed pH (3.5, 7, and 10) on the membrane performance for the nickel and chromium removal was studied.

2. Materials and methods

2.1. Membranes

NF90 and NF270 membranes were used for the removal of Ni^{2+} and Cr^{6+} from plating bath waters generated by the aviation industry. Both NF membranes are made of a composite of polyamide thin films. The membranes consist of three layers as follows: a polyester supporting structure, a microporous polysulphone interlayer and an ultrathin polyamide barrier layer (on the top). The NF270 membrane is a piperazine-based semi-aromatic polyamide thin-film composite, whereas the NF90 membrane is a fully aromatic polyamide-based thin-film composite [26,27]. The properties of the two membranes are shown in Table 1. At the beginning of all experiments, the NF membranes were immersed in distilled water at a pressure of 30 bar for 10 min.

2.2. Dead-end system

A dead-end system was used to determine the optimum conditions, as shown in Fig. 1. The stainless steel dead-end filtration system has an operating volume of 300 mL and a filtration area of 14.6 cm^2. The unit employed has a circular flat sheet cell with the two halves fastened together using bolts, and it has a porous support to allow permeation. The module was applied to ion retention/rejection study in an aqueous solution. Nitrogen gas (N_2) was used to apply pressure throughout all experiments, with a maximum working pressure of 69 bar. Additionally, the solutions were stirred at a constant rate of 500 rpm to homogenize the feed samples.

The dead-end module was operated at pressure 10–30 bar to determine the optimum conditions, and the membrane flux was monitored at 1 min intervals.

2.3. Cross-flow system

A cross-flow test unit (SEPA CF, Sterlitech), as depicted in Fig. 2, was used in this study. This system was composed of the following components: a membrane module, a hydraulic hand pump, a feed tank, a high pressure pump, a balance for the measurement of flux and a computer and the necessary fittings. The stainless steel cross-flow filtration system had a total volume of 5 L and a filtration area of 150 cm^2. The system pressure was adjusted via the by-pass valve. Wastewater was pumped across the membrane cell from the feed tank using a centrifugal pump. The amount of permeate was measured by a balance, and the membrane flux was monitored at 1 min intervals.

2.4. Experimental procedure

The wastewater containing high concentration of heavy metals was derived from an aviation industry in Turkey. The characteristics of the wastewater are shown in Table 2. The wastewater was filtered using a coarse filter to remove suspended solids before it was used for the subsequent studies.

Membrane experiments were conducted with feed solutions containing nickel and chromium at concentrations of 133 ± 5 and 60 ± 3 mg/L, respectively. Wastewater containing Ni^{2+} and Cr^{6+} was passed through NF90 and NF270 membranes that were fitted to dead-end and cross-flow modules. For these experiments, permeate flux values were calculated according to Eq. (1), with permeate flux (J_w) values representing the volumetric rate of flow through the unit membrane area [28,29]:

Table 1
Characteristics of NF membranes used in experiments

NF membrane	Molecular weight cut-off (Daltons)	Charge	Temperature (max) (°C)	pH range	Active material	Supplier
NF90	~200–400	Negative	45	2–11	Fully-aromatic polyamide	Dow Filmtec
NF270	~200–400	Negative	45	2–11	Semi-aromatic polyamide	Dow Filmtec

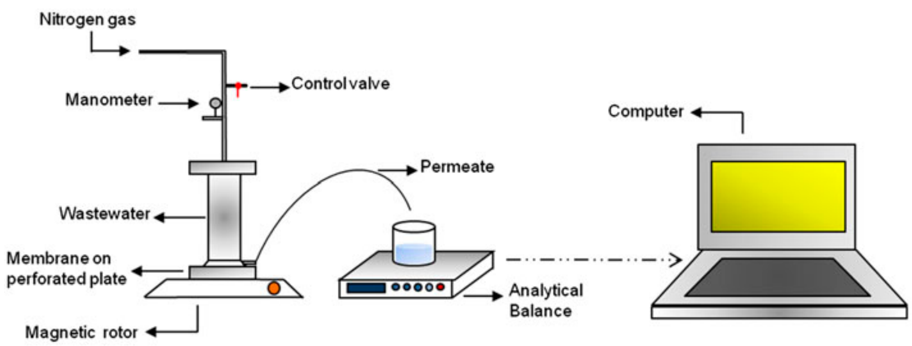


Fig. 1. Schematic diagram of the dead-end NF experimental set-up.

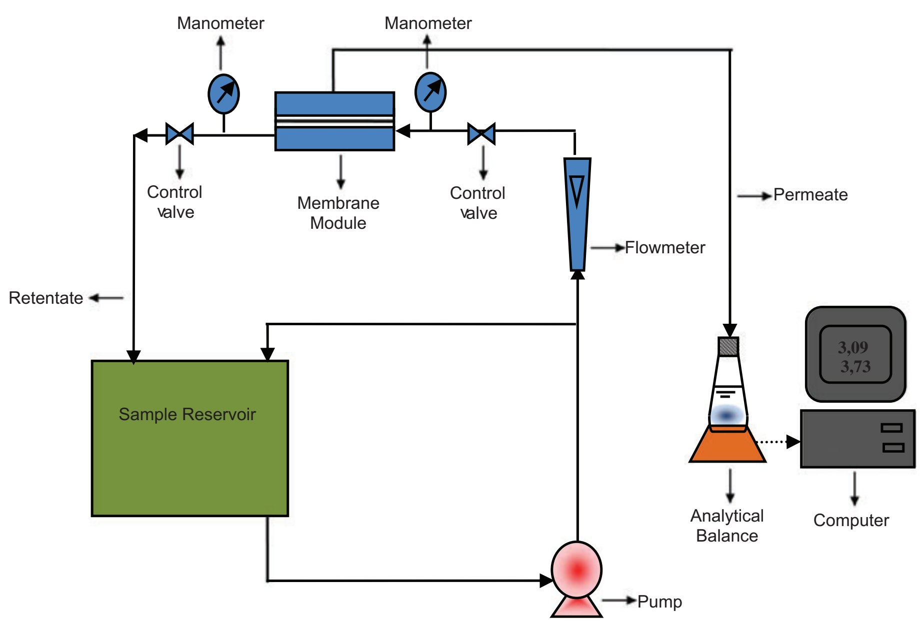


Fig. 2. Schematic diagram of the cross-flow NF experimental set-up.

$$J_w = \frac{V}{S \cdot t} \quad (1)$$

where V is volume flux (L), S is the surface area of the membrane (m^2) and t is the time (h).

Rejection % values of heavy metals were calculated as Eq. (2):

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

Table 2
Main characteristics of the aviation industry raw wastewater

Parameter	Ni^{2+} plating bath	Cr^{6+} plating bath
pH	3.44	3.66
Feed Temperature (°C)	25 ± 1	25 ± 1
Total suspended solids (TSS) (mg/L)	9.60	7.00
Ni^{2+} (mg/L)	133 ± 5	–
Cr^{6+} (mg/L)	–	60 ± 3

where C_p (mg/L) and C_i (mg/L) represent the solution concentrations in the permeate and in the initial feed solution, respectively.

Metal ion concentrations in both the feed stock and the permeate were analyzed using an atomic absorption spectrometer (Perkin Elmer AAnalyst 400AA). A Mettler Toledo pH meter was used to measure the wastewater pH. The surface morphologies of the membranes were investigated using scanning electron microscopy (SEM) (Philips XL30 SFEG) and atomic force microscopy (AFM) (Digital Instrument Nanoscope IV).

3. Results and discussion

3.1. The effect of operating pressure on rejection and permeate flux in dead-end filtration system

The wastewater containing Ni^{2+} and Cr^{6+} was filtered using NF90 and NF270 membranes at applied pressures of 10–30 bar to determine the effect of pressures on the permeate flux and the rejection. Fig. 3 shows the variation of the metal rejection (%) as a function of transmembrane pressure (ΔP) for treating Ni^{2+} and Cr^{6+} metals by NF using NF90 and NF270 membranes. As shown in Fig. 3, the rejection of nickel and chromium through the two NF membranes increased with increasing pressure. In the case of the NF90 membrane (Fig. 3(a)), the maximum rejection was obtained at ΔP = 30 bar for studied heavy metals. The rejections of Ni and Cr were the same as 95%. In the case of the NF270 membrane (Fig. 3(b)), the maximum rejection was also obtained at ΔP = 30 bar for Ni^{2+} and Cr^{6+} metals. The rejections of Ni^{2+} and Cr^{6+} were 93 and 94%, when the concentration of 133 ± 5 and 60 ± 3 mg/L, respectively. In fact, the transmembrane pressure and the metal concentration in the feed solution had no great effect on the rejection factors of the studied heavy metals. While membranes have greater permeation rates at higher pressures, the increase in pressure does not have a significant impact on the solute diffusion rate because the solute diffusion rate is primarily controlled by the solute concentration [21].

As shown in Figs. 4 and 5, the permeate flux in the NF membranes decreased with time for both metals. Initial drop occurred for all the NF membranes in the permeate flux within first 15 minutes. The flux gradually reduced after the initial drop and reached a pseudo steady-state condition within 120 min. Steady-state permeate flux values for Ni^{2+} rejection using NF90 with 10, 20, and 30 bar were

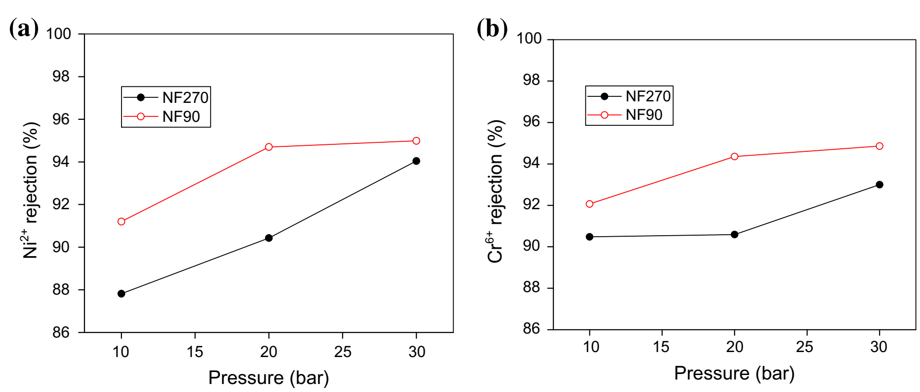


Fig. 3. Effects of pressure on rejection of (a) Ni^{2+} and (b) Cr^{6+} for NF90 and NF270 (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pH 10; time: 120 min; temperature: 25 ± 1°C).

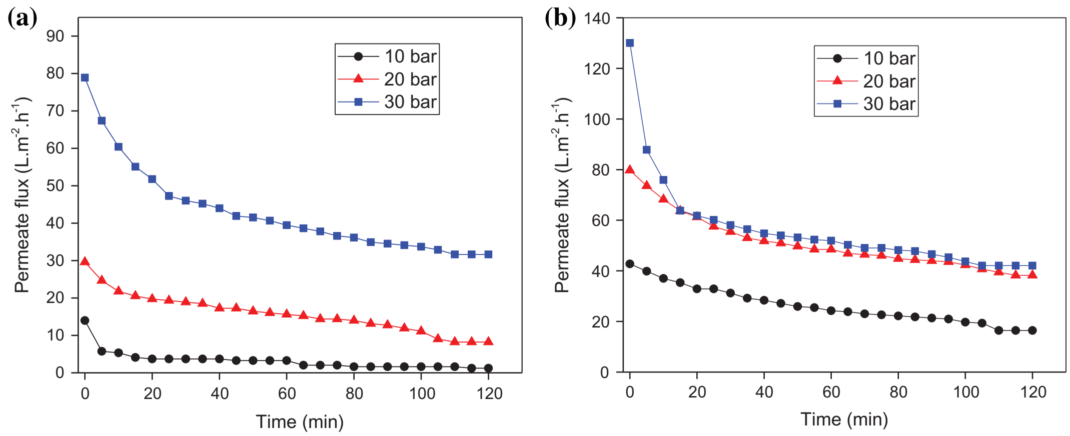


Fig. 4. Effects of pressure on flux decline of (a) Ni^{2+} and (b) Cr^{6+} for NF90 (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pH 10; time: 120 min; temperature: 25 ± 1˚C).

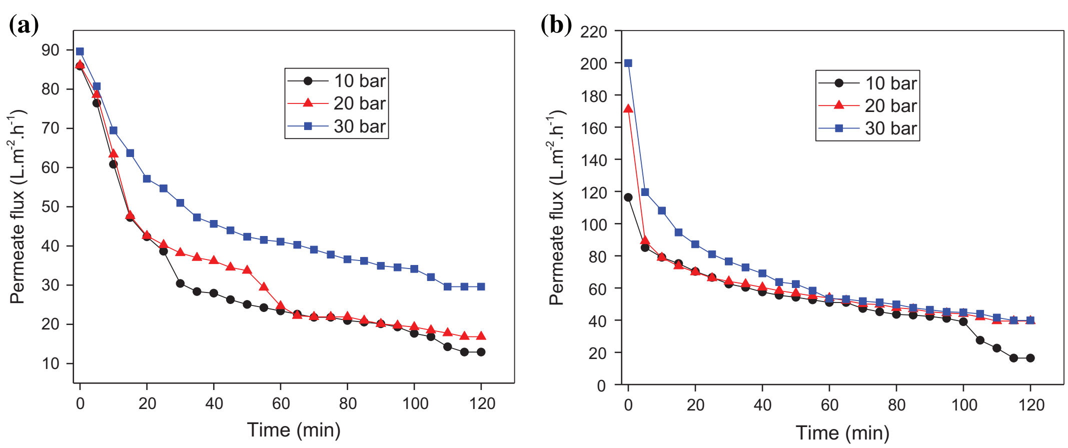


Fig. 5. Effects of pressure on flux decline of (a) Ni^{2+} and (b) Cr^{6+} for NF270 (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pH 10; time: 120 min; temperature: 25 ± 1˚C).

1.5, 9, and 32 L/m^2 h, respectively (Fig. 4(a)). However, 18, 40, and 42 L/m^2 h permeate flux values were obtained for Cr^{6+} rejection using NF90 with 10, 20, and 30 bar, respectively (Fig. 4(b)). Steady-state permeate flux values for Ni^{2+} rejection using NF270 with 10, 20, and 30 bar were 15, 18, and 31 L/m^2 h, respectively (Fig. 5(a)). However, 24, 41, and 42 L/m^2 h permeate flux values were obtained for Cr^{6+} rejection using NF270 with 10, 20, and 30 bar, respectively (Fig. 5(b)). NF90 membranes had the lowest steady-state flux value for Ni^{2+} rejection at 10 bar. NF90 and NF270 membranes yielded greater steady-state flux value for Cr^{6+} rejection than Ni^{2+} rejection at higher pressure (20 and 30 bar). Moreover, as a result of the driving forces increasing with the increased operating pressure and overcoming the resistance of the membrane, the permeate flux increased as the pressure increased [13]. This result also shows that NF270 membrane is much more sensitive to pore clogging than the NF90 membrane. We also note that in the case of both membranes, increasing the concentration of Ni^{2+} has the effect of reducing the permeate flux, as shown in Figs. 4 and 5. A similar increase in permeate flux with increasing pressure was also observed by Al-Rashdi et al. [16]. They found higher permeate flux using NF270 for various metals such as Cu(II), Mn (II), Cd(II) with increasing pressure.

Based on the experimental results, the optimum operating pressure was fixed at 30 bar; the effects of pH levels on the feed solutions were investigated at this pressure.

3.2. The effect of solution pH on rejection and permeate flux in dead-end filtration system

The effects of the feed pH on the permeate flux and the rejection parameters in wastewater containing Ni^{2+} and Cr^{6+} were determined using NF90 and NF270 membranes at pH 3.5, 7, and 10 under 30 bar.

As seen from the Fig. 6, Ni^{2+} rejection increased from 94.8 to 99.0% (Fig. 6(a)) and Cr^{6+} rejection increased from 95.0 to 96.5% (Fig. 6(b)) when pH values increased from 3.5 to 10, respectively, for NF90 membrane. With the NF270 membranes, the Ni^{2+} rejection increased from 93.0 to 98.7% (Fig. 6(a)) and the Cr^{6+} rejection increases from 94.0 to 95.4% (Fig. 6(b)) at pH values of 3.5 and 10, respectively. Urase et al. [30] also observed a similar trend using the ES-10 NF membrane for As(III) removal in the range of pH 3–10. Wang et al. [21] commented that the metal ions and the OH^- ions form insoluble hydroxides that are easily retained by the membranes when the pH feed is above 7. As a result, a higher level of metal removal is achieved.

Based on these results, the Ni^{2+} rejection increased more than the Cr^{6+} rejection as the pH value increased from 3.5 to 10. In a similar work, Maher et al. [14] explained the materials which have higher diffusion coefficient will show lower rejection values. The diffusion coefficients of nickel and chromium ions were found in the literature [31]. Subsequently, it is concluded that the rejection of nickel ions is higher than that of the chromium ions because of the difference between their diffusion coefficients (1.32×10^{-5} for nickel ion and 2.264×10^{-5} cm^2/s for chromium in water at 25℃). Therefore, Cr^{6+} could pass through the

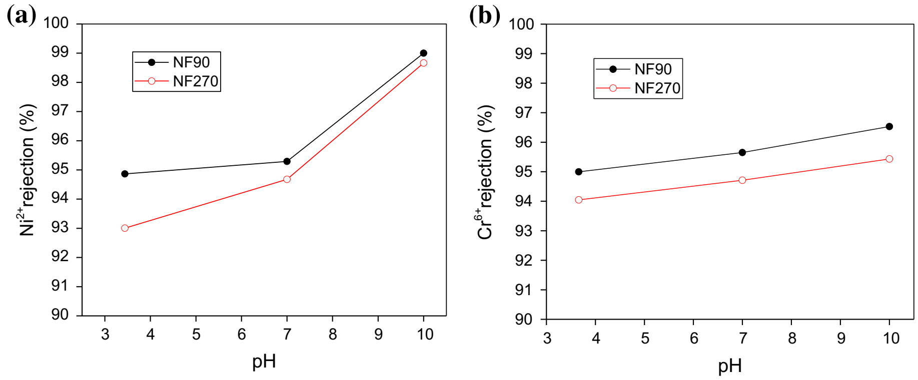


Fig. 6. Effect of pH on rejection of (a) Ni^{2+} and (b) Cr^{6+} for NF90 and NF270 membranes (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pressure: 10 bar; time: 120 min; temperature: 25 ± 1℃).

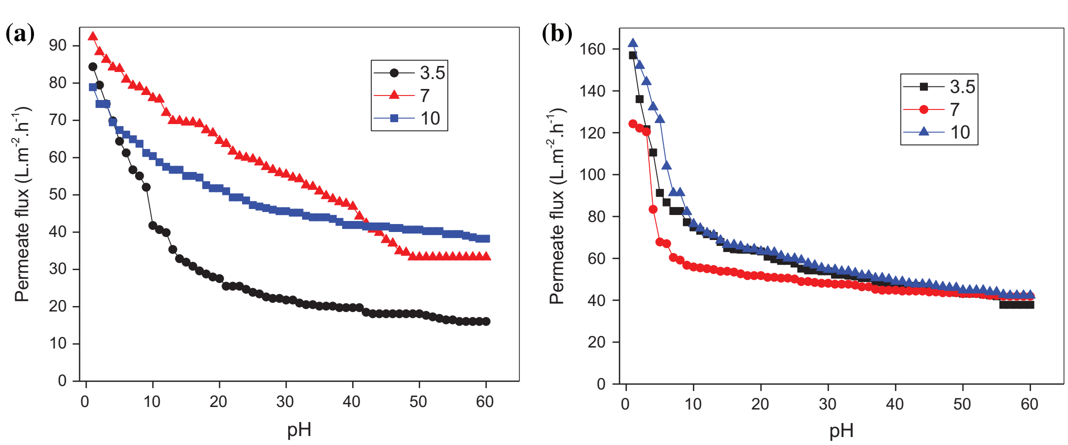


Fig. 7. Effect of pH on flux decline of (a) Ni^{2+} and (b) Cr^{6+} for NF90 (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pressure: 30 bar; time: 120 min; temperature: 25 ± 1℃).

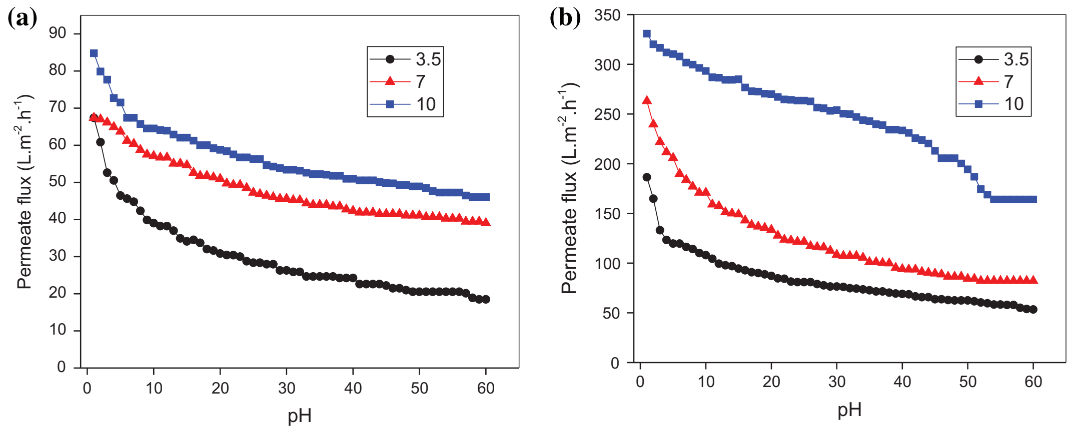


Fig. 8. Effect of pH on flux decline of (a) Ni^{2+} and (b) Cr^{6+} for NF270 (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pressure: 30 bar; time: 120 min; temperature: 25 ± 1˚C).

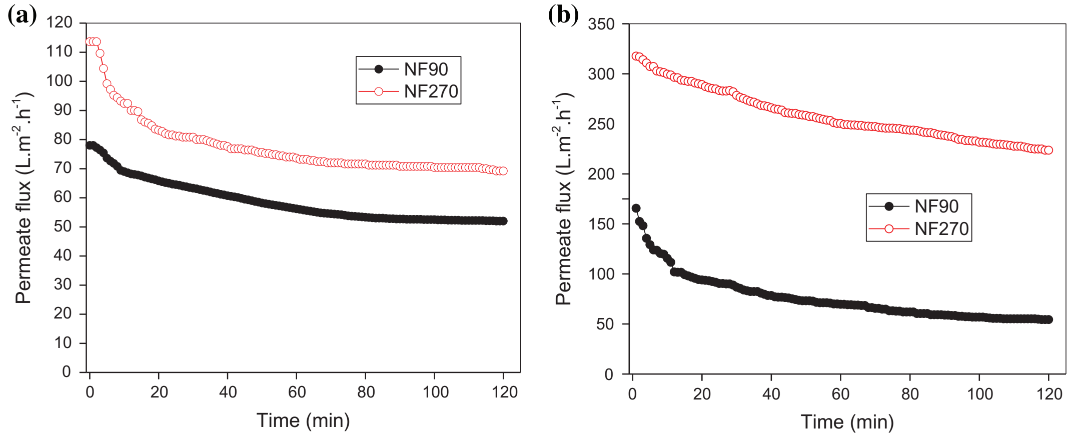


Fig. 9. Variation of flux decline as a function of time on rejection of (a) Ni^{2+} and (b) Cr^{6+} for NF90 and NF270 membranes (Ni^{2+} concentration of feed: 133 ± 5 ppm; Cr^{6+} concentration of feed: 60 ± 3 ppm; pressure: 30 bar; pH 10; time: 120 min; temperature: 25 ± 1˚C).

membrane more easily due to its higher diffusion which results in a lower rejection.

As shown in the Figs. 7 and 8, the flux gradually reduced after the initial drop and the permeate flux tends to increase with increasing pH. Steady-state permeate flux values for Ni^{2+} rejection using NF90 at pH 3.5, 7, and 10 were 16, 33, and 39 L/m^2 h, respectively (Fig. 7(a)). However, 43, 38, and 42 L/m^2 h permeate flux values were obtained for Cr^{6+} rejection using NF90 at pH 3.5, 7, and 10, respectively (Fig. 7(b)). Steady-state permeate flux values for Ni^{2+} rejection using NF270 at pH 3.5, 7, and 10 were 19, 40, and 46 L/m^2 h, respectively (Fig. 8(a)). However, 56, 82, and 164 L/m^2 h permeate flux values were obtained for Cr^{6+} rejection using NF270 at pH 3.5, 7, and 10, respectively (Fig. 8(b)). It can be clearly seen that lower pH (3.5) yielded lower steady state flux value

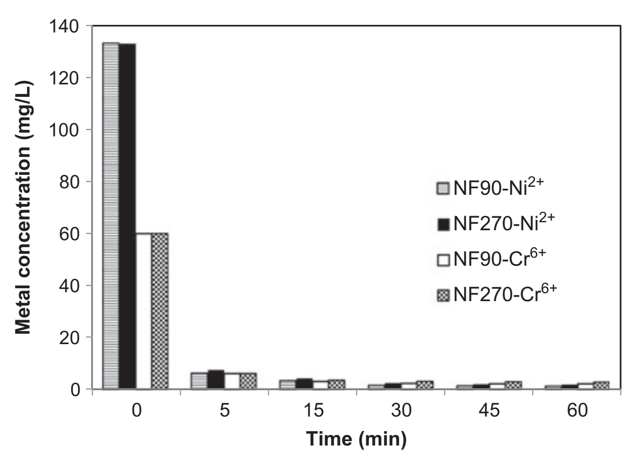
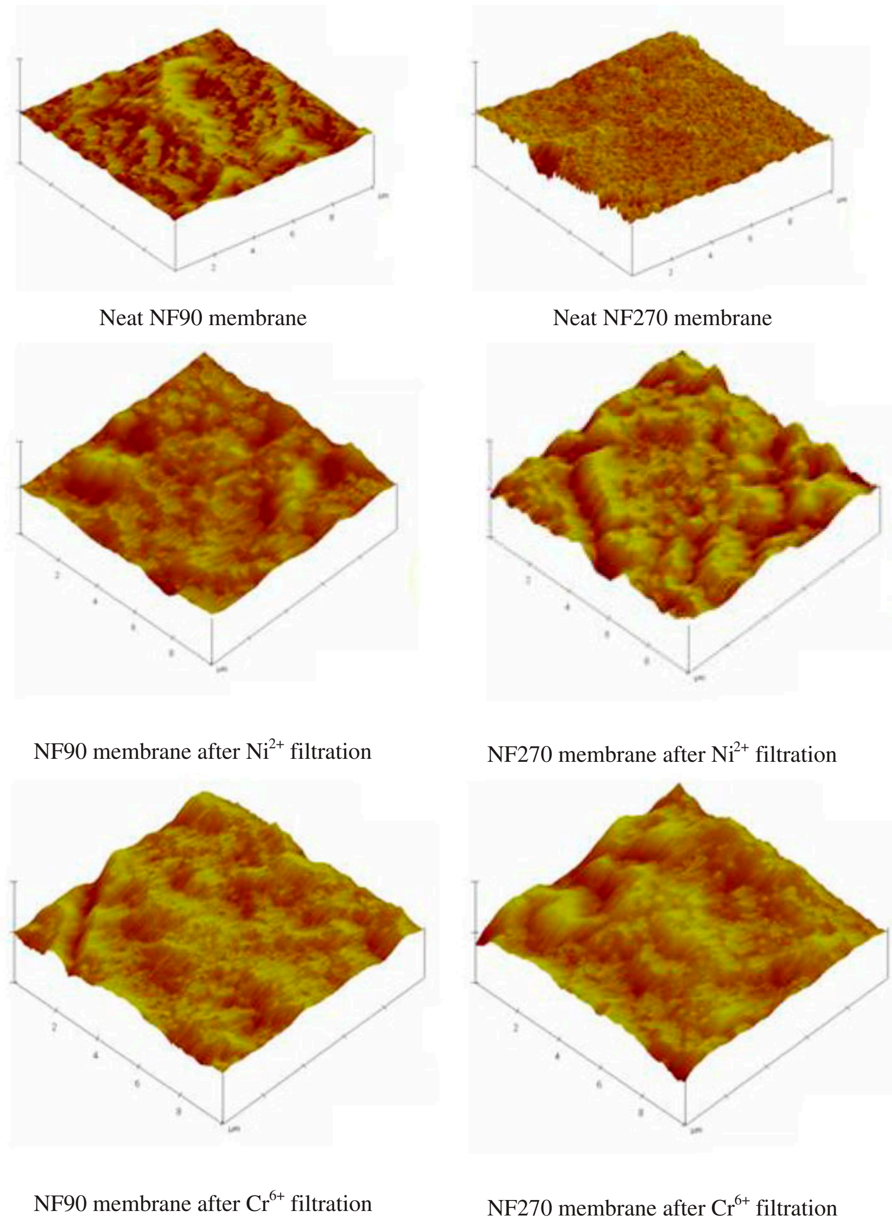


Fig. 10. Variation of metal concentration during NF process using NF90 and NF270 membranes at 30 bar and pH 10.



Neat NF90 membrane

Neat NF270 membrane

NF90 membrane after Ni^{2+} filtration

NF270 membrane after Ni^{2+} filtration

NF90 membrane after Cr^{6+} filtration

NF270 membrane after Cr^{6+} filtration

Fig. 11. AFM images of NF membranes before and after metal filtration.

for Ni^{2+} and Cr^{6+} rejection. However, higher pH (10) values obtained greater steady-state flux value and NF270 membrane yielded highest flux for all pH when used for Cr^{6+} rejection. The membrane pore size decreases with an increase in the membrane load because loaded groups adopt an extended chain configuration due to common electrostatic repulsion. Therefore, as the pH increases, the positive load decreases, which causes the membrane pore size to gradually expand and to increase the permeate flux. Similar observations have been previously reported [13,19,32,33].

3.3. Rejection and permeate flux of Ni^{2+} and Cr^{6+} in cross-flow filtration system

After determining the optimum conditions for a dead-end membrane filtration system, the conditions were applied to the cross-flow membrane filtration system. For the treatment of the industrial wastewater using NF process, an initial volume of 2.5 L was circulated through the module containing the NF90 or NF270 membrane at optimum transmembrane pressure (30 bar) and pH (10); then, 50 mL of permeate was collected every 10 min at the output of the mod-

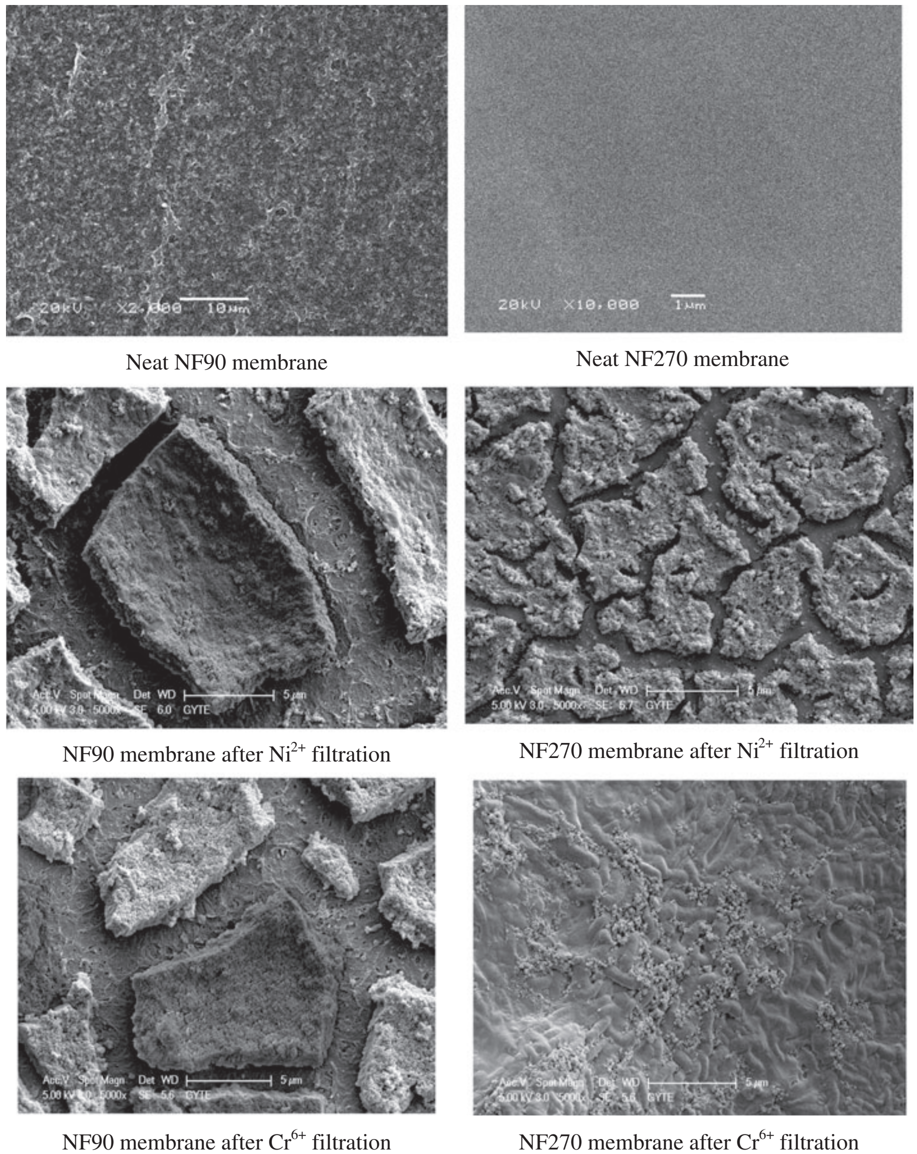


Fig. 12. SEM images of NF membranes before and after metal filtration.

ule and then analyzed. Steady-state permeate flux values for Ni^{2+} and Cr^{6+} rejection are shown in Fig. 9 using NF90 and NF270 in cross-flow filtration system operated with a pH of 10 at 30 bar of pressure. As seen from the figure, 52 and 69 L/m^2 h steady-state flux were obtained for Ni^{2+} rejection when used NF90 and NF270, respectively (Fig. 9(a)). However, 54 and 224 L/m^2 h steady-state flux were obtained for Cr^{6+} rejection when used NF90 and NF270, respectively (Fig. 9(b)). Due to its larger pore size, the NF270 membrane gave the higher flux than the NF90 membrane [27].

The concentrations of Ni^{2+} and Cr^{6+} before and after the membrane treatment are shown in Fig. 10. A considerable decrease in heavy metals (Ni^{2+} and Cr^{6+}) concentration was obtained, as shown in Fig. 10, especially in the case of nickel. According to results, rejection of Ni^{2+} under optimum conditions for NF90 and NF270 membranes were found to be 99.2 and 98.7%, respectively. Rejection of Cr^{6+} for NF90 and NF270 membranes were obtained as 96.5 and 95.7%, respectively.

In fact, 15 min of treatment was sufficient to reach 97 and 90% of nickel and chromium retention rates. However, this reduction is still insufficient to satisfy the Water Pollution Control Regulations in Turkey, which requires a total elimination of these heavy metals. This result demonstrates that a single NF using

NF90 or NF270 membrane remains insufficient to eliminate efficiently the heavy metals contained in the industrial wastewater. Therefore, we suggested to proceed with multi-stages membrane process (NF and RO) or to add ion-exchange resin in order to improve the NF performances.

3.4. Characterization of NF Membranes

AFM analysis was performed to examine the surfaces of the membranes, as fouling is a problem frequently encountered in the membrane process. Fouling frequently results in a decrease in both membrane flux and the efficiency of membrane separation. Fouling increases as the roughness of the membrane surface increases; therefore, membranes with smooth surfaces are preferred. The AFM images of the NF membranes are shown in Fig. 11. The roughness values of the NF90 and NF270 membrane surfaces for Ni^{2+} were found to be 64.42 and 75.41 nm, respectively. For Cr^{6+}, the roughness values were found to be 36.81 and 41.87 nm, respectively. Based on these results, the NF90 membrane was found to be smoother than the NF270 membrane, thus explaining the higher rejection values for the NF90 membranes compared to those for the NF270 membranes for both metals. The same result was found by Yüksel et al. [27] for bisphenol A (BPA) removal using NF90 and NF270 membranes.

SEM analyses were conducted to determine the clean and contaminated surfaces of the membranes (Fig. 12). According to the SEM images obtained after the filtration process, the membrane pores were filled and resulted in fouling of the NF membranes, effectively reducing the pore size.

4. Conclusions

In this study, the ability of NF90 and NF270 membranes to remove Ni^{2+} and Cr^{6+} from wastewater obtained from metal plating in the aviation industry was investigated. The effects of the operating pressure (10–30 bar) and the feed pH (3.5–10) on the performance of the membranes were studied. As the pressure increased, both the permeate fluxes of nickel and chromium for two NF membranes increased. However, the pressure had no significant effect on the rejection of the heavy metals studied in this work. The rejections and the permeate fluxes of the NF membranes increased with the rise in the feed pH. The Ni^{2+} rejection increased more than the Cr^{6+} rejection as the pH value changed from 3.5 to 10. The membrane rejection values for both metals were over 95% using cross-flow filtration system.

The NF270 membrane gave the higher flux than the NF90 membrane because of its larger pore size. Another finding from the present work is that the steady-state flux in cross-flow filtration system was higher than in the dead-end filtration system.

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