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A Hybrid Process for Recovering Copper from Dilute Solutions

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Abstract: The present paper describes a hybrid process comprising of reverse osmosis (RO) and solvent extraction (SX) techniques for recovering copper from dilute solutions. The solution containing copper is processed through RO to obtain two separate streams,

1. a concentrate stream containing higher concentration of copper and
2. the permeate stream [treated water] suitable for safe discharge.

The concentrated solution from RO is further concentrated through solvent extraction/stripping to recover copper. Using the RO technique the copper ion concentration was enriched up to 10.16 kg/m^3 from an initial copper concentration of 100 g/m^3. The metal ion concentration in permeate stream increased from 0.76 g/m^3 to 362 g/m^3 with increase of feed concentration from 100 g/m^3 to 6.0 kg/m^3. The permeate stream was recycled through RO to obtain a treated water containing metal ion within tolerance limit for re-use. The concentrated solution containing copper was further processed employing solvent extraction technique using LIX 84I(as the extractant). The extraction efficiency ranged between 84.27 and 92.08% at the A:O ratio of 1:1, 1:1.5 and 1:2. The percentage extraction (in a single stage) increased from 74.26 to 84.27% with increase of LIX 84I concentration from 30 to 50 vol.%. The loaded organic generated under the above optimum conditions was used in stripping studies. The stripping efficiency (in a single stage) increased from 83.30 to 99.50% with increase of H_2SO_4 concentration from 100 to 150 kg/m^3.

Keywords: Reverse osmosis, solvent extraction, LIX84I, extraction, stripping

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INTRODUCTION

Today huge quantities of ores are mined, processed, and disposed into the environment, which lead to many adverse effects on human beings. Numerous are the cases of confirmed metal poisoning due to the release of waste water containing toxic metal ions. Mining and metallurgical, plating, surface finishing, and chemical industries generate a significant amount of discharge water containing heavy metal ions. The removal of metal ions from industrial effluents containing low metal ion concentration has proved difficult and expensive. Selective recovery of metal ions from the effluent in a cost effective manner is challenging, but success in this endeavor would produce a very appealing process with three important benefits:

1. legislative compliance,
2. water recycling and
3. metal recovery.

Increasing demand for water coupled with the significant environment regulations has made it imperative for industries to look for appropriate methodology for treatment of their effluents (1). There are many processes like precipitation, co-precipitation, adsorption, cementation, and electrowinning(EW) etc. available for treating and reducing the metal ion concentration in the effluents (2). Membrane separation processes like reverse osmosis (RO) and electro dialysis(ED) have been studied for treating dilute solutions (3, 4). The metallurgical industries have used precipitation, ion- exchange (IX), solvent extraction (SX), and electro winning frequently for metal recovery (5–9).

In the last few years, the treatment of industrial effluents by membrane processes has gained more and more importance because of the need to conserve water and also due to the increasing cost of chemicals and energy. Hanra et al.(10) studied the trace level separation of zinc sulphate and lead nitrate from toxic effluent stream by reverse osmosis modular system. Electroplating wastewater containing copper was treated by means of reverse osmosis membrane on a pilot scale and the treated water was safely discharged (11). Freger et al. (12) studied the separation of concentrated organic/inorganic salt mixtures by nanofiltration. Ruey-Shin Juang et al. (13) studied the removal of single metal ions including CS^+, Sr^{2+}, Mn^{2+}, Co^{2+}, Cu^{2+}, Zn^{2+}, and Cr^{3+} from an aqueous solution (upto 200 mg/L) by ultrafiltration. Membranes are also used for decolorization of cane sugar solution (14), isolation of antibiotics from industrial fermentation broths (15), color removal, and reduction in COD of textile plant effluent (16).

Solvent extraction is a unit operation that is being commonly used to separate/recover metal ions from solutions. The extractant, LIX 84I is being selectively used for copper extraction. Using 7.5% LIX 84 in kerosene, Cu was extracted to the extent of >95% in 2-stages from a feed solution having (pH 1.8) 0.54 kg/m^3 Cu and 4.0 kg/m^3 Fe (17). Ali et al. (18) described copper extraction using LIX 84. Yun et al. (19) studied the extraction of copper through hollow fibers using LIX 84 in heptane. Parija et al. (20) studied the separation of nickel and copper from ammoniacal solutions through co-extraction and selective stripping using LIX 84 as the extractant and recovered nickel from solutions containing ammonium sulphate (21).

REMCO Engineering (22) used a hybrid process combining ion-exchange and RO to recycle/reclaim water and to recover metal. This hybrid process has an advantage over the other single unit operation since it uses benefits of IX for recovering metal efficiently and of RO for low cost and efficient removal of TDS. Wong et al. (23) developed a process combining microfiltration, U. V. irradiation, carbon adsorption, nanofiltration, and ion exchange for treating and recycling rinse water from electroless plating operation. Traditional technologies are under pressure with already stringent legislation. Redesigning of existing systems to meet expected tightening of the legislation would likely entail excessive cost.

The objective of the present study is to treat the dilute solution containing copper for safe disposal with simultaneous recovery of copper by adopting a hybrid process consisting of reverse osmosis and solvent extraction technique which are frequently used in hydrometallurgical process.

EXPERIMENTAL

Reagents

The commercial extractant LIX84I was supplied by Cognis Corporation, Arizona, USA and was used as received. Distilled kerosene (bp 190°–210°C) was used as the diluent. All other chemicals used such as $CuSO_4 \cdot 5H_2O$, H_2SO_4, NaOH, etc., were of analytical reagent grade.

Membrane

Spirally wound thin film composite reverse osmosis membrane supplied by M/s Permionics, Baroda, India was used for reverse osmosis experiment. This thin film composite membrane module was commercially available and was used without further modification. The membrane area used for the experiment was 0.72 m^2. The pore size of the membrane was 10A° units. The size of the module used was $2.5' \times 20'$ long.

Methods

The copper sulphate solution of desired concentration was prepared and 20 L of the solution was taken for each experiment. This solution was pumped from the container by the feed pump and was passed through the system by the high pressure RO pump. The pressure-regulating valve connected to the system was suitably adjusted to set the upstream pressure at the desired value. The reject stream was taken back to the feed.

Samples were withdrawn from the permeate and concentrate stream at desired time interval. The samples were diluted with 1.0M HCl (to the required concentration) and were analysed for copper using Atomic Absorption Spectrophotometry (AAS, Perkin Elmer, model 372).

In the solvent extraction studies, 10 cm^3 of the aqueous solution containing 10.16 kg/m^3 of copper was equilibrated with an equal volume of LIX84 I in a separating funnel for 5 mins. After complete phase disengagement, the aqueous phase was separated and the equilibrium pH of the raffinate was measured with a pH meter (Systronics, model 324). The raffinate was analysed for copper concentration. The copper concentration in the organic phase was calculated from the difference between the metal ion concentration in the aqueous phase before and after extraction.

RESULTS AND DISCUSSIONS

The solution containing copper (100 g/m^3) was processed through RO to obtain two separate streams,

1. a concentrate stream containing higher concentration of copper and
2. the permeate (treated water) stream suitable for safe discharge.

The concentrated solution obtained was further processed by solvent extraction technique to finally produce a more concentrated copper solution, from which copper can be recovered by electro-winning.

Enrichment of Copper Ion using RO

By treating dilute solutions containing about 100 g/m^3 of copper through solvent extraction, volume handling as well as the solvent loss will be high. On the other hand, by enriching the copper values in the solution to a certain extent by RO, the solvent loss is reduced and the throughput increases in the subsequent solvent extraction step. Hence, experiments were carried out to enrich copper values in the solution up to ~10 kg/m^3 (100 times) from a dilute solution (100 g/m^3), which can be further concentrated using solvent extraction/stripping.

Effect of Pressure on PWP

Since the hydrostatic pressure difference is the driving force of the reverse osmosis technique, the effect of pressure on membrane performance was investigated in detail. At the beginning of the experiment, distilled water was circulated and the pure water permeability (PWP) was measured using Equation (1) and the results obtained were shown in Fig. 1.

$$\mathrm{PWP} = \mathrm{W}/\mathrm{t}\ \mathrm{A} \tag{1}$$

Where,

$\mathrm{W} = \text{Weight of permeate, g}$
$\mathrm{T} = \text{Time, h}$
$\mathrm{A} = \text{Area of the membrane, m}^2$

It was observed that the PWP increased from 4.0 to 11.0 $\mathrm{g/h \cdot cm^2}$ with increase of pressure from 80 to 200 psi. Also the PWP for the membrane was checked after each 4/5 experiments and it was observed that PWP remained constant each time. This concluded that the membrane characteristics remained constant with the experimental system and the operating pressure.

Effect of Pressure on Product Rate (PR) and Percentage Rejection of Copper

Using the above equation (1), the product rate (PR) of copper solution was also measured at different pressures and at different feed solution concentrations (0.5, 1.0, 2.0, 5.0, 6.0 $\mathrm{kg/m^3}$). It was observed that for a particular concentration, PR increased linearly with increasing pressure from 80 to 200 psi

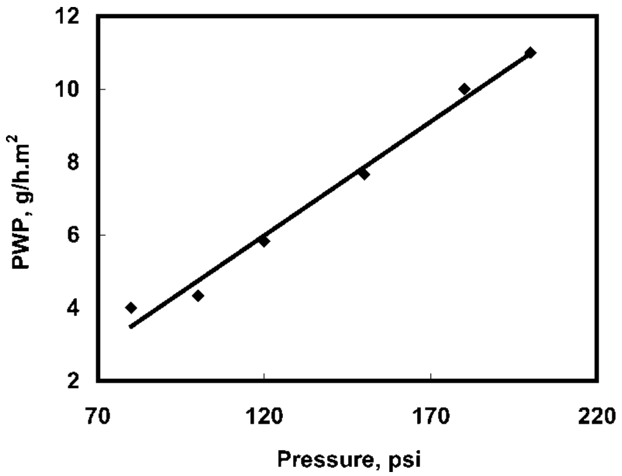

Figure 1. Effect of pressure on PWP.

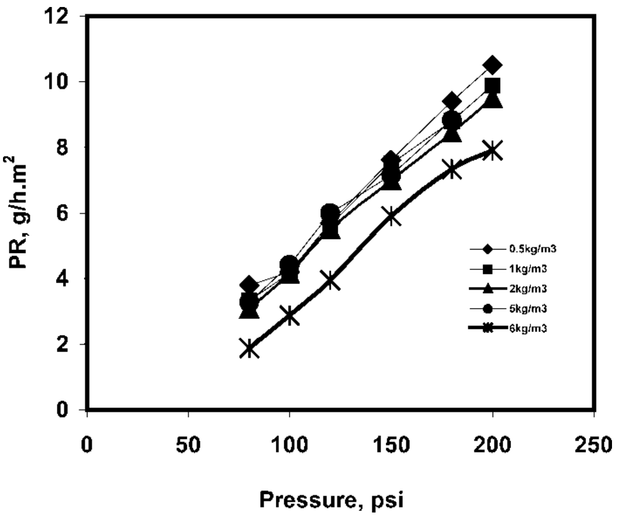

Figure 2. Effect of pressure on PR.

(Fig. 2). But with increasing initial concentration of copper in feed solution from 0.5 to 6.0 kg/m^3, the product rate decreased, because the higher the concentration of solution, the higher was the osmotic pressure and consequently, the product rate was decreased.

To study the effect of pressure on percentage rejection of copper, the applied pressure was varied in the range of 80–180 psi. The initial concentration of feed solution was kept constant at 6 kg/m^3. The solute rejections were calculated at different pressure using Equation (2) and the results obtained are shown in Fig. 3.

$$S.R = \left[1 - \frac{C_1}{C_2}\right] \times 100 \qquad (2)$$

Where, S.R. = Solute rejection, %

C_1 = Concentration of $CuSO_4$ in permeate
C_2 = Concentration of $CuSO_4$ in feed

It was observed that the percentage rejection for copper increased from 92.59 to 95.3 with increase of pressure from 80 to 180 psi. The product rate increased with increasing pressure (Fig. 3) and therefore the percentage rejection increased. The increase of rejection with the water flux is a general phenomenon because an increase in trans membrane pressure causes a higher water flux, whereas the salt flux is electrically (Donnan exclusion and electromigration effects) and sterically hindered and,

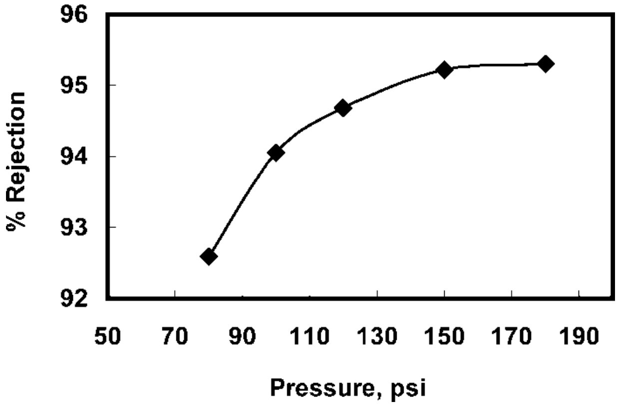

Figure 3. Effect of pressure on percentage rejection of metal ion.

therefore does not change that much (24). Further experiments were carried out at 180 psi.

Effect of Feed Solution Concentration on Percentage Rejection of Copper

To study the effect of feed solution concentration on percentage rejection of copper, the feed solution concentration was varied in the range of 0.1–6.0 kg/m^3. The pressure was kept constant at 180 psi. The permeate stream of each experiment was collected separately and was analysed for copper. The percentage rejections at different copper concentrations of feed solution are shown in Fig. 4. It was observed from Figure 4 that the percentage rejection of copper decreases from 99.24 to 93.95 with increase of copper concentration in feed solution from 0.1 to 6.0 kg/m^3. This may be due to the concentration polarization at the interface of reject stream and the membrane (25). As the water permeates through the membrane, the concentration of $CuSO_4$ increased near the ingoing interface between the membrane and the reject stream. The concentrated $CuSO_4$ depleted then by diffusion to the bulk phase of the reject stream. Since the accumulation rate is greater than the depletion rate, there is a net concentration build up at the interface. As the concentration is thus polarized, the solute flux through the membrane was enhanced and thus the solute rejection was reduced. The concentration of copper in the permeate stream increased from 0.76 to 362.5 g/m^3 with increase of feed concentration from 0.1 to 6.0 kg/m^3. With 5 kg/m^3 feed solution, the concentration of Cu in the permeate stream was 228.25 g/m^3, which was feed back to RO to obtain treated water containing metal ion within tolerance limit (<3 g/m^3). So this was taken as the cut off point for RO and the concentrate (10.16 kg/m^3) obtained at this stage was further concentrated by solvent extraction.

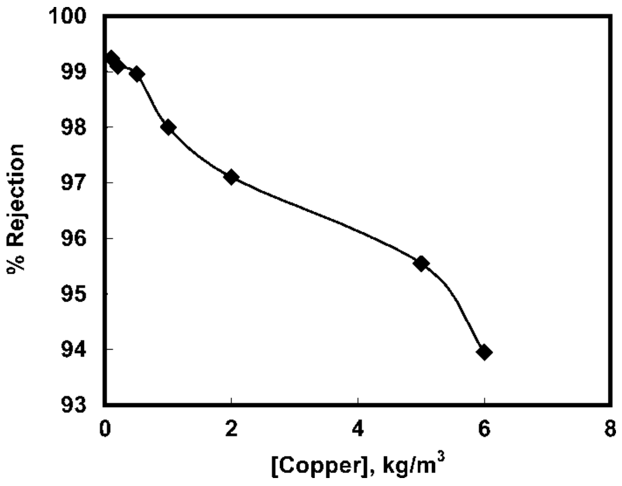

Figure 4. Effect of metal ion concentration on % rejection.

Solvent Extraction Studies of Copper with LIX84 I

The copper solution obtained through reverse osmosis treatment was used for further enrichment by solvent extraction technique in order to produce a feed for electrowinning of copper and crystallization of $CuSO_4$. LIX 84I (diluted with kerosene) was used to extract copper.

Extraction of Copper

Phase Variation Studies

Single-stage extraction tests were carried out using the RO enriched solution as the aqueous phase and 50 vol.% LIX 84 I as the organic phase at the A:O phase ratios of 1:1, 1:1.5 and 1:2. The raffinates were analysed and the extraction efficiencies obtained were 84.27, 89.60 and 92.08% respectively at the above phase ratios. The equilibrium pH values were ~0.9 at all the three phase ratios and may be the reason for a very small increase in extraction efficiency, when the ratio was increased by 1.5 and 2 times.

Variation of LIX 84 I Concentration

The extraction efficiency obtained at 1:1 phase ratio with 50 vol.% LIX 84 I was 84.27%. To find out the possibility of reducing the solvent concentration, further tests were carried out at 1:1 phase ratio using 30 and 40 vol% LIX 84 I. These studies indicated that extraction efficiencies were 74.26 and 80.45%, respectively at the LIX 84I concentration of 30 and 40 vol.%. From the extraction efficiencies obtained with 30 and 40 vol.% LIX84I it can be found that, the number of counter-current stages required for quantitative extraction of Cu would be more than 4. It was therefore decided to use 50 vol.% LIX84I for further studies.

Extraction Isotherm

Having decided to use 50 vol% LIX 84I for copper extraction, the extraction isotherm was obtained to find out the number of stages required at a chosen A:O ratio. The feed solution and 50 vol.% LIX84I were contacted at different phase ratios (A:O = 1:7 to 7:1) and after phase separation, the two phases were analysed. The extraction isotherm (plot of Cu concentration in the aqueous phase against the concentration of Cu in the organic phase, Fig. 5) and the McCabe-Thiele construction (Fig. 5) indicated the possibility of >99.5% copper extraction in three counter-current stages at 1:1 phase ratio. This was confirmed by carrying out a three-stage counter-current batch simulation study. Analysis of the raffinate at equilibrium showed the presence of 70 g/m^3 of Cu indicating 99.4% extraction efficiency.

A sufficient quantity of representative loaded organic was generated under the above-optimized conditions for carrying out stripping studies.

Stripping of Copper

The loaded organic generated as above contain 10.16 kg/m^3 of Cu. The stripping studies were carried out with sulphuric acid and the details are given below.

Variation of Acid Concentration in the Strip Solution

Strip solutions were prepared having sulphuric acid of varying concentration and single-stage stripping tests were conducted at 1:1 phase

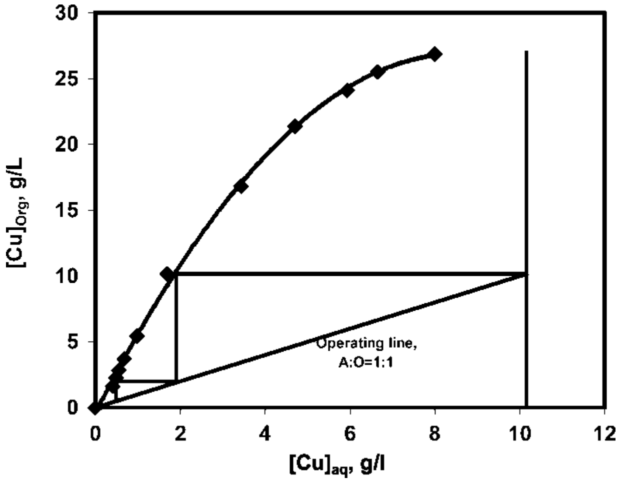

Figure 5. Extraction isotherm for copper.

ratio. The stripping efficiencies obtained at acid concentration with 100 and 150 kg/m^3 were 83.30 and 89.60% from which it can be concluded that stripping efficiencies could be ~99.53 and ~99.89% in two counter-current stages with strip solutions containing 100 and 150 kg/m^3 of H_2SO_4 respectively. Further stripping studies were carried out at 110, 120, 130, and 140 kg/m^3 H_2SO_4 concentration and the stripping efficiencies obtained at different acid concentrations were shown Fig. 6. The increase in efficiency was marginal and therefore, a strip solution with 100 kg/m^{3}of H_2SO_4 concentration was chosen.

Stripping Isotherm and Batch Simulation

To find out the number of stages required for stripping Cu from the loaded organic, the stripping isotherm and McCabe-Thiele construction were obtained using the loaded organic containing 10.16 kg/m^3 of copper and the strip solution containing 100 kg/m^3 of H_2SO_4 at different phase ratios. The McCabe-Thiele construction (Fig. 7) indicated that quantitative stripping of copper is possible in two counter-current stages at 1:1 phase ratio.

A two-stage counter-current batch simulation study carried out at 1:1 phase ratio which resulted in a spent organic having 48 g/m^3 of copper (99.5% stripping efficiency). When the same study was repeated at the A:O phase ratio of 1:1.5, copper in the spent organic increased to 170 g/m^3 (98.3% stripping) resulting in a strip solution containing ~15 kg/m^3 Cu. These studies indicate that the copper concentration in the strip solution can be increased to 30–40 kg/m^3 for the concentration, suitable for crystallisation when strip solution containing >100 kg/m^3 H_2SO_4 is used.

To achieve higher concentration of Cu in the strip solution (suitable for crystallisation), single-stage stripping tests were carried out at different phase ratios (1:3 to 1:5) using strip solutions having 178 kg/m^3 of H_2SO_4. The stripping efficiencies were 81.5, 72.5 and 65.0% respectively at the A:O phase ratios of 1:3, 1:4 and 1:5. Based on these results, 3-stage batch

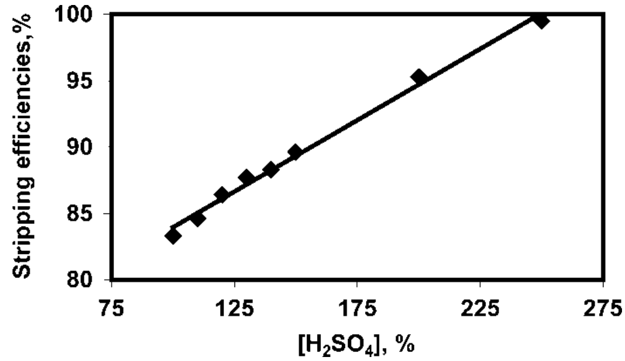

Figure 6. Stripping efficiencies at different acid concentration.

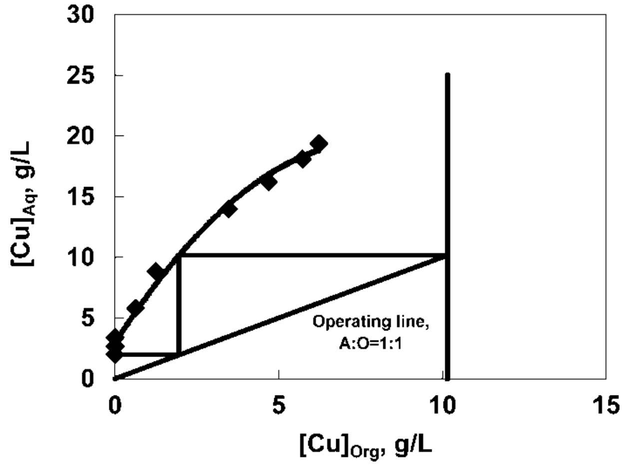

Figure 7. Stripping isotherm for copper loaded LIX84 I.

simulation studies were carried out at the A:O phase ratios of 1:3 and 1:4. The number of counter-current stages required for copper stripping would be more than five at O:A phase ratio of 1:5. The stripping efficiencies were 99.43 and 99.24%, respectively and the spent organics retained 58 and 77 g/m^3 of copper. The strip solution obtained from the batch simulation studies contained 30–40 kg/m^3 of Cu suitable for crystallization. Based on the above studies a conceptual flow sheet was made and shown in Fig. 8.

Thus by combining the reverse osmosis technique with solvent extraction for recovering copper from dilute solutions, volume handling as well as the solvent loss will be minimised The enriched copper can be recovered by electriwinning/crystallization and the treated water can be reused/disposed safely.

CONCLUSIONS

Using the RO technique copper enrichment was carried out from a feed solution containing 100 g/m^3. The cut off point of RO was taken at feed solution concentration of 5 kg/m^3, where the permeate stream contains 228.25 g/m^3 of Cu. This permeate stream can be sent back to RO to obtain treated water having Cu concentration within tolerance limit. Copper could be extracted from RO enriched feed using 50 vol.% LIX 84I in 3 counter current stages (at equal phase ratio) and the extraction efficiency obtained was 99.4%. The copper could be stripped from the loaded organic phase in 3 counter–current stages using H_2SO_4 of 178 kg/m^3 to the extent of 99.43 and 99.24% at the A:O phase ratios of 1:3 and 1:4 respectively. The spent organic retained 67 ± 10 g/m^3 of copper and the strip solution contained 30–40 kg/m^3 of Cu suitable for crystallization. Thus, it is possible to

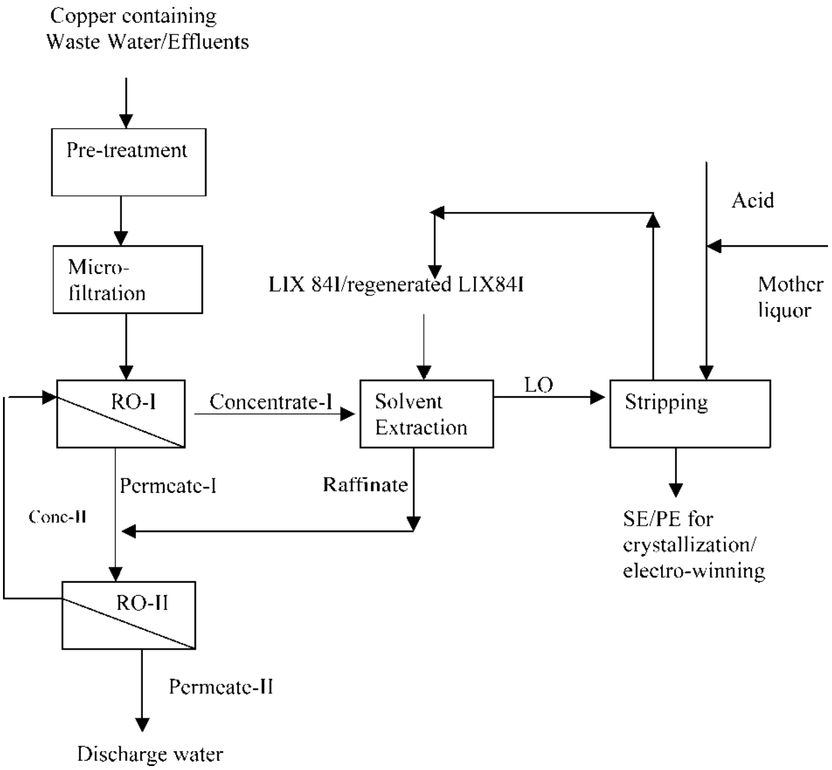

Figure 8. Conceptual flow sheet for the recovery of copper from the waste water/effluent using RO-SX technique.

recover copper from a dilute solution containing 100 g/m^3 of Cu through a hybrid process containing a combination of RO-SX techniques and to regenerate treated water for re-use/safe disposal.

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