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Concentration of ammonium and nitrate from mine water by reverse osmosis and nanofiltration

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Abstract

The mining industry uses large amounts of explosives and a part of them remains undetonated. Since most commercial blasting agents contain ammonium nitrate, there is a direct relationship between explosives use and the ammonium and nitrate levels in mine effluents. Water contamination with nitrogenous compounds can promote corrosion and eutrophication in receiving water bodies and be detrimental to fish.

Four commercial thin film composite reverse osmosis (RO) membranes and one nanofiltration (NF) membrane were studied in retaining ammonium and nitrate form mine water. The aim of the filtrations was to produce clean permeate and a concentrate suitable for downstream treatment in nitrifying–denitrifying bioreactors. Selection of membranes and operational pressure was performed with a plate and frame module. The membranes were then tested for continuous concentration with different volumetric reduction factors.

RO yielded good quality permeate and concentrate for bioreactor treatment. NF showed poor separation performance. With the selected RO-membrane, the retention of ammonium and nitrate was more than 82% and 90%, respectively, during the concentration. As membrane fouling can be a limiting factor a prefiltration step is recommended before the RO unit.

Keywords: Mine water; Ammonium; Nitrate; Reverse osmosis; Nanofiltration

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1. Introduction

The mining industry uses extensive amounts of explosives, parts of which remain undetonated. In Finland, the annual explosive consumption is estimated to be 6000 tons [1]. Forsberg and Åkerlund estimated that 15–19% of all loaded explosives will remain on extracted barren rock and ore [2]. Most commercial blasting agents contain ammonium and nitrate, resulting in discharge of these nitrogenous compounds to the aquatic environment nearby the mines. The fate of ammonium-nitrate fuel oil explosives in the mining operation and circuits has not yet been studied in detail. Ammonium and nitrate can promote corrosion and cause undesirable algal growths in the receiving water bodies [3]. Furthermore, already a relatively small concentration of ammonia in water can be detrimental to fish. Ammonia can exist as ammonium ion (NH_4^+) or as free ammonia (NH_3) in aqueous solutions [4]. The generated mine effluent usually contains also suspended solids, heavy metals, reducing agents, oxidants, and salts [5]. Considering the amount of suspended solids is particularly important, since they can block and foul the membranes.

Most of the undetonated explosives are dissolved in water in wet processes and are discharged from the mine. Nitrogenous compounds can also be rinsed from piled up waste rock. During the detonation of explosives, NO_X is formed. NO_X can also dissolve in water [2]. Other nitrogen sources in mine effluents can be flotation chemicals, effluents from cyanide destruction, and pH regulation chemicals [1]. In Finland, the discharge of nutrients from mining sites is supervised on a case by case basis by the environmental authorities, since legislation on discharge limits is lacking [1]. The European Union Directive 2000/60/EC for the protection of aquatic environment only demands a good state of aquatic environments to be attained. The European Commission's "Best Available Techniques for Management of Tailings and Waste-Rock in Mining Activities" reference document addresses only to a limited extent the discharge of inorganic nitrogen compounds from mining activities into the environment and does not provide a best available technique to minimize the amount of nutrients released or removal of nitrogen from mine effluents. Although attempts have been made to develop a process for the removal of dissolved nitrogen compounds from mine waters, no commercial process for ammonia nitrate removal is currently available [6]. Apart from biological treatment, the most common ways for removal of nitrogenous compounds have been ion exchange and catalytic oxidation [7]. The removal of total nitrogen from mine water by biological nitrification–denitrification has been reported earlier [6–10]. During this process, ammonium is converted to gaseous nitrogen species via nitrite/nitrate [11].

However, since the annual total volume of mine water discharged can be several million cubic meters, a concentration step for nitrogenous compounds prior to biological total-nitrogen removal is necessary. Nanofiltration (NF) and reverse osmosis (RO) are viable options as they simultaneously separate and concentrate all pollutants without modifying their molecular structure [12]. In addition, membrane separation requires less energy than many other alternatives since no phase change occurs, the equipment are compact, modular and capable for continuous operation [13,14]. When using RO or NF for separation prior to bioreactor, the aim is a clean permeate that could be discharged directly to the nature and only the nitrogenous compounds rich concentrate would be biologically treated.

The literature on the application of RO and NF for the separation of nitrogenous compounds from mine effluents is scarce. Malaiyandi and Sastri reported less than 30% retention of ammonium and nitrate by RO cellulose acetate membrane [15]. The Permasep Technical data reported for the about 80% rejection of ammonium and 85% of nitrate ions using hollow fine fiber B-9 [16]. Awadalla et al. tested four different RO membranes and two NF membranes for the removal of ammonium and nitrate ions from mining effluents. The separation was highest with SU RO membrane, for ammonium >99% and for nitrate about 97% but NF membranes could not be considered for further tests [13].

This paper reports on a study exploring the applicability of RO and NF for the concentration of ammonium and nitrate from mine water collected from a gold mine. The aim of the investigation was to produce a clean permeate and a concentrate suitable for downstream treatment in nitrifying-denitrifying bioreactors.

2. Experimental

The experiments were conducted on actual mine water from Pahtavaara gold mine. The Pahtavaara mine is situated in Central Lapland. The amount of formed water in the mine is approximately 250 000 m^3/a. The composition of the mine water is presented in Table 1. The water quality is monitored on-site and the values reported in Table 1 are representative to the values measured on-site. The electric conductivity of the effluent was 460 µs/cm and pH 7.9.

For the experiments, a DSS Labstak M20 laboratory equipment, manufactured by Danish Separation Systems AS was used. The maximum operating pressure of the equipment is 80 bar, and it can be equipped with flat sheet membranes. The effective membrane area is 0.036 m^2. It is possible to put a maximum of 20 membranes to the packing, thus increasing the total membrane area to 0.72 m^2. In the M20-filter membranes are in series. However, as the total flow rate of the permeates was much smaller than the retentate flow, the membranes were practically in parallel and feed flow composition on each membrane can be considered similar. Incoming and outgoing pressures are shown at the indicators. The feed amount to the membrane can be adjusted by changing the recirculation flow rate. The original mine water was filtered without any pre-treatment at a temperature between 12.6 and 15°C, in order to simulate the conditions in the mine.

Five different commercial membranes were tested. The characteristics of these membranes are presented in Table 2.

The experiments were performed in two stages: first a membrane selection stage and later a concentration stage. The first stage of the experiment was carried out in a batch mode, both the permeate and concentrate were returned to the feedtank. The membranes were packed in five membrane pair bundles with a control plate between each membrane pair and also below and above the package. Permeate was collected from each membrane pair. The concentrate from all membranes came out from the same tube. This design made it possible to test different types of membranes at the same time. The recirculating

Table 1
Composition of the tested mine water, concentrations in mg/L

NO_3–N	NH_4–N	Cl	Li	Na	Mg	Al	P	S	K	Ca	Mn
15.5	9.53	14.03	<0.01	28.1	15.9	<0.01	<0.01	14.6	15.0	32.2	0.04
B	Co	Fe	Ni	Cu	Zn	As	Cd	Sr	Ba	Pb	Cr
0.10	0.01	0.01	<0.01	0.02	0.08	<0.01	<0.01	0.28	0.12	<0.01	<0.01

Table 2
Technical data for membranes used in the experiments (data from manufacturers' homepages)

Membrane identification	Supplier manufacturer	Membrane type and material	Retention (%)	Operating pH range
SW30 HR	Filmtec Corp.	RO, thin film composite	99.6[a]	2–11
Espa2	Hydranautics	RO, thin film composite	99.5[b]	3–10
TFC ULP	KOCH Membrane Systems	RO, thin film composite	98.0[c]	4–11
RO1	Sepro	RO, thin film composite	98.0[d]	3–10
NF	Filmtec Corp.	NF, thin film composite	–	–

[a] Tested with 32 000 mg/L NaCl, 55 bar and at 25°C.
[b] Tested with 1500 ppm NaCl feed water solution.
[c] Nominal rejection conductivity percent.
[d] Tested with 2000 ppm NaCl feed water, with 8.6 bar pressure at 25°C.

flow rate was kept at 80% all the time. With this recirculating flow rate, the concentrate flow rate was approximately 490 L/h. This corresponds to cross-flow velocity of approximately 1.2 m/s at the surface of the membrane. The operating pressure was raised stepwise from 6 to 35 bar. The tested pressures were 6, 10, 15, 20, and 25 bar for Espa2, TFC ULP, RO1 and NF membranes. Because of the small permeate flux from SW30 HR, the samples were taken only at 15, 20, 25, 30, and 35 bar. After raising the pressure, samples were taken only after half an hour, when the conditions and flow rates would stabilize. The samples were taken from the permeate tubes and refrigerated and stored in cool if not tested immediately. The optimum pressure was chosen based on the measured fluxes and calculated retentions of electric conductivities. After the most suitable membrane and pressure value were selected, the effluent was concentrated using the selected membrane by circulating the concentrate back to the feed vessel and by removing the permeate to a separate vessel. The pressure was maintained at 15 bar. Due to the large amount of the feed water and small permeate flow, the second concentration stage was carried on as a continuous process for approximately 30 h. Samples were collected from the feed and permeate vessels at five different volumetric reduction factors (VRFs) which were calculated according to following equation:

$$\mathrm{VRF} = \frac{\text{Total feedwater amount}}{\text{Total feedwater amount} - \text{The amount of permeate}} \qquad (1)$$

The retention of electric conductivity with different VRFs was calculated according to Eq. (2):

$$R(\text{electric conductivity}) = 1\frac{C_{\mathrm{pa}}}{C_{\mathrm{c}}} \times 100\% \qquad (2)$$

where C_{pa} is the concentration in accumulated permeate at a specific VRF (mg/L) and C_{c} is the concentration in residual concentrate (feed) at a specific VRF (mg/L).

The amount of ammonium, nitrate, and chloride was measured with Hach Lange DR2800 spectrophotometer. The analysis method of nitrate is equivalent to the ISO 7150-1 method and the analysis of nitrate to ISO 7890-1-2-1986 method. The metal ion content was analyzed by using ICP-AES. The retention for ammonium, nitrate, chloride, and metal ions was calculated according to Eq. (3):

$$R = 1\frac{C_{\mathrm{pa}}}{C_{\mathrm{f}}} \times 100\% \qquad (3)$$

where C_{pa} is the concentration in accumulated permeate (mg/L) and C_{f} is the concentration in feed at the beginning (mg/L).

Membrane fouling was calculated by comparing the pure water permeability before and after the filtration as shown:

$$\mathrm{Fouling} = \left(1 - \frac{\mathrm{PWP_a}}{\mathrm{PWP_b}}\right) \times 100\% \qquad (4)$$

where $\mathrm{PWP_a}$ is pure water permeability after effluent filtration (L/m^2 h bar)] and $\mathrm{PWP_b}$ is pure water permeability before effluent filtration (L/m^2 h bar).

3. Results and discussion

3.1. *Selection of the most suitable membrane*

The results of the membrane selection stage are presented in Fig. 1. As can be seen from Fig. 1, the RO membranes had quite similar retentions based on measured electric conductivity, whereas the NF membrane had much poorer retention. In the case of SW30 HR, it can be seen that the permeate fluxes were quite small even with the highest pressure due to the tight structure of the membrane. The other three RO membranes had more similar fluxes, Espa2 had the smallest flux and RO1 the highest of these three. The retention of electric conductivity for all the tested RO membranes was more than 90% in all pressures. The retention improved as the pressure was raised; however, at higher pressure values it started to decrease slightly. The fluxes with the NF membrane were much higher than the four RO membranes had. When the pressure was increased to 20 bar, the retention of the NF membrane started to decrease because the flux became too high. With high fluxes, the concentration polarization is also larger and, therefore, the diffusion through the membrane also becomes higher. Another reason for the poor retention with the NF membrane is that it tends to let monovalent ions to penetrate the membrane. None of the membranes had a substantial influence on the permeate pH. In all cases, the permeate pH was smaller than the pH of the feed water and, therefore, all the tested membranes retained more OH^- ions than H^+ ions due to negative charge of the membranes at the applied pH.

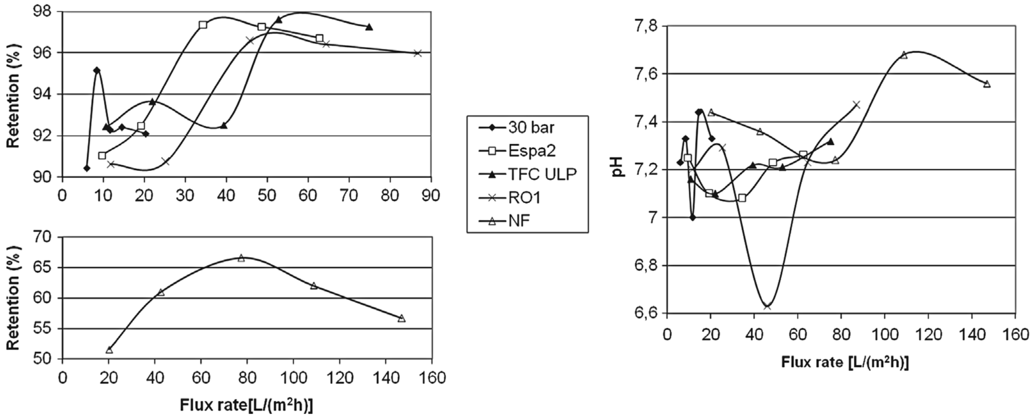

Fig. 1. Permeate flux versus retention of electric conductivity and pH of the five different membranes (pressures 6, 10, 15, 20, and 25 bar except SW30 HR 15, 20, and 25 bar except SW30 HR 15, 20, 25, 30, and 35 bar).

No direct relationship between the fluxes and pH was observed.

Based on the fluxes and retentions of the conductivity, samples from 15 bar were chosen for further analysis with Espa2, TFC ULP, RO1, and NF. The pressure chosen for the SW30 HR was 25 bar, because of the smaller permeate flux.

The results from nitrate, ammonium, and chloride analyses are presented in Table 3. The TFC ULP membrane had the best retention to NO_3–N and Espa2 to NH_4–N. Out of these two membranes, TFC ULP had a slightly better permeate flux.

The results from the permeate ICP-AES – analyses are presented in Table 3. There were no substantial differences in the ion retentions of the membranes. The NF membrane removed some of the ions similarly as the RO membranes did; however, the ions separated were multivalent. One exception was zinc (Zn^{2+}) which had a deficient retention. Poor retention can be partly due to the small size of the ion. The only

Table 3
The results from the permeate analyses for the five tested membranes

		Feed	SW30 HR	Espa2	TFC ULP	RO1	NF
NO_3–N	mg/L	15.6	0.94	0.40	0.36	0.61	11.5
	Retention (%)		93.9	97.4	97.7	96.0	24.9
Cl	mg/L	14.0	2.86	<1.30	<1.02	<1.16	5.07
	Retention (%)		79.6	>90.8	>92.7	>91.8	63.9
NH_4–N	mg/L	9.53	1.64	0.54	0.81	0.80	4.17
	Retention (%)		82.8	94.3	91.5	91.6	56.2
S	mg/L	14.6	0.83	0.07	0.07	0.24	0.17
	Retention (%)		94.3	99.5	99.5	98.4	98.8
Na	mg/L	28.1	1.96	1.60	0.67	1.85	13.7
	Retention (%)		92.0	93.3	96.6	92.4	50.7
Mg	mg/L	15.9	0.85	0.09	0.06	0.15	2.89
	Retention (%)		94.7	99.4	99.6	99.1	81.8
K	mg/L	15.0	2.50	2.13	1.77	2.21	7.04
	Retention (%)		83.3	85.8	88.2	85.3	53.1
Ca	mg/L	32.2	1.37	0.11	<0.01	0.17	7.24
	Retention (%)		95.8	99.7	99.9	99.5	77.5
Sr	mg/L	0.28	0.01	<0.01	<0.01	<0.01	0.06
	Retention (%)		95.4	>96.4	>96.4	>96.4	79.1
B	mg/L	0.10	0.02	0.04	0.03	0.04	0.09
	Retention (%)		81.4	60.8	71.1	55.7	4.1
Zn	mg/L	0.08	0.03	<0.01	<0.01	<0.01	0.07
	Retention (%)		67.5	>87.5	>87.5	>87.5	7.8
Ba	mg/L	0.12	<0.01	<0.01	<0.01	<0.01	0.02
	Retention (%)		>91.4	>91.4	>91.4	>91.4	80.8
Mn	mg/L	0.04	<0.01	<0.01	<0.01	<0.01	<0.01
	Retention (%)		>75.0	>75.0	>75.0	>75.0	>75.0
Cu	mg/L	0.02	<0.01	<0.01	0.06	<0.01	0.07
	Retention (%)		>50.0	>50.0	0	>50.0	0
As	mg/L	<0.01	0.02	0.02	<0.01	0.13	0.04
Pb	mg/L	<0.01	<0.01	0.07	<0.01	0.01	<0.01

considerable difference between the four RO membranes was in their ability to remove boron, which was retained best by SW30 HR and TFC ULP. The amount of arsenic was higher in permeate than it was in the feed water with all the other membranes except for TFC ULP. The amount of lead was also higher in the Espa2 permeate than it was in the feed water. The membrane permeation rate for these elements was higher than that of water in these cases.

The fouling percentages to Espa2 membrane was 0.4%, TFC ULP 5.3%, RO1 4.6%, and to NF 11.7%. With SW30 HR, no fouling had occurred. All of these percentages are acceptable. The selected membrane for the concentration was TFC ULP due to the membrane's rejection of NO_3–N and NH_4–N and the high permeate flux.

3.2. Concentration of mine water

In the mine water concentration stage, the feed water temperature was kept between 13.6 and 13.9°C. The effective membrane area of the used TFC ULP membrane was 0.144 m^2, which was gained by using four membrane pairs. The feed water was re-analyzed before filtrations. The five VRFs in which the samples were taken were 8.9, 10.1, 12.7, 15.5, and 20.4. With 15-bar pressure, the permeate fluxes were 49.1, 42.9, 42.3, and 44.5 L/m^2 h starting from the lowest membrane. The flux range is under 15% and, therefore, normal.

The retention calculated based on electrical conductivity was 99.0% or more with all the tested VRFs. Retention improved slightly as the feed became more concentrated and it was 99.2% with the highest VRF. Hence, the retention percentages improved compared to the membrane selection stage with the same membrane. In the permeate pH value, there were significant differences between different VRFs.

The concentration of nitrate and ammonium in permeate increased when VRF was increased (Table 4). According to these figures, the deterioration in permeate quality was only small. When the non-concentrated water was compared to the smallest tested VRF, the difference in the permeate content of NO_3–N was only about 1 mg/L and for NH_4–N slightly more than 0.5 mg/L. As the increase in the amount of

Table 4
The results from the TFC ULP membrane permeate analyses and electric conductivity measurements from the concentration stage

		Feed	VRF				
			8.9	10.1	12.7	15.5	20.4
EC	Permeate (μs/cm)	460	30.2	29.7	31.2	31.7	33.2
	Concentrate (μs/cm)		3080	3260	3680	3940	4380
NO_3–N	Permeate (mg/L)	15.3	1.33	1.38	1.48	1.47	1.54
	Separation (%)		91.3	91.0	90.3	90.4	89.9
	Concentrate (mg/L)		135	173	139	163	203
Cl	Permeate (mg/L)	15.7	2.00	<1.18	<1.03	<1.25	2.4
	Separation (%)		87.3	92.5	93.4	92.0	84.7
	Concentrate (mg/L)		105	119	138	150	161
NH_4–N	Permeate (mg/L)	9.48	1.53	1.54	1.6	1.63	1.69
	Separation (%)		83.9	83.8	83.1	82.8	82.2
	Concentrate (mg/L)		74.3	86.3	83.6	98.2	104

Table 5
Elemental composition of permeate and concentrate after the concentration stage with the TFC ULP membrane

		Feed (mg/L)	VRF				
			8.9	10.1	12.7	15.5	20.4
S	Permeate (mg/L)	14.6	0.32	0.29	0.19	0.15	0.28
	Retention (%)		97.8	98.0	98.7	99.0	98.1
	Concentrate (mg/L)		107	111	127	133	151
Na	Permeate (mg/L)	28.1	3.04	2.90	3.02	3.04	3.17
	Retention (%)		89.2	89.7	89.3	89.2	88.7
	Concentrate (mg/L)		218	228	261	278	312
Mg	Permeate (mg/L)	15.9	0.25	0.21	0.21	0.21	0.21
	Retention (%)		98.4	98.7	98.7	98.7	98.7
	Concentrate (mg/L)		122	126	143	151	169
K	Permeate (mg/L)	15.0	1.29	2.04	1.93	2.00	2.32
	Retention (%)		91.4	86.4	87.1	86.7	84.5
	Concentrate (mg/L)		104	108	123	130	146
Ca	Permeate (mg/L)	32.2	0.74	0.54	0.49	0.50	0.51
	Retention (%)		97.7	98.3	98.5	98.5	98.4
	Concentrate (mg/L)		253	259	282	283	316
Sr	Permeate (mg/L)	0.28	<0.01	<0.01	<0.01	<0.01	<0.01
	Retention (%)		>96.4	>96.4	>96.4	>96.4	>96.4
	Concentrate (mg/L)		2.76	2.94	3.43	3.59	4.21
B	Permeate (mg/L)	0.10	0.07	0.06	0.06	0.07	0.08
	Retention (%)		27.8	33.4	33.3	23.0	17.9
	Concentrate (mg/L)		0.37	0.38	0.40	0.44	0.48
Ba	Permeate (mg/L)	0.12	<0.01	<0.01	<0.01	<0.01	<0.01
	Retention (%)		>91.7	>91.7	>91.7	>91.7	>91.7
	Concentrate (mg/L)		0.92	0.95	1.05	1.06	1.15
Mn	Permeate (mg/L)	0.04	<0.01	<0.01	<0.01	<0.01	<0.01
	Retention (%)		>75.0	>75.0	>75.0	>75.0	>75.0
	Concentrate (mg/L)		0.21	0.20	0.21	0.20	0.28
Zn	Permeate (mg/L)	0.08	0.02	0.01	<0.01	<0.01	<0.01
	Retention (%)		76.5	82.5	>82.5	>82.5	>82.5
	Concentrate (mg/L)		0.34	0.29	0.28	0.19	0.47
Cu	Permeate (mg/L)	0.02	<0.01	<0.01	<0.01	<0.01	<0.01
	Retention (%)		>50.0	>50.0	>50.0	>50.0	>50.0
	Concentrate (mg/L)		0.32	0.36	0.40	0.36	0.69
P	Permeate (mg/L)	<0.01	<0.01	<0.01	0.04	<0.01	<0.01
Li	Concentrate (mg/L)	<0.01	0.03	0.03	0.03	0.03	0.04
Al	Concentrate (mg/L)	<0.01	0.47	0.33	0.35	0.34	0.39
Co	Concentrate (mg/L)	0.01	0.02	0.02	0.03	0.02	0.03
Ni	Concentrate (mg/L)	<0.01	0.08	0.08	0.09	0.09	0.11
Cr	Concentrate (mg/L)	<0.01	<0.01	<0.01	<0.01	0.01	0.02

ammonium and nitrate in permeate was not substantial, even higher VRFs could have been gained without a substantial deterioration of the permeate quality.

The increase in VRF affected only slightly the retention of most elements (Table 5). Only boron passed the membrane considerably better in the concentration stage as compared with the membrane selection stage. The results show that only copper and aluminium were enriched to the concentrate. However, the Al and Cu concentrations are far below reported values for a 50% inhibition of nitrification by cadmium, copper, and zinc, i.e. 15, 30, and 10 mg/L [17]. Therefore, heavy metals in the formed concentrate should not hinder the actions of the bacteria in the bioreactor.

Membrane fouling percentages varied from 0.4 to 4.2%, depending on the membrane's place in the package. The membrane in which the permeate flux was highest had fouled the most, and the membranes that had the lowest permeate flux rates had fouled the least. Notwithstanding, due to low fouling percentages, fouling is of minor significance.

Finally, it was observed that the salinities of the concentrates were from 2.7 to 4.0 g/L. In general, nitrifying bacteria have shown to tolerate the NaCl concentrate of 17.5–23.4 g/L [18] and denitrification bacteria the NaCl concentrate of 28–30 g/L [19]. Consequently, the formed concentrate was concluded to be suitable for biological treatment.

4. Conclusions

Membrane technology was tested for the concentration of nitrogenous compounds in mine water prior to treatment in a bioreactor. Four RO and one NF membranes were tested. All the tested RO membranes removed ammonium and nitrate adequately from the tested mine water. Of the RO membranes, Espa2, TFC ULP, and RO1 had the best performance. Espa2 gave a retention of >97.4% for NO_3–N and >94.3% for NH_4–N, TFC ULP >97.7% for NO_3–N and >91.5% for NH_4–N, and RO1 >96.0% for NO_3–N and >91.6% for NH_4–N. The NF membrane had a poor retention to ammonium, nitrate, and chloride; therefore, it is not applicable as a pretreatment process prior to a nitrification–denitrification process in a bioreactor.

In the concentration stage, the TFC ULP membrane was used. Retention of 82.2% for ammonium and 89.9% for nitrate were achieved, which are very satisfying. Heavy metals, except for aluminium and copper, did not enrich into concentrate, thus minimizing the risk of metal toxicity to microorganisms in bioreactors. Therefore, it can be concluded that RO is suitable for the concentration of mine water by VRFs higher than 20, without a considerable deterioration of the permeate quality.

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Nomenclature

C_c	concentration in residual concentrate (feed) (mg/L)
C_f	concentration in feed at the beginning (mg/L)
C_{pa}	concentration in accumulated permeate (mg/L)
PWP_a	pure water permeability after filtration (L/m^2 h bar)
PWP_b	pure water permeability before filtration (L/m^2 h bar)
R	retention of electric conductivity
VRF	volumetric reduction factor

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