Evaluation of Nanofiltration and Reverse Osmosis in the Treatment of Gold Acid Mine Drainage

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ABSTRACT

With the rising restrictive discharge standards, the reuse of effluents such as acid mine drainage (AMD) has become a viable option both environmental and economically. Membrane separation processes especially nanofiltration (NF) and reverse osmosis (RO) produces a permeate with high quality, which can usually be reused in the process. Therefore, the purpose of this study was to compare nanofiltration and reverse osmosis treating an acid mine drainage effluent from Minas Gerais, Brazil. Initially, a pretreatment study was conducted to compare the performance of the NF when operating with raw effluent and two pre-filtered effluents: one with qualitative filter and another using microfiltration. The performance was evaluated in terms of fouling and effluent quality (ions concentration, total solids and conductivity). The results showed that the type of pretreatment does not considerably affect membrane fouling, and effluent average flow was between 73 and 86% of the flow with water. The retention efficiency of major contaminants was very high: greater than 79% for solids, 98% for conductivity and 99% for sulphate. Moreover the pretreatment improved effluent quality in all aspects analyzed. Secondly, a comparative study of three membranes of NF and two membranes of RO was conducted. The membranes of NF analyzed were NF90, NF270 and MPS-34. The membranes of RO analyzed were TFC-HR and BW30. All five membranes showed high pollutants retention (sulphate >99% and conductivity >84%). The pollutants retention efficiencies of nanofiltration membrane (NF 270) were comparable to the retention efficiencies of the RO membranes; yet it was associated with the highest permeate flux (10 and 88 L/h.m² for RO and NF membrane respectively).

Keywords: Gold acid mine drainage; Nanofiltration; Reverse osmosis

INTRODUCTION

Gold is a substance of extreme important as monetary reserve and much of what was produced throughout history is now stored on national central banks or National Treasury of many countries. Its importance is also associated with the production of jewelry and its use in the electronic industry as well as its demand for dental and medical purposes (GFMS, 2014). Nevertheless, despite the clear economic benefits, exploitation and processing of gold are associated with various environmental impacts. Acid mine drainage (AMD) is one of the main environmental impacts related to gold mining, due to the difficulty of AMD control once initiated, the high volumes involved and associated treatment costs, and its perpetuity.

Mining effluents are usually treated by neutralization, precipitation and sedimentation (Langsch et al., 2012), although other technologies such as anaerobic bioreactors (Wildeman et al., 2006), sorption (Acheampong & Lens, 2014), coagulation and flocculation (Oncel et al., 2013), and crystallization (Fernández-Torres et al., 2012) may also be used. However, these methods may be insufficient to adjust the effluent properties to meet the discharge and/or reuse standards, require high consumption of chemical products, and may generate large sludge volumes (Wang et al., 2007).

Membrane separation processes (MSP) are promising technologies for treating acid mine drainage (AMD). According to Habert et al. (2006), MSP are technologies that use a selective barrier (membrane) that under a driving force can promote the separation of the components of a solution or suspension. Membrane separation process has significantly developed over the past few years, since it has unique characteristics compared to conventional separation processes. Some example of MSP characteristics are that they do not need phase transition to perform components separation (as in distillation), thus contributing to energy savings; do not require the addition of chemicals (as in liquid-liquid extraction); have high selectivity; can be easily scaled since they are modular; do not require extensive workforce, etc.

Among the MSP, nanofiltration (NF) and reverse osmosis (RO) stand out. These process are effective technologies to remove salts and metals from aqueous medium (Al-Rashdi et al., 2013) presenting high potential to treat mining effluents for reuse.

NF process is an intermediate process between RO and ultrafiltration (UF) that may retain dissolved molecules with molecular weight ranging between 200 and 1.000 g/mol and multivalent ions (Yu et al., 2010). Many works have shown that NF is an efficient system for secondary or tertiary treatment of effluents intended to supply water for industrial, agricultural and/or indirect reuse as drinkable water (Acero et al., 2010). The use of NF has been increasing due to advantages such as the ease of operation, reliability, low power consumption and high efficiency (Fu & Wang, 2011).

RO systems use membranes that are permeable to water, but substantially impermeable to salts, and therefore are suited to separate ions, dissolved metals and organic molecules of low molar mass (Baker, 2004). One of the main applications of RO membranes is seawater desalination, which accounts for more than 20% of all desalinated water supplied around the world (Fu & Wang, 2011). Moreover, RO membranes have become a promising technology for industrial effluents reclamation (Qi et al., 2011; Kurt et al., 2012).

Sierra et al., 2013 studied nanofiltration process for treating an acid mine drainage from an abandoned mercury mine. It was able to remove up to 99% of aluminum, iron and arsenic content, and 97% of sulfate content. Chan & Dudeney, 2008 evaluated the treatment of wastewater from gold ore bioleaching treated by neutralization, precipitation and sedimentation followed by a post-treatment with GE Osmonics TFM-100 RO membrane. It was found that more than 90% of the arsenic that had not been removed by the first treatment was retained by the membrane. Another study evaluated NF and RO membrane performance to treat two synthetic acid mine drainage, including one with higher metal content (Al-Zoubi et al., 2010). It was found that NF membrane was more suited for such use as it handled higher permeate flux at lower power consumption, although its rejections were smaller than in RO membrane treatment.

Despite the successful cases using different membranes for treating mining plant effluents, there remains the need for further examination of the most appropriate system types, that is, NF or RO, membrane selection and operating conditions such as type of pre-treatment for each specific application. Such assessment would be targeted to find ways to increase retention efficiencies, decrease membrane fouling formation, reduce costs, and optimize the whole system.

Therefore, the aim of this study was to evaluate the use of MSP in the treatment of gold AMD. Initially, a study of pretreatment requirement was conducted. It compared the performance of the NF when operating with raw effluent, an effluent pre-filtered with qualitative paper filter (Fmaia - 8μm) and an effluent pre-filtered using microfiltration. After defining the appropriate pretreatment, the operation of two RO and three NF membranes was compared and the best membranes for the system studied were selected.

METHODOLOGY

Acid mine drainage

Acid mine drainage was collected in a gold mining company in the state of Minas Gerais, Brazil which has two underground gold mines and an industrial processing plant. AMD was collected in the underground mine, on the fourth level below ground. AMD characteristics vary throughout the year; the main characteristics of the AMD used in this study are presented on Table 1.

Table 1 AMD main characteristics

AMD	pH	Conductivity ($\mu S/cm^2$)	Total solids (mg/L)	Sulfate (mg/L)	Chloride (mg/L)	Calcium (mg/L)	Magnesium (mg/L)
First collection	3.35	2,841	2,409	985	4.5	323	97
Second collection	2.74	2,744	2,926	1,406	1.6	282	125

Unit description

For the nanofiltration and reverse osmosis filtration tests a bench scale unit was used. The system is comprised of: one supply tank (ST); one pump; one valve for pressure adjustment; one rotameter; one manometer; one thermometer and one stainless steel membrane cell. Figure 1 shows a schematic of this unit.

The stainless steel membrane cell has 9.8 cm in diameter, providing a filtration area of 75 cm^2. The membranes tested were properly cut before being placed in the cell. A feed spacer was placed over the membrane to promote flow distribution. Permeate flow was measured by collecting the volume of permeate in a measuring cylinder over 60 seconds for nanofiltration tests and 180 seconds for reverse osmosis tests. Permeate was collected for analysis and retentate was returned to the supply tank.

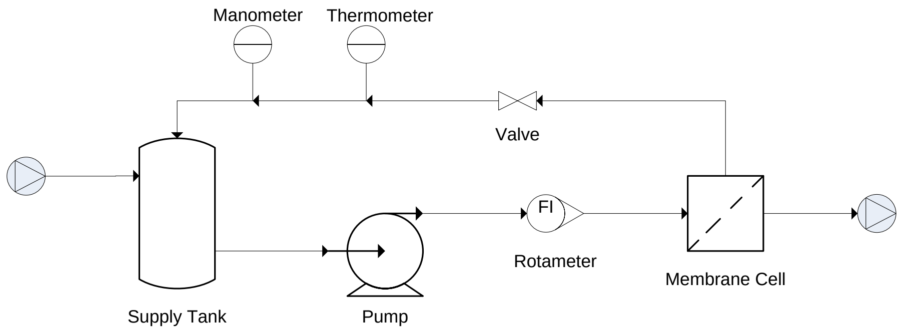


Figure 1 Schematic of NF and RO unit

Pretreatment study

Initially, a pretreatment study was conducted to compare the performance of the nanofiltration when operating with raw effluent and two pre-filtered effluents: one with qualitative filter paper (Fmaia - 8μm) and another using microfiltration. Microfiltration (MF) was performed using a submerged membrane module provided by Pam Membranas Seletivas Ltda., with filtration area of 0.04 m^2, average pore diameter of 0.4 μm and polyetherimide-based polymer. MF occurred at a pressure of 0.7 bar up to a recovery rate of 60%.

The nanofiltration tests were carried out with AMD from the first collection and used the NF90 membrane provided by the supplier Dow Filmtec. Initially, the membrane was washed in ultrasound bath first with citric acid solution at pH 2.5 followed by 0.1% NaOH solution for 20 minutes each. Water permeability at 25°C (K) was obtained by monitoring the normalized value of the stabilized permeate flux of clean water at pressures of 10.0; 8.0; 6.0 and 4.0 bar. Normalization to 25°C was accomplished by means of a correction factor calculated by the ratio of the water viscosity at 25°C and the water viscosity at the temperature of permeation:

$$K = \frac{J \cdot \mu(T)}{\Delta P \cdot \mu(25^{\circ}C)} \qquad (1)$$

Where J is the permeate flux in $m^3/h.m^2$, μ is the permeate viscosity (water) in $N.s/m^2$ and ΔP is the system pressure in Pa. With the normalized water permeability, the intrinsic membrane resistance to filtration (R_m) was calculated:

$$R_m = \frac{1}{K \cdot \mu(25^{\circ}C)} \qquad (2)$$

The nanofiltration of the acid mine drainage was carried out for two hours at fixed pressure of 10 bar, feed flow rate of 2.4 LPM and temperatures ranging between 25 and 35°C. Permeate flux and temperature were measured each 15 minutes. Final accumulated permeate was collected for analysis. Retentate was returned to the supply tank. The fouling resistance to filtration (R_f) was determined with the values of permeate flux and temperature obtained at the end of the experiment, as demonstrated by the equation:

$$R_f = \frac{\Delta P - \Delta \pi}{\mu(T) \cdot J} - R_m \quad (3)$$

Where (ΔP- $\Delta\pi$) is the effective pressure of the system in Pa and $\Delta\pi$ is the osmotic pressure difference between the retentate and the permeate in Pa. The $\Delta\pi$ was calculated using the software ROSA 9.1 (The Dow Chemical Company, USA). The initial concentration of the main ions in solution inserted in the software were: 2,000 mg/L of sulfate, 10 mg/L of chloride, 150 mg/L of magnesium and 593 mg/L of calcium. These ions concentrations were typical values obtained for the AMD.

Feed and permeates were analyzed for conductivity (Hanna conductivity meter HI 9835), total solids and ions concentrations (Metrohm 850 Professional IC, Herisau, Switzerland, equipped with column type Metrosep C4-100/4.0 and Metrosep A Supp 5-150/4.0), in accordance with the Standard Methods for the Examination of Water and Wastewater (APPA 2005).

Evaluation of different NF and RO membranes

A comparative study of three nanofiltration (NF) membranes and two reverse osmosis (RO) membranes on the treatment of AMD was conducted. The NF membranes analyzed were NF90, NF270 and MPS-34. The RO membranes analyzed were TFC-HR and BW30. Table 2 shows the main characteristics of these membranes as provided by the suppliers, unless otherwise specified.

Table 2 Membranes characteristics as provided by the suppliers

Characteristic	TFC-HR	BW30	MPS34	NF90	NF270
Supplier	Koch Membranes	Dow Filmtec	Koch Membranes	Dow Filmtec	Dow Filmtec
Membrane Material	Composite Polyamide	Composite Polyamide	Composite	Composite Polyamide	Composite Polyamide
NaCl Retention	99.55% [a]	99.5% [b]	35% [c]	85-95% [b]	n.a.
$MgSO_4$ Retention	n.a.	n.a.	n.a.	>97% [d]	97% [d]
Molecular weight cutoff (Da)	100 [e]	n.a.	200 [f]	100 [g]	200-300 [f]

n.a. Not available

[a] Feed solution containing 2,000 mg/L of NaCl, filtration at 15.5 bar, 25°C, and recovery rate of 15%.

[b] Feed solution containing 2,000 mg/L of NaCl, filtration at 4.8 bar, 25°C, and recovery rate of 15%.

[c] Feed solution containing 50,000 mg/L of NaCl.

[d] Feed solution containing 2,000 mg/L of $MgSO_4$, filtration at 4.8 bar, 25°C, and recovery rate of 15%.

[e] Reference: (Xu et al., 2005)

[f] Reference: (Wang & Tang, 2011)

[g] Reference: (Zulaikha et al., 2014)

Membrane evaluation procedure was similar to the pretreatment study procedure. First the AMD was filtered using the MF submerged membrane module provided by Pam Membranas Seletivas

Ltda., at a pressure of 0.7 bar and up to a recovery rate of 60%. All five membranes were then washed in ultrasound bath first with citric acid solution at pH 2.5 followed by 0.1% NaOH solution for 20 minutes each.

AMD nanofiltration was carried out up to a recovery rate of 10% (defined as permeate volume by initial effluent volume) at 10 bar pressure, 2.4 LPM feed flow rate and temperatures ranging between 25 and 35°C. Permeate flux, temperature and permeate accumulated volume were measured from time to time and final accumulated permeate was collected for analysis. Retentate was returned to the supply tank.

Feed and permeates were analyzed for conductivity (Hanna conductivity meter HI 9835), total solids and ions concentrations (Metrohm 850 Professional IC, Herisau, Switzerland, equipped with column type Metrosep C4-100/4.0 and Metrosep A Supp 5-150/4.0), in accordance with the Standard Methods for the Examination of Water and Wastewater (APPA 2005).

RESULTS AND DISCUSSION

Pretreatment study

The initial osmotic pressure of the acid mine drainage calculated using the software ROSA 9.1 (The Dow Chemical Company, USA) was 0.75 bar.

The NF90 membrane water permeability (K), membrane resistance to filtration (R_m) and fouling resistance (R_f) obtained according to Equation 1, 2 and 3 respectively are presented on Table 3. The variations on water permeability are usual and often occur due to minute variations on membrane characteristics or the effectiveness of the membrane cleaning.

Table 3 Water permeability, membrane resistance, fouling resistance and average ratio of effluent flux by clean water flux of the NF90 for each of the pretreatment studied

	Water permeability ($m^3/s.m^2.Pa$)	Membrane resistance (m^{-1})	Final fouling resistance (m^{-1})	Effluent flux by water flux (%)
Raw effluent	9.6×10^{-12}	$1.2 \times 10^{+14}$	$3.6 \times 10^{+13}$	86.3
Qualitative filter	1.7×10^{-11}	$6.6 \times 10^{+13}$	$3.9 \times 10^{+13}$	73.6
Microfiltration	1.1×10^{-11}	$1.1 \times 10^{+14}$	$4.3 \times 10^{+13}$	78.5

To minimize the influences that variations on water permeability could have on the comparison of the effluent permeate fluxes, it is usual to express the ratio of the effluent flux by the water flux. The average ratios obtained are shown on Table 3. The average permeate flux of the microfiltered effluent was 26.4 $L/h.m^2$. Sierra et al., 2013 obtained with the NF2540 membrane a permeate flux of approximately 45 $L/h.m^2$ for mercury AMD at an effective pressure of 10 bar and feed flow rate of 1,000 LPM. Besides the difference in the applied membrane characteristics, the increase in feed flow rate increases the shearing forces and decreases membrane fouling which could explain the difference in permeate flux.

Moreover the small variations on fouling resistance indicate that the AMD do not cause severe fouling during nanofiltration. This is corroborated by the low initial concentrations of fouling components such as suspended solids and organic matter in the feed solution. One might think then that it is best to use directly the raw effluent. However, it is important to consider that the presence of small quantities of fouling components (such as suspended solids and organic matter) can directly increase the frequency of cleaning and maintenance and therefore reduce membrane lifespan. Moreover, an industrial effluent such as AMD varies greatly throughout the year, which could cause an increase in fouling components and consequently damage the system.

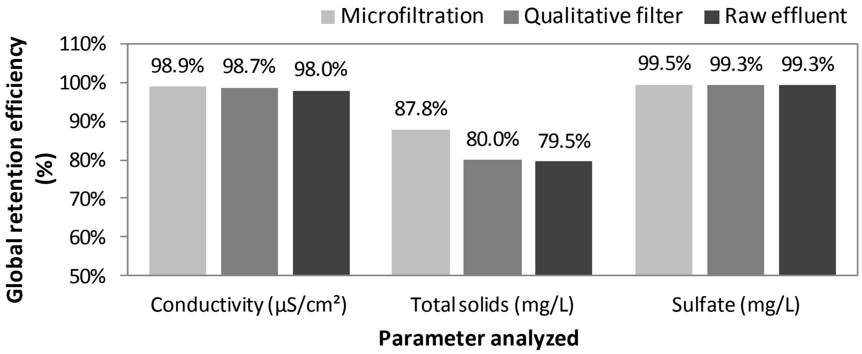


Figure 2 shows the global retention efficiencies of the main pollutants on the accumulated NF permeate for each pretreatment studied. These efficiencies comprised both the pretreatment efficiency as well as the NF efficiency and were called global retention efficiencies. All retention efficiencies obtained were very high, nevertheless it is observed that the stronger the pretreatment used, the better the final permeate quality. For instance, the total solids concentration on permeate with raw effluent was 495 mg/L, while the concentration with microfiltration was 293 mg/L, 41% smaller. The retention efficiencies for the microfiltered effluent were 98.9% for conductivity, 87.8% for total solids and 99.5% for sulfate.

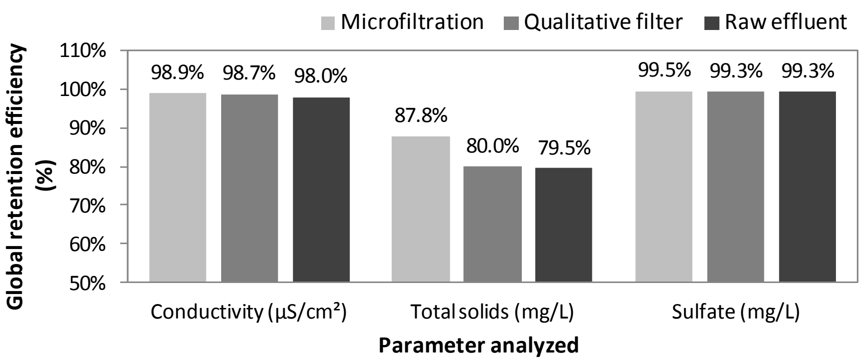
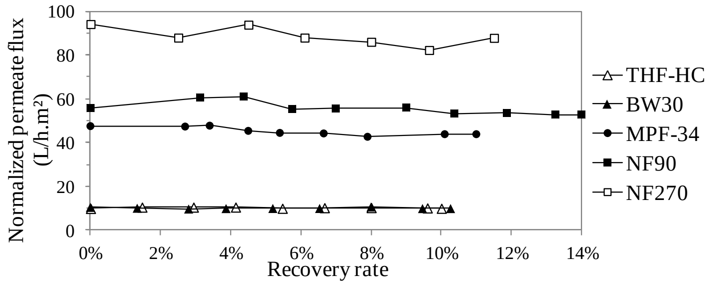


Figure 2 Pollutants retention efficiency for the three pretreatments studied

As the main objective of this treatment system is to obtain a permeate with quality appropriate for reuse as process water, the retention of sulfate and calcium must be optimized as these ions can precipitate in pipes and equipments, damaging systems. The increase in retention efficiency of sulfate corroborates that the use of microfiltration before the nanofiltration is ideal for the treatment system.

Evaluation of different NF and RO membranes

The normalized permeate fluxes for each of the five membranes as a function of effluent recovery



rate is presented on

Figure 3. The RO membranes (THF-HC and BW30) showed the lowest permeate flux during the filtration (the average permeate fluxes were 10.2 and 10.0 L/h.m² for THF-HC and BW30 respectively). These results are compatible with the structure of these membranes. RO membranes have a more closed polymeric structure, which increases membrane resistance and decreases permeate flux (Tu Nghiem et al., 2011). Among the NF membranes, the NF270 showed the highest permeate flux with average permeate flux of 88.6 L/h.m².

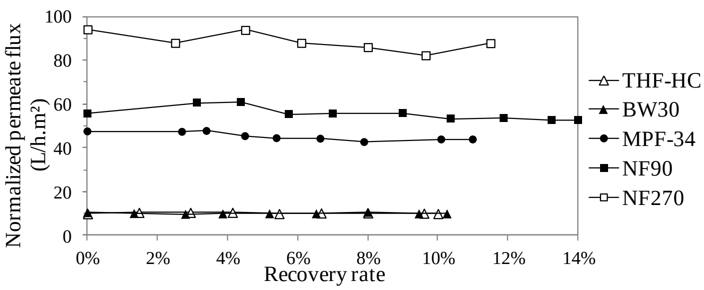


Figure 3 Normalized permeate fluxes of the five membranes as a function of effluent recovery rate

Table 4 shows the main pollutants final concentration of the accumulated permeates obtained for the five membranes tested. The final concentration of the permeate of the THF-HC membrane were the lowest for all parameters analyzed. This RO membrane is typically used on effluent treatment and water reuse due to its high efficiency (Fujioka et al., 2012). The BW30 membrane however showed higher permeate final concentration than the NF membranes studied. On the other hand pollutants concentrations of the NF membranes were only slightly higher than those of the THF-HC membrane. Permeate total solids may include other ions present in the AMD, especially monovalent ions which have smaller retention efficiencies.

Table 4 Pollutants final concentration on the permeate for the five membranes studied

	THF-HC	BW30	MPS-34	NF90	NF270
Conductivity (μS/cm²)	78	517	442	170	379
Total solids (mg/L)	38	849	295	146	207
Sulfate (mg/L)	2.2	9.7	4.1	4.5	2.6
Calcium (mg/L)	< 2.5	< 2.5	10.3	3.1	8.8

In the selection of the best membrane for a given application it should be considered the membrane area required since the cost of the NF or RO system directly depends on it. Given the low fluxes obtained for the THF-HC and BW30 membranes, its use on the treatment of gold AMD proved to be cost-prohibitive. Moreover, the pollutants retentions of the three NF membranes were similar to those of the THF-HC membrane, besides allowing for a much higher permeate flux. As a result, the NF membranes were selected over the RO membranes for this application, and among the NF membranes the NF270 was chosen as the membrane with the highest potential.

CONCLUSION

The pretreatment study showed that AMD filtration did not considerably affect initial membrane fouling resistance, indicating that the AMD initially does not cause severe fouling during nanofiltration. The average ratio of permeate flux by water flux were 86.3%, 73.6% and 78.5% for the raw effluent, the effluent filtered with qualitative filter and the microfiltered effluent respectively. On the other hand, the pretreatment of the AMD improved permeate quality in all aspects analyzed. The retention efficiencies of the microfiltered effluent were 87.8% for total solids, 98.9% for conductivity and 99.5% for sulfate.

Additionally, the five membranes studied showed high pollutants retention and the RO membrane THF-HC showed the highest pollutants retention among then. However, the NF membranes had a pollutant retention similar to the THF-HC retention associated with higher permeate fluxes. The NF270 membrane showed the highest permeate flux (88 L/h.m²) and was chosen as the most indicated for this application.

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NOMENCLATURE

K	water permeability at 25°C ($m^3/h.m^2.Pa$)
J	permeate flux ($m^3/h.m^2$)
μ	permeate viscosity (water) ($N.s/m^2$)
ΔP	system pressure (Pa)
$\Delta\pi$	osmotic pressure (Pa)
R	resistance to filtration (m^{-1})

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