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Nanofiltration investigations on tailings flotation residue wastewater for metal preconcentration and water decontamination

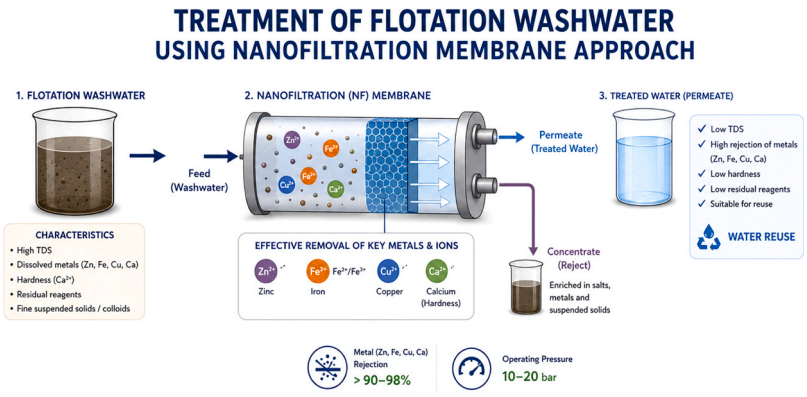
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HIGHLIGHTS

- The efficient treatment of the flotation residue waste washwater using nanofiltration membranes.
- Higher metal rejection (85–97%) and larger water flux (70–80 LMH) through the membranes were found.
- Two cycles were sufficient for the complete removal of all investigated metal ions in the permeate water.
- The pre-concentration of metal ions in the retentate, i.e Zn(II) (about 4000 mg/L), was achieved.

GRAPHICAL ABSTRACT



ARTICLE INFO

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ABSTRACT

Flotation is one of the most effective methods for processing ore and tailings. The water generated during various stages of the flotation process contains significant amounts of metal ions, which cause environmental concerns. The present study focuses on the employment of the nanofiltration membrane process with flat sheet polymeric polyamide thin film composite membranes namely DK and DL containing molecular weight cutoff of 100–300 Da, for the effective removal of metal ions such as Zn (II), Fe (II, III), Cu (II), Al (III), Cu (II), Cd(II) having concentrations of around 400 mg/L, 10 mg/L, 25 mg/L, 180 mg/L, 540 mg/L and 6 mg/L, respectively, from the flotation residue waste washwater as a function of various experimental parameters. The parameters include the effect of pH(2−3), pressure (15–20 bar), volume (2–6 L), flow rate (2–6 L/min), temperature (20–50

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°C), different membranes, and membrane stacking. In addition, we explored the process for obtaining the preconcentration of metals in the retentate for further separation. Furthermore, the number of cycles needed to complete the removal of the investigated metal ions from flotation residue waste washwater was studied in detail. The results show that higher rejection of 85–95% of multivalent metal ions was achieved for all the parameters, with concentrations decreasing in the range of 1–20 mg/L after studies for different metal ions, and complete decontamination of the investigated metal ions, i.e less than 1 mg/L, was achieved within two nanofiltration cycles at 20 bar operating pressure. The metal ion concentrations in the permeate were below the applicable regulatory limit values published in environmental policies and regulations by the WHO and German authorities. Furthermore, the preconcentration of Zn(II) reached about 4 g/L, with a 500% concentration enrichment in two cycles in the retentate studies. The nanofiltration membrane process is an efficient method for treating water from metal production or recycling, as well as for concentrating metals for further recovery. Therefore, it offers numerous advantages for the water circular economy for complete decontamination and metal recovery from metal-containing wastewater streams, to make overall tailings management more sustainable.

1. Introduction

Metals are crucial for modern civilization due to their utilization in numerous technologies, the energy sector, infrastructure development, and so on [1,2]. Metals are primarily obtained via the processing of various mining sectors through metallurgical procedures [3]. However, the continued mining and mineral processing of ores over the last few decades have resulted in a lot of dust, slag, gangue, and tailings [4,5]. Mine tailings are very fine rocks that remain after extracting valuable minerals from ore, and their production is about 9 billion tons per year in 2025 across the world [4,6,7]. In addition, mine tailings also contain process water that contains dissolved metals and chemicals [8]. The mine tailings are very important due to the fact that they contain hazardous as well as valuable metal ions with higher concentrations, and cause environmental concerns [9,10]. Mine tailings management is one of the biggest challenges for the metal mining and processing industries at the moment. The conventional tailings disposal methods are hillside dams, underground backfilling of mines, direct disposal into lakes, rivers, or the ocean, dry stacking on the land, etc. [10–12]. However, continuous accumulation at these sites leads to difficulties such as land occupancy, water, air, and soil contamination, aquifer toxicity, environmental disputes, and catastrophic dangers caused by tailings dam failures [13,14]. Therefore, proper treatment of mine tailings is essential to minimize environmental problems and the possibility of recovering valuable minerals from them for future applications [15,16].

The flotation process is one of the conventional and well-established methods used for effective recovery of various minerals from different ores and tailings processing [17–20]. It was used initially for processing sulfides; however, it is applied to all kinds of minerals or mining ores [17]. Currently, this technique is the most used in the world for processing minerals, suggesting that the tailings produced by it include a considerable amount of water and chemicals [18–21]. In the flotation process, the flotation reagents, such as collectors, frothers, and other modifiers, are used to recover the necessary minerals from mine tailings under various parameters [22]. The water generated during various stages of flotation of mine tailings, such as residue washing, dewatering of fine particles, etc., contains significant amounts of metals ranging from mg/L to g/L depending on the conditions [23,24]. For safety and economic reasons, flotation effluent must be treated so that it can be reused or utilised for further industrial or domestic purposes.

The management of water at various stages of mineral processing is continuously increasing, with the aim of a water circular economy approach and the issues caused during its disposal to the environment [25]. Treatment of mine-related waters is often seen as an end-of-pipe process aimed at producing a discharge stream that meets specified limits of acidity and concentrations of metals to be introduced into natural water streams or aquifers [26,27]. There are several methods to treat wastewater, such as neutralization, coagulation, precipitation, sedimentation, and anaerobic digestion for domestic and industrial usage [28,29]. Among all, ion exchange and membrane technologies are the most effective methods for treating mine-related waters [30–33]. Furthermore, the membrane treatment technologies, particularly reverse osmosis (RO) and nanofiltration (NF), are excellent for removing heavy metals from wastewater, allowing for compliance with severe discharge standards. These approaches are known for their great efficiency, simple operating requirements, and small geographical footprints [32,33]. Amongst these two techniques, NF is generally favoured over RO for mine-related water treatment due to its higher permeate flux at lower pressures, which consequently reduces both capital investments and operational costs. Furthermore, NF has the unique capacity to selectively concentrate and recover valuable commodity metals while avoiding the concentration of all the dissolved solids in the solution [33–35]. The literature study shows that membrane separation is used to treat both synthetic and actual mine water under different experimental parameters, such as solution temperature, operating pressure, feed flow, and solute concentration, to evaluate solute rejection and permeate flux [34,35]. In view of this, Mullet *et al.* investigated the effect of various factors on simulated or actual wastewater and discovered high rejection rates of multivalent metal ions [36]. Similarly, other authors have investigated the NF membrane performance with respect to water flow and solute rejection, and the findings show significant variations in metal rejection upon changes in the parameters [34–39]. These results highlight the complexities of NF separation mechanisms and the need for more studies to completely understand their effectiveness as well as optimized applications for the different tailings run-off and processing water. The objective of the present study was to investigate the performance of nanofiltration membranes for metal rejection from the flotation residue waste washwater under various experimental conditions. In addition, a demonstration of the fewer number of cycles necessary to get metal-free water from the flotation residue waste washwater for its reuse in various applications. The work also aimed at the development of an optimized procedure to obtain the pre-concentration of metals in the retentate at minimal experimental conditions in a lower number of cycles, for further sustainable recovery, using hydrometallurgical routes.

2. Experimental

2.1. Membranes, water samples, and reagents

The NF investigations were carried out with two different types of polymeric polyamide thin film-composite membranes, namely Desal DK and DL, acquired from OSMO Membrane Systems in Germany. These nanofiltration membranes have 96–98% $MgSO_4$ rejection and a molecular weight cut-off of 150–300 Da. In the experiments, an effective surface area of 0.016714 m^2 was used. According to the literature, both have an isoelectric point (IEP) of around pH 3 [36,40]. The waste washwater has been collected from mine tailings flotation residue obtained from Davidschacht tailings from Freiberg, Germany [41]. Three batches of the tailings flotation residues with a total mass of 42.5 kg were split into 2.5 kg samples each and washed five times (N = 5) with

Table 1
The composition of various metals in the flotation residue waste washwater.
(Since As, Pb, and In content was below 1 mg/L, they haven't been investigated further.)

Metal ions	Composition (mg/L)
Zn(II)	411 ± 46.67
Fe(II,III)	9.36 ± 7.25
Cu(II)	25.4 ± 1.63
Al(III)	186 ± 25.45
Ca(II)	540 ± 66.18
Cd(II)	6.02 ± 0.71
$[H^+]$	pH= 2.4–2.6

Table 2
XRD phase composition of flotation residue (batch 1) with 99.7% (3σ) confidence interval.

		Before washing	After washing
Mineral	**Chemical formula**	**% (m/m)**	**% (m/m)**
Quartz	SiO_2	50.8 ± 0.6	49.3 ± 0.6
Pyrite	FeS_2	13.1 ± 0.2	14.5 ± 0.3
Chlorite	$(Fe,Mg,Al)_6(Si,Al)_4O_{10}(OH)_8$	4.9 ± 0.3	4.8 ± 0.5
Muscovite	$KAl_2(Si_3Al)O_{10}(OH,F)_2$	12.4 ± 0.5	13.4 ± 0.5
Gypsum	$CaSO_4{\cdot}2\ H_2O$	5.7 ± 0.3	3.9 ± 0.3
Sphalerite	(Zn,Fe)S	0.3 ± 0.1	0.2 ± 0.1
Marcasite	FeS_2	4.9 ± 0.2	5.2 ± 0.3
Arsenopyrite	FeAsS	0.2 ± 0.1	0.0 ± 0.0
Jarosite	$KFe_3^{3+}(SO_4)_2(OH)_6$	7.0 ± 0.4	8.3 ± 0.4
K-Feldspar	$KAlSi_3O_8$	0.8 ± 0.5	0.4 ± 0.4

an S/L ratio of 1:2 in 10 L beakers until an electrical conductivity of < 2.3 mS/cm and a pH > 3.8 was reached. All the filtrates were combined, and Table 1 shows the experimental composition of the flotation residue waste washwater used in this investigation.

2.2. X-ray diffraction (XRD) characterization studies

The bulk mineralogy of the residue material before and after washing with water, as well as the precipitate after the membrane experiment, was determined via X-ray diffraction (XRD) analysis, as shown in Table 2. For sample preparation, the material was wet-milled in ethanol to a particle size of approximately 4 μm using a McCrone micronizing mill (Retsch, Haan, Germany) equipped with zirconium oxide grinding elements. Following drying, all the samples were deagglomerated and loaded into sample holders using the back-fill method to minimize preferred orientation effects. Diffractograms were collected using a PANalytical Empyrean diffractometer (Malvern Panalytical, Kassel, Germany) operating with a Co-target tube (35 kV, 35 mA) and a PIXcel 3D Medipix 1 × 1 area detector. An iron filter was placed on the primary beam side to suppress K-beta radiation. An automated divergence slit maintained a constant irradiated sample area of 12 × 15 mm^2. Data were acquired over a 2-theta range from 5° to 80° with a step size of 0.0131° 2-theta, resulting in a total measurement time of approximately 2.5 h per sample. Phase identification was performed using the ICDD PDF-4 + database, and phase quantification was carried out by Rietveld refinement using the Profex/BGMN (v. 4.1) software package.

2.3. Membrane filtration experiments

The nanofiltration membrane unit used in the entire study was a pilot-scale MAXIMEM high-pressure NF separation cross-flow membrane system from PS Prozesstechnik GmbH, Germany, equipped with a flat-sheet membrane module [39,40], as shown in Fig. 1. The nanofiltration cell setup consists of a feed tank (14 L capacity), pump, permeate and concentrate valves, feed and permeate flow meters, a flat sheet membrane module, and a temperature chiller with an online monitoring facility. Initially, the membranes were soaked with 1% Ultrasil solution for 30 min to remove any inorganic impurities if any; then the membranes were washed with Milli-Q water several times. Later, the membrane was inserted into the NF unit and conditioned with Milli-Q water for at least 1 h before the original experiment to keep the membranes hydrated and maintain them in the same conditions for all experiments. The nanofiltration studies were carried out using at least 2 L of real flotation residue waste washwater as a function of various parameters. After adding the feed, the pump was started, which pumped the feed into the module for the separation of multivalent metals and purified water. The permeate was collected separately in a different chamber, and the concentrate was recirculated to the feed tank again. The feed, permeate, and retentate samples were collected at regular intervals, and also before and after the experiment. The elemental concentration was determined using inductively coupled plasma-optical emission spectroscopy (ICP-OES) with a PlasmaQuant PQ9000 spectrometer (Analytik Jena, Jena, Germany) equipped with a cyclonic glass spray chamber and a seaspray TM glass nebulizer. All the NF studies were performed in duplicate, and results were within 5% of the standard deviation. The pH of the feed solutions was adjusted using dilute H_2SO_4 and water, and the corresponding metal concentrations were determined. For the complete decontamination and retentate concentration experiments, the permeate or retentate flotation residue waste washwater from the first cycles was collected and mixed together in the desired quantity, and the mixed water was used to test for complete removal or decontamination of permeate and preconcentration of metal in retentate in the second cycle, respectively. The organic and inorganic content of the permeate water after second cycle was analysed by using Sievers InnovOx TOC analyzer (Suez, Boulder, USA) equipped with autosampler unit. The % of metal rejection (% R) was determined by using Eq. 1, and the % of up concentration and permeate flux were calculated using Eqs. 2 and 3, respectively [40].

$$\%\ R = \frac{C_F - C_P}{C_F}.100\% \tag{1}$$

$$\%\quad Upconcentration = \frac{C_R - C_F}{C_F}.100\% \tag{2}$$

$$J = \frac{V}{t.S.\quad 3600} \tag{3}$$

where c_P and c_F are the concentration of metal of the permeate and feed solution (mg/L), V is the volume of permeate, S is the effective surface area of the membrane, and t is time interval for sampling since the starting of experiment, respectively.

3. Results and discussion

3.1. Washwater preparation and characterization of flotation residue

The wash procedure for the flotation residue as feed material was optimized concerning S/L ratio, number of contacts, and contact time, giving S/L 1:2, N = 5, and 30 min as feasible parameters for the removal of mobilized metal ions, reduction of conductivity, a practical filtration procedure, and minimization of water consumption. Fig. 2 depicts the determined concentrations of selected metal ions in separated filtrates after each contact with fresh water. The concentrations of Zn(II), Cu(II), and Al(III) after the 4th contact were 97–99% lower in comparison to the initial washing step, while removal of hazardous Cd(II) was 100% but secured by the execution of a fifth contact, which also gave an electrical conductivity < 2500 μS/cm (threshold value for drinking water in Germany [43]).

Interestingly, the concentration of Ca(II) was 464–590 mg/L in all filtrates, indicating the continuous dissolution of a mineral phase in the residue during the washing procedure. The comparison of the XRD

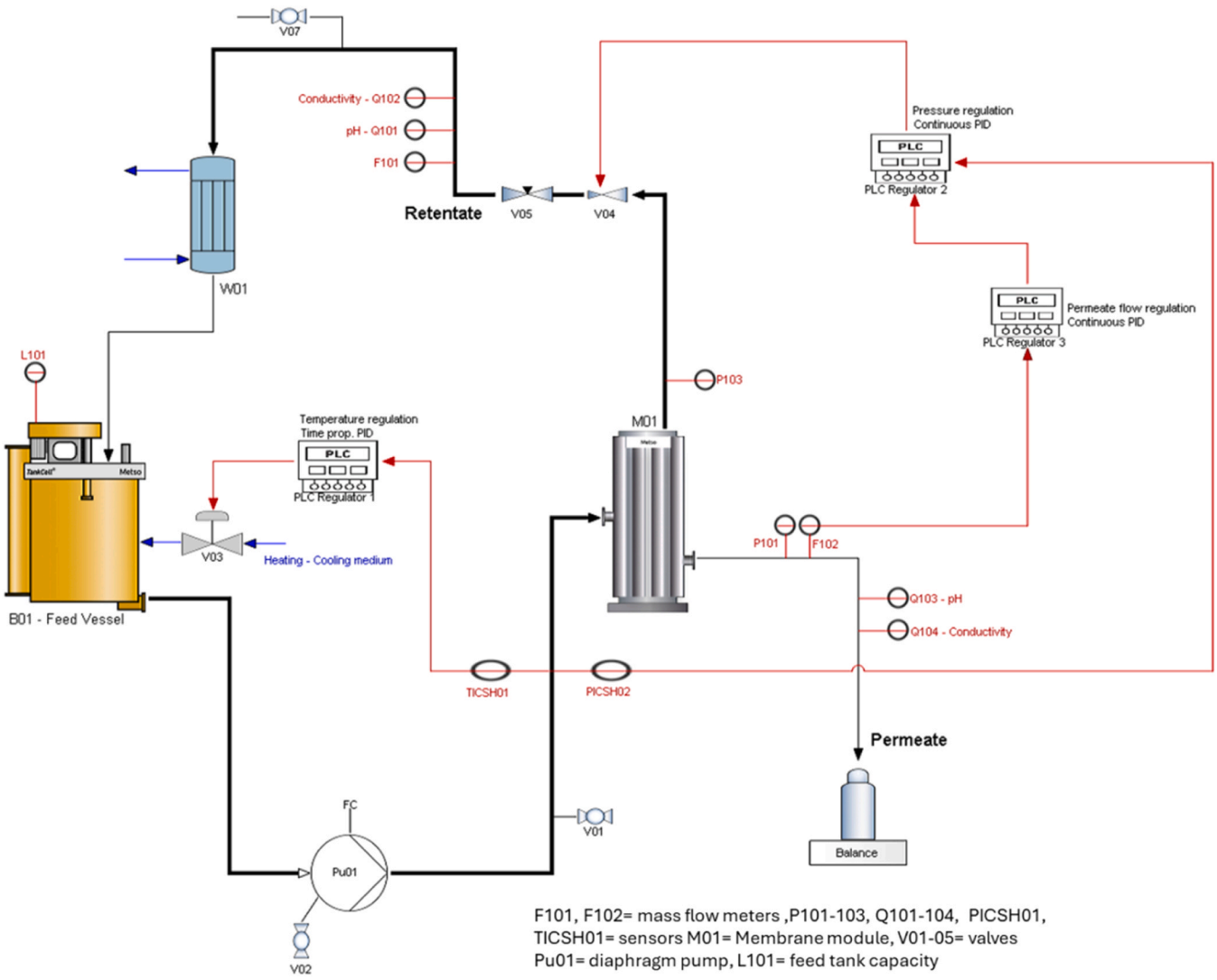


Fig. 1. Schematic pictoral representation of a pilot-scale high-pressure nanofiltration membrane (MAXIMEM) device [42].

phase composition before and after the procedure (Table 2 and S1) revealed that gypsum was partially dissolved, which is in agreement with its low but significant solubility in water.

3.2. Effect of pH on metal rejection

The pH of the feed solution is a very important parameter for the rejection of metal ions in NF because the membrane employed in the present study is an amphoteric polyamide membrane containing carboxylic acid as well as amines on the active layer of the membrane, and it has an isoelectric point around pH 3 [36]. This indicates that the membrane is more positively charged on the active layer below pH 3 and more negatively charged above pH 3 [40,44,45]. The studies on the effect of pH were evaluated under these conditions and are shown in Fig. 3. The metal rejection is found to be in the range of 85–98% for all di and multivalent metal ions, and similar rejection of multivalent metal ions (eg Zn(II) is 95–97% rejection) was observed across the entire pH range of the investigations. This confirms that the pH of the solution has minimal effect. However, above pH 2.5, the metal ions Fe(II, III) and other metals are precipitating or settling, and it was decided to continue the experiment at a lower pH for compatibility with the membrane process.

3.3. Effect of Pressure

The pressure on the membranes plays a crucial role in increasing the flux of the permeate and in the metal rejection ability. Fig. 4 shows the effect of pressure on the metal rejection in the DK membrane. The results show that the metal rejection was found in the range of 83–97% for different metals, and the performance was negligibly affected by pressure, but the permeate flux increased with pressure from 68 to 104 LMH. However, the longer exposure eventually caused membrane damage and scaling on the membrane (see Figure S1). The upconcentration ability increased with a change in pressure (Fig. 5), and it showed around a 2–3 times metal concentration increase for all the studied elements. This behaviour contributes to reinforcing the concentration polarization [44, 46].

3.4. Effect of temperature

Since feed temperature is an important parameter for the optimization of flux and membrane performance, the rejection of metal ions was explored as a function of various temperatures ranging from 20 to 50 ° C ,and the results are depicted in Fig. 6 and Table S4. The results from Fig. 6 revealed that the metal rejection, ranging from 83% to 98% for different metals, was not affected (e.g. Zn(II) rejection is 95–97%) by an increase in temperature [35–38]. However, it was noticed that the

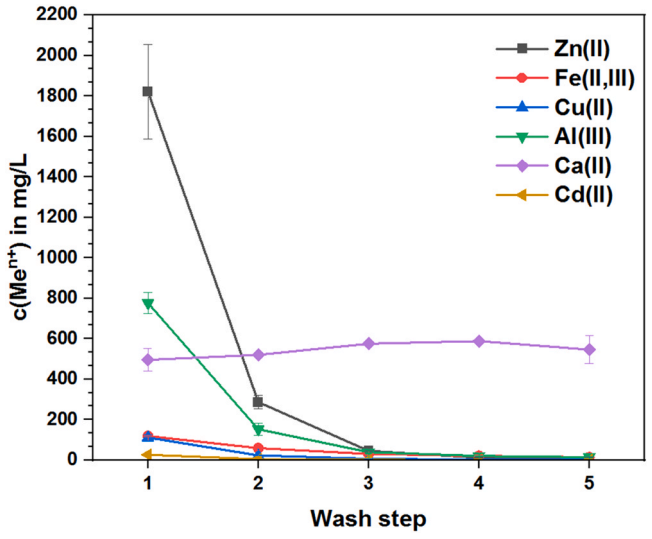


Fig. 2. Development of metal ion concentrations during washing. The concentrations of Pb(II) and In(III) were always < 0.5 mg/L; for As(III, V) = (2.69 ± 1.25) mg/L (N = 1), (1.11 ± 0.00) mg/L (N = 2) and < 1 mg/L (N = 3, 4, 5), respectively. Experimental conditions: S/L ratio of 1:2, T = 293 K, N_{total} = 5, and t = 30 min (per step).

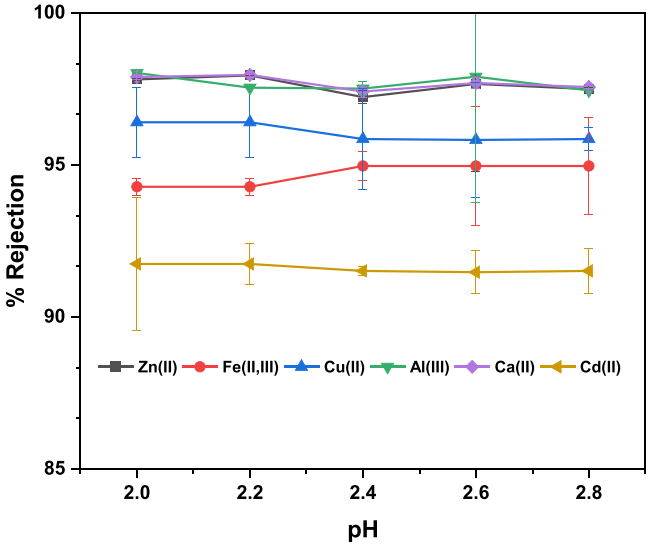


Fig. 3. The % metal rejection as a function of pH of the feed solution for the real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH = 2.0–2.8, T = 293 K, Q = 2 L/min, V_F = 2 L, DK membrane and t = 1 h.

permeate flux (82 LMH to 150 LMH) increased significantly due to the lowering of the viscosity of the solution, leading to ease of flow through the membranes [45–47]. In addition, the membranes got damaged upon increasing the temperature for the longer operation period, which is attributed to structural changes in the membrane.

3.5. Effect of flow rate and volume of the feed

The effect of the flow rate and volume of the feed solution on the metal rejection was studied and depicted in Fig. 7 and S2 to understand how the flow rate and volume of the feed would result in the metal rejection and flux. It is noticed that there was a negligible effect on the metal rejection of single metal ions, ranging from 85% to 98% for different metal ions, for both flow rate and volume of the feed. However, the permeate flux was moderately increased with flow rate to a

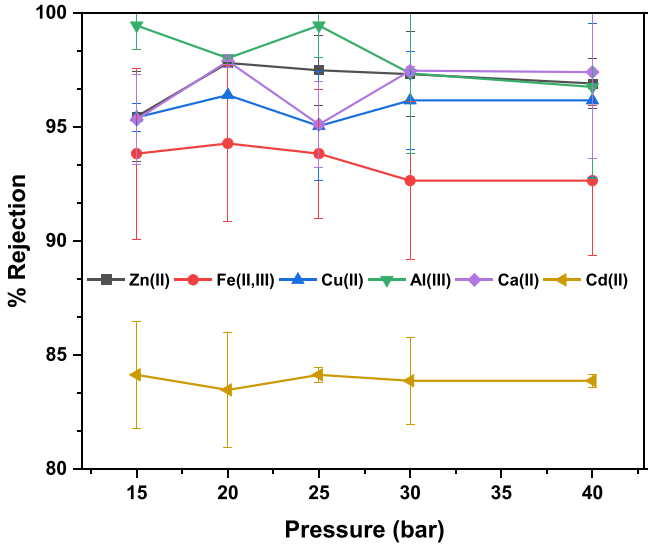


Fig. 4. The % metal rejection as a function of operating pressure for the real flotation residue waste washwater. Experimental conditions: p = 15–40 bar, pH2.0, T = 293 K, Q = 2 L/min, V_F = 2 L, DK membrane and t = 0.5–1 h.

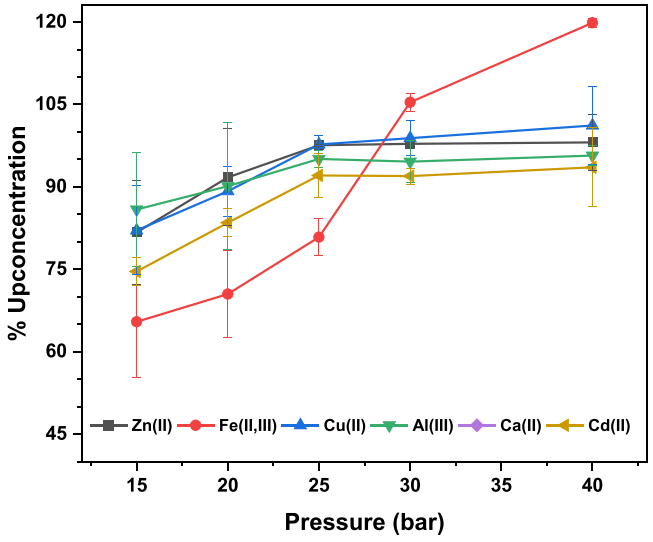


Fig. 5. The % upconcentration as a function of operating pressure for the real flotation residue waste washwater. Experimental conditions: p = 15–40 bar, pH = 2.0, T = 293 K, Q= 2 L/min, V_F = 2 L, DK membrane and t = 0.5–1 h.

reduction in concentration polarization. The flux was reduced with an increase in the volume due to an increase in the volume reduction ratio as well as concentration polarization [36,48,49]. Similarly, the flux with different volumes of the feed sample in Figure S3 shows that the flux decline is minimal and the membrane is quite stable, and it can be operated for a longer duration with greater stability in the flux.

3.6. Effect of different membrane types

Fig. 8 shows the metal rejection behavior with two different membranes. In this study, DL membranes were compared with DK membrane. DL is also a tight nanofiltration membrane with 96% $MgSO_4$ rejection and a higher flux rate. The results show that both membranes have similar ability for metal rejection (87–97% for different metals) for all the studied elements. However, it can be seen from Fig. 9 that DL has a higher flux ability compared to DK decreasing drastically with increasing time, indicating DL cannot sustain longer operation periods.

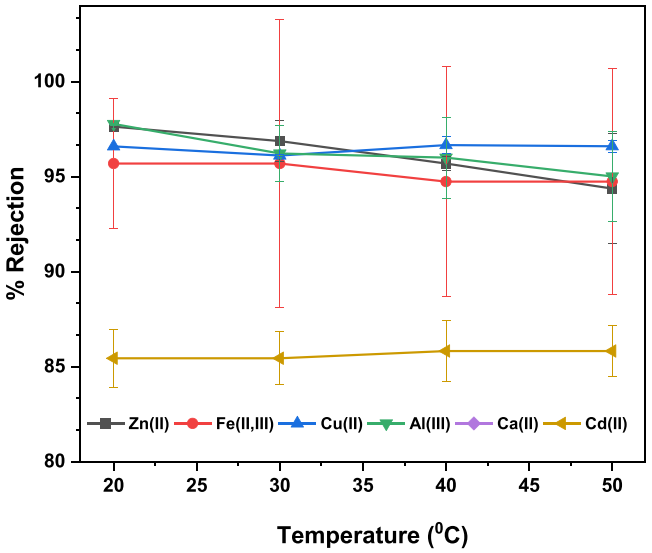


Fig. 6. The % metal rejection as a function of Temperature for the real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH 2.0, T = 293–323 K, Q = 2 L/min, V_F = 2 L, DK membrane and t = 0.5–1 h.

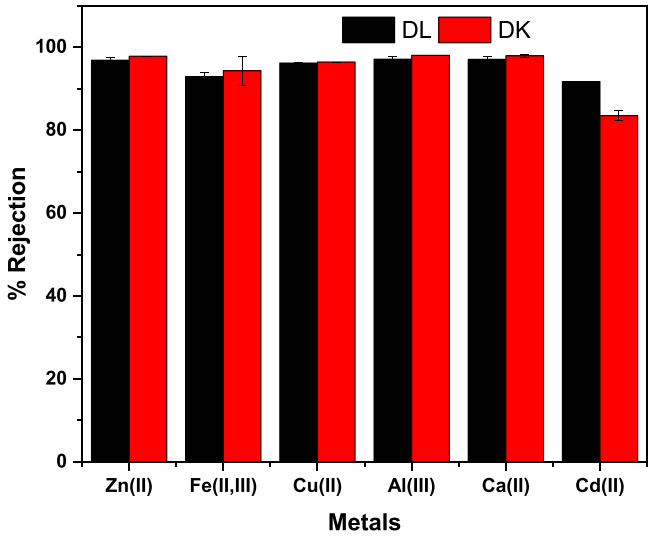


Fig. 8. The % metal rejection with different membranes for the real flotation residue waste washwater. Experimental conditions. Experimental conditions: p = 20 bar, pH = 2.0, T = 293 K, Q = 2 L/min, V_F = 2 L, DK or DL membrane, and t = 0.5–1 h.

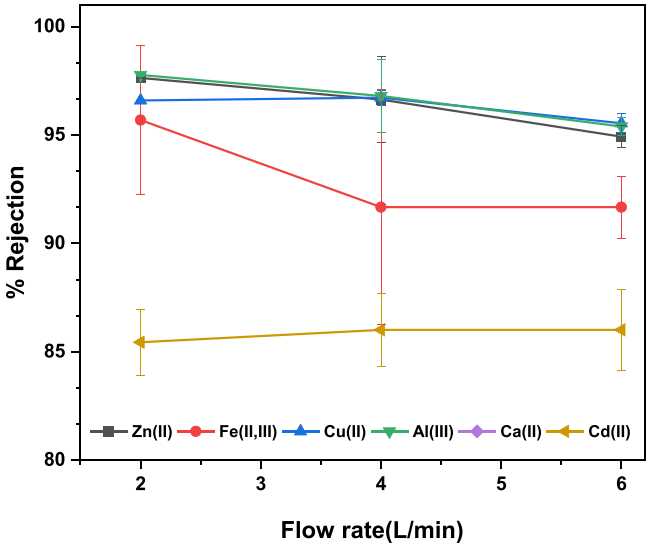


Fig. 7. The % metal rejection as a function of feed flow rate for the real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH = 2.0, T = 293 K, Q = 2–6 L/min, V_F = 2 L, DK membrane and t = 0.5–1 h.

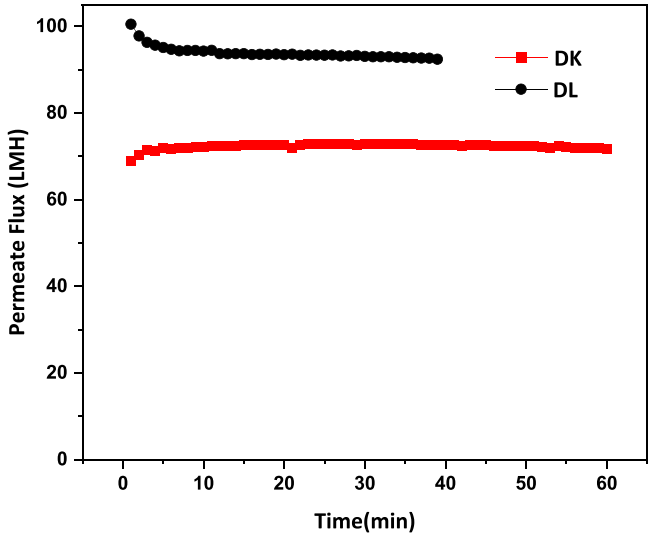


Fig. 9. The variation in flux of the permeate for the two different membranes using real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH = 2.0, T = 293 K, Q = 2 L/min, V_F = 2 L, DK or DL membrane, and t = 0.5–1 h.

By collecting all the parameters from above, the DK membrane was selected as the preferred material, and a pressure of 20 bar, a flow rate of 2 L/min, and a temperature of 293 K were identified as the best conditions for effective water purification as well as metal recovery, and their details have been presented in Table S2.

3.7. Membrane module stacking

The effect of employment of two membranes, i.e, two membrane modules, was investigated by connecting them in series for the same experimental conditions mentioned above (see Figure S4), and the results are shown in Fig. 10. The metal rejection for the single membrane set-up was comparable to the double membrane set-up. It was expected that the first membrane module would have removed most of the metal ions, and the solution would pass to the other membrane module to achieve complete decontamination upon usage of the membrane stacking approach. However, the solution initially passes through the first membrane. The concentrate or retentate from the first membrane is passed into a second membrane, which extracts more metal ions (see Figure S4). In practice, metal rejection in the second stage is often comparable to, or somewhat greater than, that of the first membrane. This happens when the second membrane gets a more concentrated feed, which might improve rejection owing to variables such as concentration polarization. Though rejection/membrane of the second membrane alone is the same, the overall metal removal across the two-stage system is greater, since more metal ions are gradually rejected by sequential treatment [49,50]. However, the flux of the permeate was increased as shown in Fig. 11. This is attributed to similar behaviour, resulting in the second module removing more concentrated solute, and the flux is slightly less than the combination of two membranes [49,50].

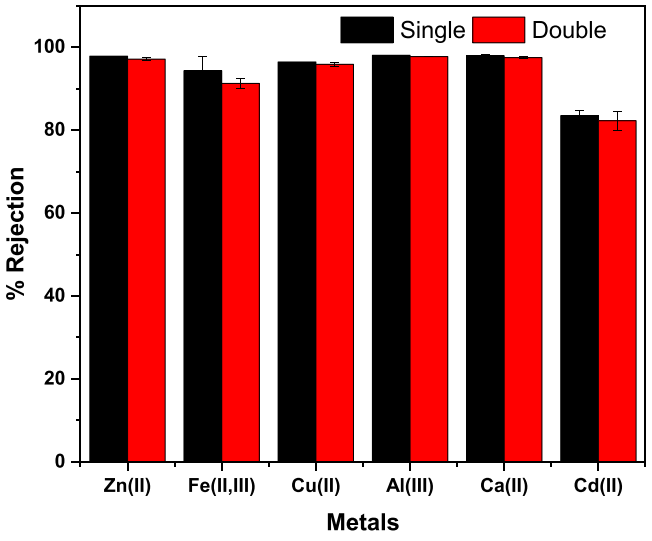


Fig. 10. The % metal rejection using two DK membranes modules connected in series for the real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH = 2.0 T = 293 K, Q = 2 L/min, V_F = 2 L, DK membrane, and t = 0.5–1 h.

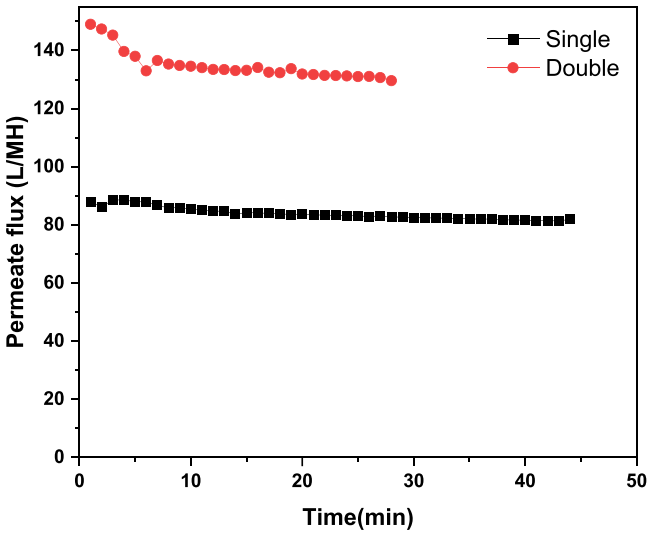


Fig. 11. The variation in the permeate flux with two DK membrane modules connected in series for the real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH = 2.0, T = 293 K, Q = 2 L/min, V_F = 2 L, DK membrane.

3.8. Retentate concentration

The nanofiltration membrane technique is not only useful for the purification of wastewater but also allows the increase of the metal ion concentration in the retentate as volume is reduced, and the metal ions can be again extracted or recovered using hydrometallurgical processes. To understand the behavior and how many cycles are required to attain the pre-concentration level, e.g above 2 g/L of metal ions, the studies have been performed in detail. As described in earlier sections, a single cycle treatment was not sufficient for getting a desired level of metal ions. Therefore, 5 L of retentate from the first cycle was taken, and NF membrane investigations were performed. The results are shown in Fig. 12. The metal rejection percentage ranges from 92% to 98%, and its rejection ability is almost the same in single metal ion (i.e for Zn (II), about 95–97%) in every litre collected, which confirms that the membrane easily sustains more recycles or iterations of the retentate for

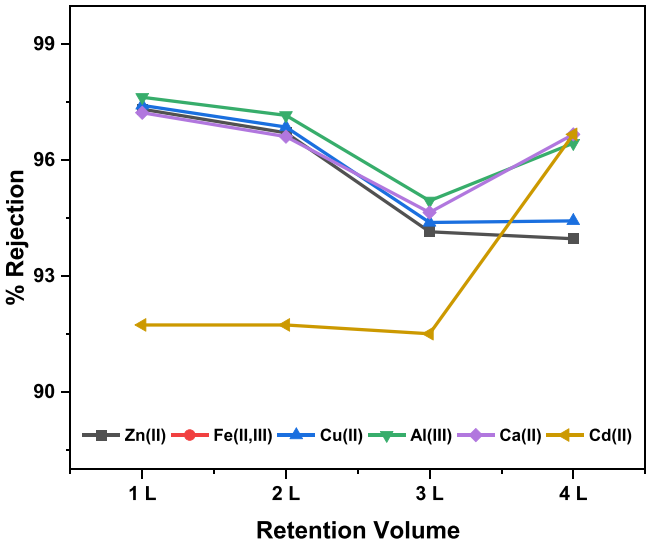


Fig. 12. The % metal rejection upon mixing retentate solutions from the first cycle of the real flotation residue waste washwater. Experimental conditions: p = 20 bar, T = 293 K, Q = 2 L/min, V_F = 5 L, DK membrane.

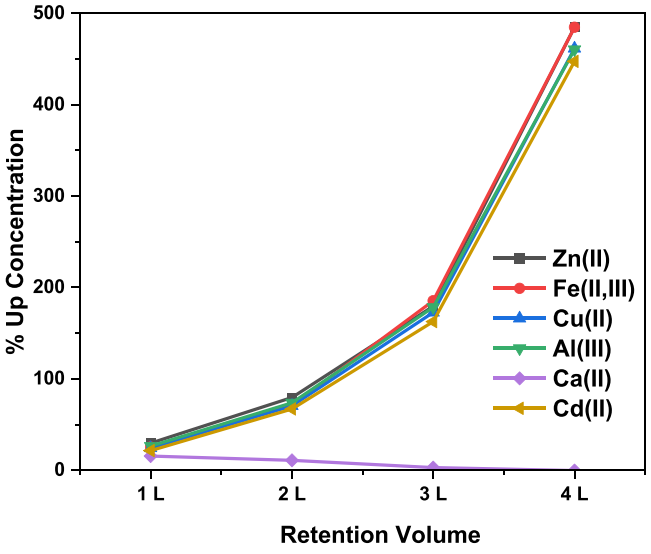


Fig. 13. The % upconcentration upon mixing retentate solutions from first cycle of the real flotation residue waste washwater. Experimental conditions: p = 20 bar, T = 293 K, Q = 2 L/min, V_F = 5 L, DK membrane.

further treatment or to increase the metal concentration. Fig. 13 and Table S3 show that the retention concentration ability was enhanced by 500% (concentration enrichment), and it reached the desired concentration of metal of about 4000 mg/L for Zn(II) in two cycles. The calcium content decreased, which is mainly due to the formation of supersaturated calcium sulfate in the retentate, which was further filtered and washed with Milli-Q water then dried at room temperature. The precipitate was analysed with XRD analysis and confirmed (Figure S5).

3.9. Number of cycles for decontamination

The metal rejection or removal from the permeate was found to be great, but there was not complete decontamination of the metals from the feed washwater solution. In order to investigate how many cycles are necessary for complete decontamination of metal ions and align the criteria of water that can be used for industrial usage or domestic purposes, the studies have been performed by combining permeate from the

Table 3
The % metal rejection upon mixing permeate solutions from the first cycle of the real flotation residue waste washwater. Experimental conditions: p = 20 bar, pH = 2, T = 293 K, Q = 2 L/min, and V_F = 2 L.

Metal ion	Zn(II)	Fe(II,III)	Cu(II)	Al(III)	Ca(II)	Cd(II)
First cycle						
Feed	**411**	**9.36**	**25.4**	**186**	**540**	**6.02**
Permeate	**36.1**	**2.3**	**2.25**	**16.4**	**27.0**	**BDL**
Retentate	**1970**	**50.8**	**129**	**885**	**497**	**28.4**
% Rejection	**96.25%**	**89.32%**	**96.06%**	**96.56%**	**96.26%**	**91.69%**
Second cycle						
Feed	**36.1**	**2.3**	**2.25**	**16.4**	**27.0**	**BDL**
Permeate	**< 1**	**< 1**	**< 1**	**< 1**	**< 1**	**< 1**
Retentate	**430**	**12.6**	**26.3**	**180**	**349**	**BDL**
% Rejection	**99.74%**	**89.32%**	**96.06%**	**99.46%**	**99.81%**	**-**

BDL=below detection limit

first cycles and contacting it with the NF membrane system. The details are shown in Table 3. It can be seen from the table that the complete decontamination of all metal ions has been achieved within two cycles (Total organic and inorganic content of the permeate water is about 10–20 mg/L, pH=5.8–6.8, conductivity=0.50–1 μS/cm are our observed value after the decontamination which are below the regulatory limits), and the values agree with the specifications given by the water regulatory authorities [43]. In addition to this, the flux in the second cycle increased due to a lower concentration of metals and fewer suspended particles in the second-cycle feed solution. Therefore, it would be one of the best ways to use nanofiltration membranes to purify/decontaminate the metal-containing water to eliminate not only metal ions, but it also has higher efficiency with less energy and /or chemical consumption compared to other approaches.

4. Conclusion

In this study, the treatment of waste washwater obtained from mine tailings flotation residues containing various metals such as Zn, Fe, Cu, Al, Ca, and Cd with concentrations ranging from 5 to 400 mg/L, using a nanofiltration membrane approach was investigated with different polyamide flat sheet thin-film composite membranes such as DK and DL membranes. The studies were conducted on various parameters such as the effect of pH, pressure, temperature, flow rate, volume, membrane stacking, and different membranes. The results revealed that metal rejection was found for multivalent metals in the range of 85–98%, and the rejection ability was significantly similar with changes in pH, pressure, temperature, volume, flow rate, or different kinds of membranes. Whereas the flux of the permeate varied significantly with pressure, different membranes, flow rate, and temperature due to concentration polarization, steepness, and a decrease in viscosity, respectively. The pre-concentration of Zn(II), i.e about 4 g/L, with 500% concentration enrichment was achieved with two cycles of retentate concentration studies. This pre-concentration allows the effective recovery of zinc via hydrometallurgical technologies such as precipitation, solvent extraction and electrowinning. The complete decontamination, i.e below 1 mg/L, of the investigated metal ions from flotation residue waste washwater was successfully attained within two cycles of studies. The permeate water after two cycles was tested for pH, conductivity, and TOC analysis, and the values were found to match the standard regulations provided by WHO and German authorities. Therefore, the results have been evaluated and discussed from the perspective of nanofiltration technology as a suitable option for the treatment of flotation residue wash water for safe discharge into the environment within two cycles and as a means of pre-concentrating valuable metal ions for further recovery operations.

CRediT authorship contribution statement

K. Rama Swami: Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Norman Kelly:** Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Data curation, Conceptualization. **Chaouki Boucena:** Investigation, Formal analysis. **Doreen Ebert:** Investigation, Formal analysis, Data curation. **Ulrike Fischer:** Investigation, Formal analysis. **Frank Haubrich:** Writing – review & editing, Formal analysis, Data curation. **Sebastian Braune:** Formal analysis, Data curation. **Roland Haseneder:** Writing – review & editing, Formal analysis. **Ajay B. Patil:** Writing – review & editing, Validation, Supervision, Project administration, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.dwt.2026.101980.

Data availability

Data will be made available on request.

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