

ICEWEST-2015 [05th - 06th Feb 2015]
International Conference on Energy, Water and
Environmental Science & Technology

PG and Research Department of Chemistry, Presidency College (Autonomous),
Chennai-600 005, India

Recovery of nickel from aqueous solution using bulk liquid membrane

K. Mubeena^{1*}, G. Muthuraman¹

Presidency College, Department of Chemistry, Chennai-5, India

Abstract: This paper deals with the facilitated through a bulk liquid membrane (BLM) system of aqueous phase Nickel (II) using trimethyl amine dissolved in an appropriate organic solvent. The effect of TMA concentration dissolved in kerosene on the extraction efficiency reveals that TMA combined with Ni (II) in the ratio of 1:1. Using a BLM system, more than % of the initial Nickel (II) content in the feed phase was extracted and stripped in aHCl aqueous receiving phase. The study has highlighted the importance and influence of membrane composition for maximizing the extraction of Nickel (II). For this reason, effect of various parameters such as effect of extractant concentration, effect of pH, effect of stripping solution type, effect of mixing speed, effect of stripping solution concentration were studied and optimum conditions were estimated.

Key words : BLM, Kerosene, Recovery of nickel(II), TMA.

Introduction:

Nowadays, industrial and municipal wastewater frequently was contaminated with metal ions. metals are among the most important pollutants in source and treated water. Nickel (II) is a highly toxic metal ion that is readily encountered in the environment, which it enters primarily from land based resources such as oxidic and sulfide ores. Nickel is the most widely used metals in various industries due to the superior properties they possess. Nickel was long thought to be essential to plants and some domestic animals, but not considered to be a metal of biological importance until 1975, when Zerner discovered that urease was a nickel enzyme¹. It is known that inhalation of nickel and its compounds can lead to serious problems, including nasopharynx, lung and dermatological diseases and malignant tumors^{2,3}. So, separation of toxic Ni(II) is of intense current interest in research and environmental cleanup.

The separation of nickel in aqueous has been a problem in hydrometallurgy⁴. Hydrometallurgy processes are among the most applied methods for the recovery of heavy metal ions from various sources⁵. Commercial extractants cyanex 272⁶ and cyanex 923⁷ have been examined for the extraction of nickel and cadmium ion from synthetic mixtures. A hydrometallurgical method was developed for recovery of nickel and cadmium from used rechargeable batteries⁸.

The liquid membrane (LM) has been studied under different geometrical configurations Such as bulk liquid membrane (BLM), supported liquid membrane (SLM), emulsion liquid (ELM) membranes⁹. In some cases the method of bulk volume membrane extraction can be used for the separation of various metal ions¹⁰. Low

capital costs, space requirements and energy consumptions are certain advantages offering by this class of separation techniques¹¹. The transport of other metal ions such as zinc, mercury, silver, palladium and gold in the anionic form of the complexes has also been reported^{12,13}. In the present study, we selected the nickel (II) for solvent liquid membrane study. Nickel (II) is the cationic metal. So it requires an anionic carrier for the extraction purpose. TMA acts as anionic carrier in this study. Some other influencing parameters such as effect of pH, effect of stripping solution type, effect of mixing speed, effect of stripping solution concentration etc. were also thoroughly studied and reported here.

Experimental:

Reagents

The liquid membrane consists of a carrier, an extractant, and a diluent. The chemical used were of analytical grade reagents. The Ni (II) solution (feed phase) was prepared by dissolving a known amount of NiSO₄ in deionized water, which was used as the simulated waste water. Commercial kerosene (Chennai Petroleum Corporation, India) was used as diluents. The following inorganic salts, acid and organic solvents were used in the experiments without further purification. HCl (35.4%), TMA (99.0%), NiSO₄.6H₂O (99.0%), H₂SO₄ (98%), H₃PO₄ (85.0%), DMG, Acetone (99%) were purchased from Merck and were directly as received from the manufacturer.

Instrumentation

The absorbance of nickel (II) concentration in the aqueous phase was determined with uv visible spectra photo meter (Elico SL 159) and the absorption wavelength was 470nm using Dimethyl glyoxime as the indicator. The pH adjustment of source phase was done using Elico. For agitation of solution a shaker and stirrer was used (IKD-KS 50, India). Systronics Electrophoresis 606 was used to find out whether the metal is cationic or anionic.

Membrane preparation:

The experimental studies were carried out applying the BLM technique using a stirred transfer lewis type cell with bulk liquid membrane layered over the feed and product phase. The inner dimension of transport cell is 70 mm diameter × 195 mm depth for H-type. The size of the equipment is 120mm × 30mm × 40mm (length × width × height). The aqueous feed phase containing (30mg/L, 260 mol/L) nickel solution and strip phase containing (0.5N, 260 mol/L) Hydrochloric acid were taken in the BLM apparatus and the solutions stirred by mechanical stirrers at 250 rpm. These two layers were separated by the organic solvent such as Kerosene which acts as liquid membrane phase. Experiment samples were taken out from the feed and strip phases at known time, and the Ni (II) concentration was measured using spectrophotometer ($\lambda_{max} = 470\text{nm}$).

Results and discussion:

Effect of diluents in the membrane phase:

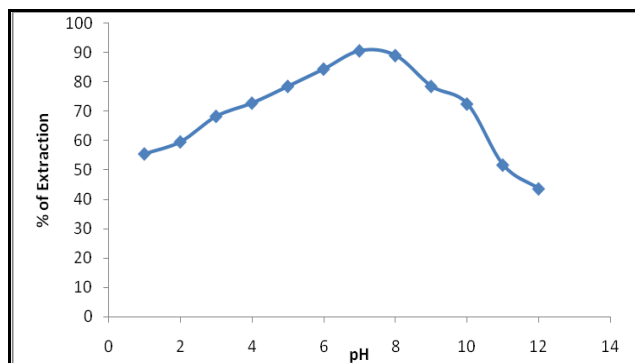
In this experiments, the carriers (1.3M/ TMA) prepared with various diluents such as Hexane, Kerosene, toluene, Xylene, Dichloromethane. It indicates that the aliphatic hydrocarbons have less transport flux (J_a^{max}) value compared with aromatic hydrocarbons. About 90% of Ni (II) was extracted in Kerosene-TMA. So, further studies were carried out using kerosene as diluents. This result was presented in Table.1.

Diluents	Percentage of extraction
Hexane	65.4
Kerosene	90.0
Toluene	63.9
Xylene	71.3
Dichloromethan	45.2

Effect of pH:

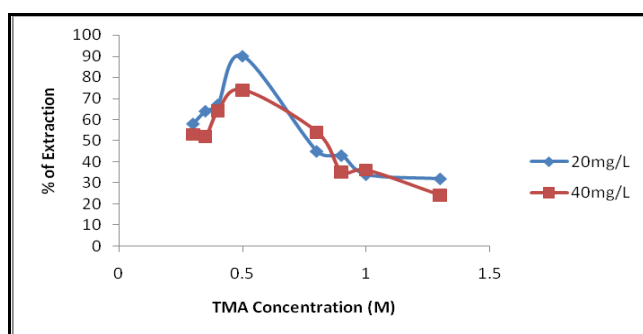
In order to investigate the effect of pH parameter experiments of nickel (II) from neutral medium through kerosene membrane containing TMA and the pH of the feed phase in the range 1 to 12 were carried out

Fig.1 shows the transport efficiency of Ni (II) gradually increasing in pH 1 to 7. The extraction efficiency of nickel also increases up to 90% at pH 7.0 ± 0.1 . After pH 8 to 12, the extraction efficiency decreased. This can be explained as when alkaline pH, the extraction was low, because the amine is strong base, in low pH. Thus, the further experiments to study the other parameters was carried out at pH 7.0 ± 0.1 . The maximum Ni(II) transport occurred at pH 7.



Effect of extractant concentration:

The extraction of nickel (II) increased by increasing of TMA concentration from 0.33M to 1.35M. While increase of extractant concentration from above moles hardly affected the extraction performance. Fig.3. The highest percentage value obtained in 0.5M. Further increase of extractant concentration decreased the extraction efficiency. It is due to the access of free extractant in membrane phase. Further increase in the extractant concentration leads to the decrease in the stripping reaction rate. This is because of nickel (II) remains in the complex form (in the membrane phase) without getting stripped. This in its turn affected the final recovery by the BLM process. Similar results were obtained by Othman *et al*¹⁴ result, thus is an optimum extractant concentration around 0.5M in the membrane phase. Therefore TMA concentration 0.5M was accepted as appropriate extractant concentration.



Effect of stripping solution type:

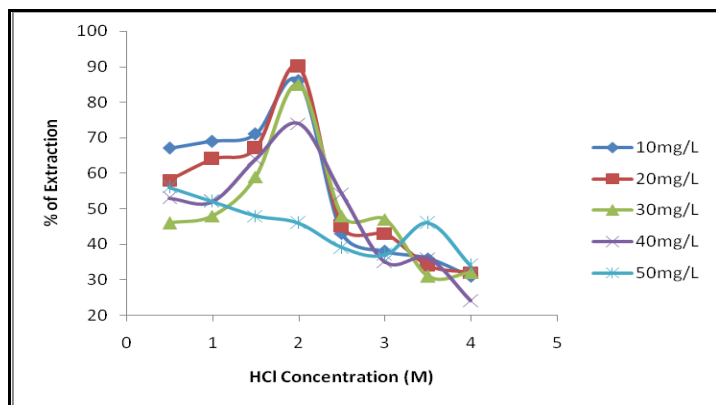
As reported in literature, the main factor for the BLM applicable is stability. In addition to mixing speed, extractant, another parameter is stripping solution types. Therefore, the selection of suitable stripping solution is considered to be one of the key factors for solute extraction. Here we examined the availability of stripping solution, H_3PO_4 , HCl , H_2SO_4 . The results are shown in Fig.2. As seen from Fig.2, HCl solution is more preferable in making the stripping solution. Therefore 2N HCl solution is selected as the best stripping solution.

Stripping agent	Percentage of Stripping
Sulphuric acid (1M)	60.3
Hydrochloric acid (2M)	75.5
Phosphoric acid (1M)	56.9
Nitric acid (1M)	34.8

Effect of stripping solution concentration:

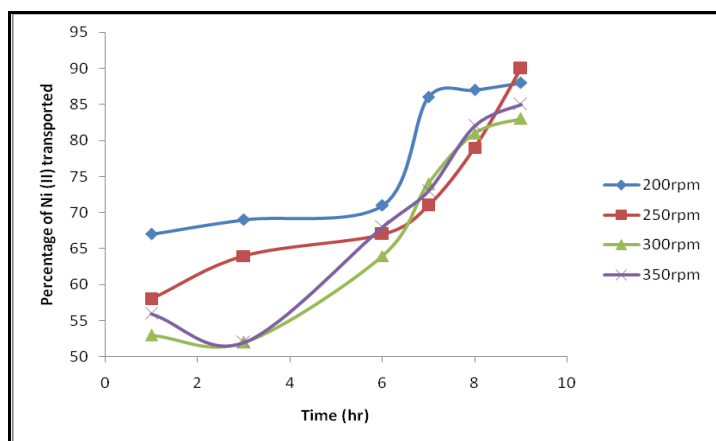
As the extraction step occurs in the interface between the acidic solution and the liquid membrane, the transport of metal necessarily requires a simultaneous back-extraction step at the opposite side of the

membrane. In the back extraction stage, The extractant is regenerated and the metal is stripped. The literature contains many options for accomplishing, the stripping of the nickel (II) complex, among them, solutions of H_3PO_4 , HCl , H_2SO_4 have been used. From this list, HCl solution was used as the stripping agent in the stripping side. The normality of (HCl) was varied between 10 to 50 mg/L. Results obtained were shown in Fig.4. it shows that the mass transfer rate is increased with increasing stripping concentration from 0.5 N to 2N. further increase in HCl concentration it has no significant changes on the transfer rate []



Effect of mixing speed:

The mixing speed plays a major role in the mass transfer rate of nickel (II) through BLM. The effect of stirring speed was studied in the range of 200 to 400 rpm in order to obtain optimal mixing speed that allows effective extraction of nickel (II) in the BLM system (Fig.5). Increase in extraction was observed when the mixing speed was increased from 200 to 400 rpm and beyond which no appreciable decrease in nickel (II) extract was observed up to 250 rpm. Above 250 rpm the extraction again decreased. As a result, an increase in the level of mixing speed would increase the interfacial area. Thus a 250 rpm mixing speed was maintained through the subsequent investigations. These results were presented in the fig.



Application of the developed BLM for textile wastewater

The wastewater from a local industry was selected for testing the applicability of the developed bulk liquid membrane system, which was neutral in nature ($pH 7 \pm 0.1$). Under optimized condition (feed phase = 260 mL of 30 mg/L, receiving phase = 260 mL of 0.5M Hydrochloric acid, membrane phase = 260 mL of diluents with kerosene, stirring speed = 250 rpm), the industrial wastewater was extracted and the extracted metal was successfully stripped into Hydrochloric acid solution.

Conclusions

The bulk liquid membrane method offers a simple approach for selective extraction of cationic nickel (II) for removal and recovery. The extraction efficiency of Ni (II) decreases with increasing the concentration of Ni (II). The extracted Ni (II) was successfully stripped into hydrochloric acid solution from loaded organic phase. The maximum extraction efficiency was obtained at $pH = 7.0 \pm 0.1$. Stirring speed was found at 250 rpm. Under optimized condition industrial wastewater also tested and the result was found to be satisfactory.

Acknowledgement

Our sincere thanks to UGC New Delhi, provided the laboratory facilities to carry out this research work.

References

1. Thauer RK. Enzymology- Nickel to the fore. Science., 2001, 293: 1264–1265.
2. Kalyakina, OP, Kononova ON, Kachin SV, Kholmogorov AG. Sorption preconcentration and determination of nickel in wastes of heat power industry by diffuse reflection spectroscopy. Bull. Kor. Chem. Soc., 2003, 24: 173–178.
3. Kristiansen J, Christensen JM, Henriksen T, Nielsen NH, Menne T. Determination of nickel in fingernails and forearm skin (stratum corneum). Anal. Chim. Acta., 2000, 403: 265–272.
4. Ahmetsurucu, Volkaneyupogu, Osman Tutkun. selective separation of cobalt and nickel by flat sheet supported liquid membrane using Alamine 300 as carrier, J. ind. eng. chem., 2012, 18: 629-634.
5. Xue Z, Hua Z, Yao N, Chen S. Separation and Recovery of Nickel and Cadmium from Spent Cd-Ni Storage Batteries and Their Process Wastes., Sep. Sci. Technol., 1992, 27: 213 -221.
6. Nogueira CA, Delmas F. New Flowsheet for the Recovery of Cadmium, Cobalt and Nickel from Spent Ni-Cd Batteries by Solvent Extraction, Hydrometallurgy, 1999, 52., 3: 267-287.
7. Gupta B, Deep A, Malik P. Extraction and Recovery of Cadmium Using Cyanex 923, Hydrometallurgy., 2001, 61: 65-71.
8. Rudnik E, Nikiel M. Hydrometallurgical Recovery of Cadmium and Nickel from Spent Ni-Cd Batteries, Hydrometallurgy., 2007, 89: 61-71.
9. Ho WSW, Sirkar KK, eds. Other new membrane processes, membrane Hand book, van Nestrand Reinhold., New York 1992, 885-012.
10. Yaddlah Yamini, Marzieh Chaloosi, Homeira Ebrahimzadeh. Highly selective and efficient transport of bismuth in bulk liquid membranes containing Cyanex 301. Separation and purification tech., 2002, 28: 43-51.
11. Jafari S, Yaftain MR, Parinejad M. Facilitated transport of cadmium as anionic iodo-complexes through bulk liquid membrane containing hexadecyltrimethyl ammonium bromide. Separation purification technology., 2009, 70: 118-122.
12. Parham H, Shamsipur M. Highly selective and efficient transport of mercury as $\text{Hg}(\text{NO}_2)_4^{2-}$ ion using Ba^{2+} -18-crown-6 as carrier. J. Membr. Sci., 1994, 86: 29-35.
13. Akhond, M., Shamsipur, M., Specific uphill transport of zinc as $\text{Zn}(\text{SCN})_4^{2-}$ ion using NA-Dicyclohexyl-18-crown-6 as carrier. J. Chin, Chem. Soc., 1996, 43: 225-229.
14. Othman N, Mat H, Goto M. Separation of silver from photographic wastes by emulsion liquid membrane system. J. membr. Sci., 2006, 282: 171-177.
