Evaluation of the nanofiltration membrane technology for copper removal from copper mine effluent stream

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Membrane separation is a highly effective water treatment method with numerous applications in mining. It offers several potential benefits to the mining industry, including significant water recovery, excellent metal upgrades, and reagent recycling from process streams. This paper aims to evaluate the use of membrane technology for copper recovery and recycling clean water from an aqueous effluent stream generated by copper mines. The study tested six commercial flat-sheet membranes at a laboratory scale and assessed their performance and the impact of various process parameters. The study revealed that nanofiltration technology is an efficient method to treat wastewater generated by copper mines. Five of the six evaluated membranes (AMS, A3011, A3012, A3014, B4021, and DOW N90) rejected more than 90% of copper into the concentrate at operating pressures of 20, 30, and 40 bar at ambient temperatures. While an increase in operational pressure improved operational flux, the study found that the rejection of copper and other metals decreased with an increase in pressure and temperature. Moreover, the study emphasised the need to find the optimum operating temperature as higher temperatures could compromise copper rejection. Optimising operational parameters of DOW N90 showed that increasing operational pressure improved operational flux, increased copper rejection, and minimised flux depreciation over time. Overall, this study highlights the potential of membrane technology in copper recovery and clean water recycling from mine wastewater.

Keywords: Copper, Effluent; Membranes, Removal, Recycle

INTRODUCTION

South Africa has a severe water shortage and is under increasing pressure to find environmentally and economically sustainable water resources. Acid mine drainage is particularly detrimental to water quality in the mining sector (Jeeten *et al.*, 2017). Hazardous metals are important pollutants today, and their disposal in waste streams must be minimised (Yasser and Ahmed, 2012). The most prevalent heavy metals include uranium, nickel, zinc, silver, lead, iron, chromium, copper, arsenic, and cadmium, which are frequently found in industrial wastewater. However, in the construction, transportation, and electrical industries, copper is considered to be one of the most valuable and widely utilised all around the world (Sajeda *et al.*, 2017).

Copper removal from industrial wastewater has been tackled in a variety of ways during the past few years (Sajeda *et al.*, 2017), including chemical precipitation (Huimin *et al.*, 2017), adsorption (Alcaraz *et al.*, 2020), electrochemical (Caprarescu *et al.*, 2014), and membrane filtration (Ab Hamid *et al.*, 2022). Membrane separation processes have progressed from basic laboratory equipment to industrial products with substantial technological as well as commercial significance (Nath, 2008). Membrane separation technologies, especially nanofiltration (NF), aim to displace more common separation techniques to lower capital and operating costs (Mortazavi, 2008).

The membrane technology offers many potential benefits to the mining industry; such as recovering and subsequently purifying water to acceptable standards; this applies to any metallurgical process (Mortazavi, 2008). Recovery and recycling of reagents such as sulfuric acid from base metals process solutions - thereby minimising the consumption of neutralising reagents required (minimises lime consumption) (Fornarelli and Mullett, 2014; Archer *et al.*, 2014); and metal separation and concentration; separation of copper from cyanide-containing solutions with an upgrade of the latter (Lien, 2008).

Membrane filtering techniques have been researched in recent years for a variety of uses, such as desalination in water treatment operations and metal and reagent recovery in mining (NanoReTech Systems (Pty) Ltd, 2014; Van der Bruggen *et al.*, 2008). Numerous case studies involving membrane separation procedures in the mining industry have been published in the past 10 to 15 years.

Membrane technology is now widely used in industrial applications, because of the minimal pollutants it produces. As a result, the Best Available Technologies manual for wastewater treatment encourages their use. There has been a rise in the use of membranes as a result of the development of new membrane processes and new membrane materials that are tailored to specific process specifications (Staszak and Wieszczycka, 2023).

Effluents and wastewater from copper mines and processing plants contain varying amounts of copper. To comply with environmental regulations and reclaim copper as a valuable metal that can be sold for additional revenue, waste should be treated for copper removal. To remove heavy metals from water, particularly copper, more efficient and affordable technologies must be developed.

The objective of this study is to assess the efficiency of nanofiltration (NF) membranes in recovering clean water for reuse and eliminating copper from wastewater for environmental reasons. NF membrane technology is efficient and innovative for recovering valuable copper while minimizing environmental impact and operational costs. Its selectivity, energy efficiency, and versatility make it a robust approach for addressing the challenges of copper recovery from industrial effluent.

Experimental

Membranes tested

In this paper, flat sheet membranes were evaluated using a dead-end cell. The physical properties of the membranes that were evaluated are given in Table I.

Table I. Properties of the commercial membranes evaluated

Name	Company	pH range	Max temperature (°C)	Max pressure (bar)	MCOW (Da)
A3011	AMS	0-12	80	70	100
A3012	AMS	0-12	80	70	200
A3014	AMS	0-12	80	40	400
B4021	AMS	3-14	80	60	100
NF90	Dow	2-11	45	41	100-150
NF245	Dow	2-11	50	54.8	200-400

Feed composition

The feed solution used for these tests is a mine water sample originating from a closed pit copper mine. The solution had a pH of 3.87 and contained 427 mg/L copper. The analytical methods used during the test work program and their respective detection limits are given in Table II.

Table II. Analytical techniques and detection limits

Method	**Elements**	**Detection limit**
Inductively Coupled Plasma Optical-Emission Spectroscopy (ICP-OES) (base metals)	Mg, Al, Si, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Pb, Mo, SO_4^{2-},	2 mg/L for all elements, except for SO_4^{2-} which is 5 mg/L
Potentiometric titration	H_2SO_4	1 g/L
Wet chemistry	SO_4^{2-}, Cl, F	1 mg/kg, 0.03 % - 50 %, 0.01 – 30 %
Atomic adsorption spectroscopy (AAS)	Na, Cu, Ca, and K	2ppm except for Ca = 10ppm

The feed composition in Table III had relatively high-level of calcium (Ca), magnesium (Mg), and sulphate (SO_4^{2-}) and traces of aluminium (Al), cobalt (Co), iron (Fe), manganese (Mn), nickel (Ni), silicon (Si), and zinc (Zn).

Table III. ***Composition of the Cu stream***

Element	**Concentration (mg/L)**	**Element**	**Concentration (mg/L)**
K	31.4	Mg	441.1
Na	454	Mn	14
Al	8.6	Mo	<2
As	3.33	Ni	5.65
Ca	343.75	Pb	<2
Cd	<2	S	966.5
Co	1.37	Si	21.7
Cr	<2	Ti	<2
Cu	426.72	V	<2
Fe	<2	Zn	<2
Li	<2	SO_4^{2-}	3124.35

Set-up and procedure

A dead-end filtration stirred cell, as illustrated in Figure 1, was used for laboratory scale studies. Table IV lists the cell's technical parameters.

Table IV. Stirred cell characteristics and technical specifications

Parameter	**Units**	
Membrane size(diameter)	mm	53
Active membrane area	cm^2	13
Processing volume	mL	150
Maximum pressure	Bar	69
Maximum temperature	-	121 °C at 55 bar
Cell body	-	Hastelloy
O-rings	-	EPDM
Gaskets	-	EPDM
Stir bar	-	Teflon
Dimensions: Cell Height	cm	19.8
Cell Diameter	cm	4.8

This device has one membrane module that functions in the 1 to 69 bar pressure range and has an effective membrane area of 13 cm^2. A 150 mL feed tank, pressure gauges, and a permeate outlet

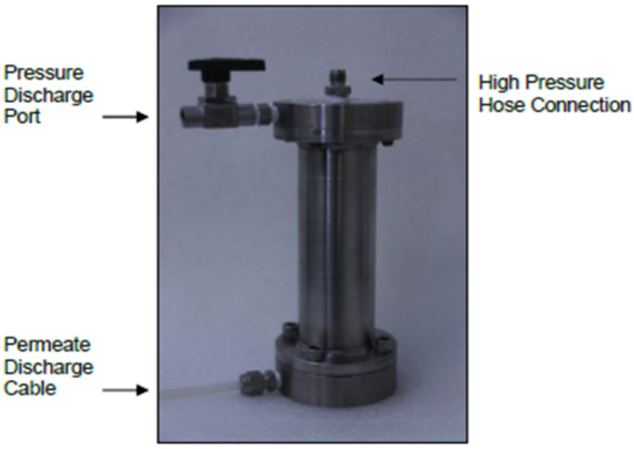

Figure 1. Laboratory apparatus for membrane filtration processes.

The experiments were conducted in the dead-end cell. The unit reservoir was filled with 100 mL of feed solution. Following that, the unit was tightly sealed to prevent pressure loss during filtration. Nitrogen gas was used to pressurise the cell and the solutions were constantly stirred at 750 rpm to homogenise the feed samples. The permeate samples were collected at hourly intervals. Upon collecting 80 % of the volume into the permeate; the test was stopped and the concentrate (retentate) was collected. The volume of the concentrate stream was measured and sampled. After completing a test, the membrane was removed from the module and rinsed with deionised water. Figure 2 shows the different solutions before and after filtration.

Figure 2. Solutions generated during filtration feed, permeate (P), and concentrate (C).

RESULTS AND DISCUSSION

Nanofiltration screening tests

At operating pressures of 20, 30, and 40 bars, the performance of various membranes were compared. Equation 1 was used to calculate the permeate flux (J), which is defined as the amount of permeate per unit of time per unit membrane area (L/h.m^2) and thus indicates how quickly the permeate passes through the membrane (Nel *et al.*, 2013). The permeate flux profiles generated during the evaluation of membrane performance at 20, 30, and 40 bars are presented in Figure 3.

$$J = Q/A \quad [1]$$

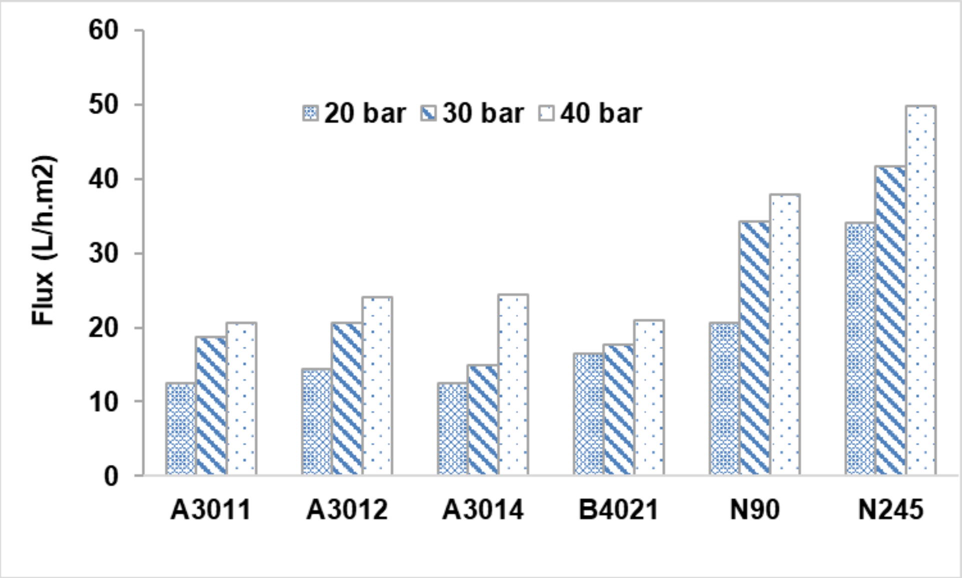

Figure 3. Nanofiltration membrane real solution permeate flux profiles average.

In all the cases, the permeate flux decreased with the volume passed through the membrane. DOW membranes (N245 and N90) membranes showed the most rapid decline in flux among all other NF membranes studied. AMS membranes (A3011, A3012, A3014 and B4021) showed a gradual slow decrease in flux. This decline in flux was possibly attributed to the initial deposition of the contaminants on the membrane surface or fouling, although significant fouling was not observed when membranes were removed from filtration equipment. Further decline in flux could be due to increased concentration of copper and other cations and anions in the concentrated phase as the filtration progressed.

The rate of the flux decrease can characterise the susceptibility of a membrane to degradation or fouling. However, a long-term evaluation in real conditions is required to confirm this suspicion. For direct comparison of different membranes operated at various pressures, the permeate flux was calculated for the time required to collect ~80 % of the feed into the permeate stream. It can be seen that for all membranes evaluated, an increase in operational pressure resulted in higher flux. Dow NF245 membrane had the highest permeate flux of the membranes tested at the pressure of 20, 30, and 40 bar. Another factor used to characterise membrane performance was copper rejection by the membrane and it was calculated using Equation 2. Rejection/retention (Rej), is expressed as a percentage of the solute concentration removed from the system feed by the membrane and this describes the membrane's capacity to prevent substances from passing through the membrane.

$$Rej = (1 - \frac{C_p}{C_r}) \cdot 100 \qquad [2]$$

The copper rejection into retentate was obtained after passing 80% feed solution through the membrane (Figure 4). The rejection of copper was not impacted by the increase of pressure for AMS (A3011, A3012, A3014, and B4021) and DOW N90. However, DOW N245 had poor rejection of copper at an increased pressure.

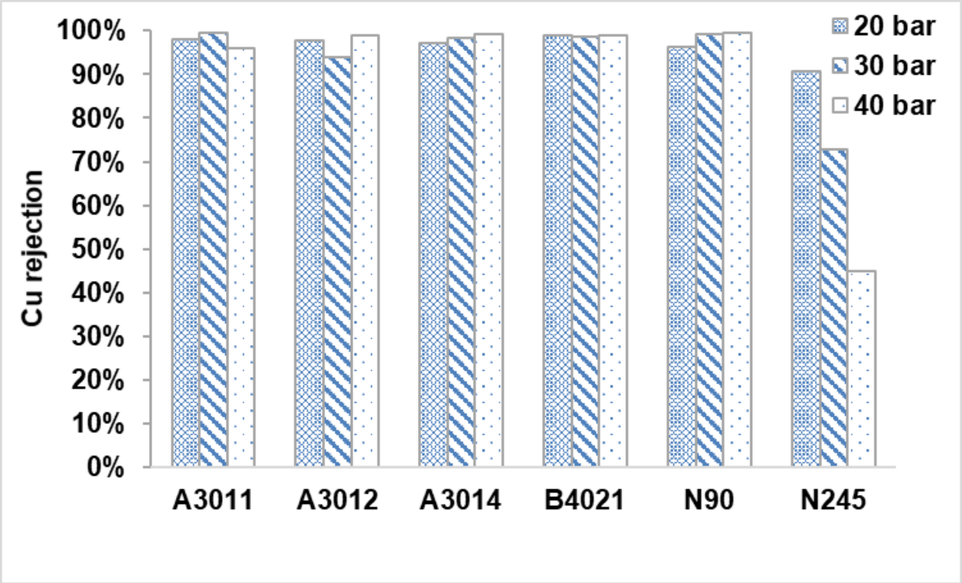

Figure 4. Copper rejection at 80 % permeate recovery for various operational pressures.

The summary of NF membrane screening tests at various pressures is given in Table V. Five of six evaluated membranes recovered more than 70-80% permeate with ≥90% copper rejection into the concentrate. The membranes successfully remove low levels of copper in streams, but they may not fulfil drinking water regulations in a single filtration step. According to the recommendations, the maximum permitted concentration of copper ions in drinking water is 2 mg/L(U.S., 2002). High copper levels can be toxic, hurting the nose and throat when inhaled and causing nausea, vomiting, and diarrhoea when consumed. Severe copper exposure can cause liver and kidney damage, as well as death (U.S., 2002).

Combined copper permeate passing through the membranes at tested pressure was not impacted by the pressure, except for DOW N245; an increase in pressure also increased the amount of copper passing through the membrane.

Table V. Summary of NF membrane screening tests at tested pressures

20 bar						
	Permeate		**Copper**			
Membranes	**Recovery**	**Flux**	**Combined permeate**	**Upgrade**	**Retentate**	**Copper rejection**
	%	L/h.m^2	mg/L		mg/L	%
A3011	85%	12	9.34	4.3	1820	98%
A3012	83%	14	12.18	4.2	1780	98%
A3014	80%	12	14.46	3.5	1510	97%
B4021	77%	16	6.37	4.3	1830	99%
N90	80%	21	19.32	3.7	1600	96%
N245	84%	34	24.72	4.3	1840	91%
30 bar						
	Permeate		**Copper**			
Membranes	**Recovery**	**Flux**	**Combined permeate**	**Upgrade**	**Retentate**	**Copper rejection**
	%	L/h.m^2	mg/L		mg/L	%
A3011	86%	19	3.38	4	1710	99%
A3012	82%	21	29.8	3.8	1610	94%
A3014	81%	15	9.64	3.7	1560	98%
B4021	82%	18	7.6	4.3	1830	99%
N90	84%	34	3.94	4.9	2100	99%
N245	78%	42	139.7	3.5	1510	73%
40 bar						
	Permeate		**Copper**			
Membranes	**Recovery**	**Flux**	**Combined permeate**	**Upgrade**	**Retentate**	**Copper rejection**
	%	L/h.m^2	mg/L		mg/L	%
A3011	80%	21	20.46	3.9	1660	96%
A3012	80%	24	4.93	4.8	2050	99%
A3014	80%	24	3.53	4.1	1740	99%
B4021	81%	21	6.27	5.7	2450	99%
N90	71%	38	2.42	4.8	2035	100%
N245	84%	50	286.12	1.6	703	45%

One membrane was selected to conduct an optimisation test DOW N90 at an optimum pressure of 40 bar with a high flux of 38 L/h.m^2 and less than 3 mg/L copper passing through the membrane with 100 % copper rejection. The additional reason for using the DOW N90 membrane is also its availability in different configurations.

Optimisation of copper removal via membrane process

The effectiveness of membrane separation operations is influenced by the operating conditions as well as the chemical characteristics of the membrane (Nel *et al.,* 2013). For this reason, additional tests were conducted to ascertain the effects of temperature, pressure, pH, and the rate of agitation inside the cell to support final recommendations for the operation of the laboratory membrane testing unit (which operates in the dead-end configuration mode). Several operational conditions must be considered when constructing an NF process. The following are the primary operational elements that affect NF membrane performance.

Agitation inside the cell

For the laboratory tests, the cell was stirred to minimise the effect of concentration polarisation that occurs during dead-end filtration. On a commercial scale, cross-flow filtration would be employed and this parameter will not be relevant. The effects of stirring on the permeate flux and copper rejection were evaluated by varying the agitation speed. The following speeds were tested: 250, 500, and 750 rpm. This test was done at ambient temperature and a pressure of 40 bar; the feed solution containing 427 mg/L copper was passed through the membrane until 80% feed solution.

An increase in agitation speed improved overall permeate flux with time; it had a positive impact on a decrease in the rate of fouling. Because in the dead-end filtration configuration, the permeate solution moves perpendicular to the membrane, and with time the membrane pores clog (effect from concentration polarisation). Mixing the solution during filtration can reduce the amount of clogging, enhancing NF separation (Lenntech BV, n.d.). At 250 rpm the combined copper leakage into the permeate was 4 mg/L, at 500 rpm 1.97 mg/L, and 750 rpm 1.85 mg/L. An increase in agitation speed s minimises the leakage of copper, in this case, and the optimum agitation speed for the operation of the membrane test cell was found to be 750 rpm.

Operating pressure

The operational pressure was varied at 10, 15, 20, 25, 30, 35, and 40 bar to determine its impact on the permeate flux and copper rejection. All other parameters were kept constant, as stated by Bastos *et al.*, in 2009, at lower operational pressures, a diffusive transport of metal is responsible for the reduced rejections, but at higher pressures, convective transport of salts through the membrane takes control (Bastos *et al.*, 2009). In this study, despite a change in the transport mechanism across the membrane DOW N90, the permeate flux and copper rejection into retentate increased linearly with pressure, and copper passing through the membranes at tested pressures was not impacted.

Temperature

The impact of temperature on the flux and copper rejection in the 25, 30, 35, and 45°C range for DOW N90 was investigated. The operational pressure and agitation were kept constant at 40 bar and 750 rpm, respectively. The permeate flux increased with temperature, most likely due to a decrease in viscosity of the liquor at temperature and a faster diffusion rate of feed solution through the membrane pores. The increase in flux may also indicate membrane pore expansion due to temperature increase (Goosen *et al.*, 2002).

The temperature was found to be another important parameter affecting the concentration of copper in permeate. An increase in temperature increases the amount of copper passing through the membrane, at 25°C combined permeate passing was 8.31 mg/L, at 30°C 13.49 mg/L, at 35°C 29.60 mg/L, and 45°C 50.84 mg/L. An increase in temperature also impacted copper rejection inversely, increasing the amount of copper passing through the membranes.

Copper rejection into the retentate decreased from 98% at 25°C to 90% at 45°C. A decrease in copper rejection was most likely caused by an increase in the rate of copper diffusion through the membrane pores. This could be because pore size changed as the temperature rose. At the conditions tested, the optimum temperature for the separation of water and copper was determined to be 25°C.

Impact of pH on copper rejection and permeate flux

The removal and recovery of copper are significantly influenced by the pH of the sample. To examine the differences in permeate flux at various pH ranges, the input parameters were kept constant at a feed concentration of 427 mg/L, operating pressure of 40 bar, and speed of 750 rpm .1 M NaOH (sodium hydroxide) was used to adjust the pH. Figure 5 shows the evaluation of the pH solutions.

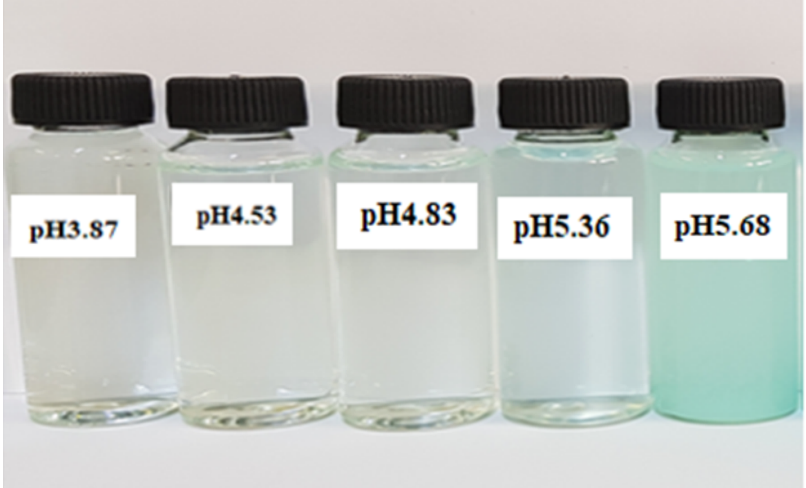

Figure 5. pH-adjusted feed solution.

The highest permeated flux was observed at pH 3.87. At higher pH, permeate flux was affected by the precipitates formed during the pH adjustment. The profiles of the copper concentration detected in the permeate at various pH levels showed that the copper concentration in permeate increased initially (when pH increased from 3.87 to 5.36) and then dropped to very low concentrations of pH 5.68 at which the Cu hydrolyses with the formation of $Cu(OH)_2$ precipitate. Higher Cu in permeate and lower Cu rejection at pH 5.36 can be due to this pH corresponding to the isoelectric point of the membrane.

Thin-film polyamide membranes (NF90) with an isoelectric point between pH 3.5 and 5.0 have amphoteric properties. As a result, for pH values below the isoelectric point, the membranes have a positive charge due to the protonation of amine groups, whereas for pH values above the isoelectric point, the membranes have a negative charge due to deprotonation of carboxyl groups, and the zeta potential becomes more negative as the pH rises. This amphoteric property of membranes plays a significant part in NF-promoting electrostatic interactions with ionisable solutes like atenolol, for example, which can lead to drastically different performances in terms of permeate flux or rejection depending on the pH range of the stream being treated (Soares *et al.*, 2021). The rejection of copper at various pHs, more than 97% of copper was rejected due to Cu precipitation at pH 5.68.

NF fouling/ scaling test

The lifespan of the DOW N90 membrane was examined at 40 bar. Five tests were conducted using one flat sheet membrane evaluating the performance of membrane reuse. Throughout the test, flux decrease is less noticeable after five tests; after the initial decline, flux stabilised and only changed slightly during subsequent cycles. Probably cake layer formed on the surface of the membrane and the solution was filtered through this layer.

There was no loss of copper rejection in the course of Tests 1-5. The results for Test 1 until Test 5 show a stable rejection during the filtration tests. In all the tests, the rejection of copper was more than 95%. DOW N90 membrane at ambient temperature performed well while being reused, however for better accuracy more tests should be conducted for better conclusions concerning its life span and in different conditions.

CONCLUSION

Recent developments in nanofiltration have resulted in the technology being seriously considered for use by the mining industry for effluent treatment. Membrane systems provide opportunities to treat dilute streams and recover water in the copper mines. This process provides benefits not only from an economic point of view but also in terms of environmental factors such as reduced quantities of waste from neutralisation.

The performance of six commercially available NF membranes from two different producers (AMS and Dow) was evaluated using a copper dilute stream generated in a copper mine to recover copper and clean solution. The evaluated membranes were found to reject about 90% of copper into the concentrate with > 80% of permeate at an operating pressure of 20, 30, and 40 bar at room temperature. Of all the evaluated membranes, an increase in the operational pressure increased operational flux. However, an increase in pressure and temperature decreases the rejection of copper and other metals. Pressure optimisation tests are required to find the optimum conditions for the membrane operation since the energy requirements of the NF process are directly linked to operational pressure

Regarding the NF membranes tested, the Dow NF 90 membrane showed better potential for the treatment of a copper dilute stream compared to the other membranes. A3011, A3012, A3014, and B4021 behaved similarly. Hence, the Dow N90 membrane was evaluated further at different parameters, and optimisation work was done to improve the performance of the membranes. During the optimisation of the DOW N90 parameters, high flux values with copper rejection > 90% at 40 bar ambient temperature were observed.

This study suggests that membrane technology can be applied in a copper mine to minimise freshwater demand; it is recommended that further tests should be conducted to explore this technology for mine waste treatment.

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