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Research Article

Extraction of Ni (II) and Co (II) using the emulsified liquid membrane (ELM) technique

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Abstract – Experiments were carried out to extract Ni (II) and Co (II) ions using a quaternary amine extractant (Aliquat336). The chemical process used is the emulsified liquid membrane (ELM) technique. The process study revealed the optimization of experimental parameters for extracting metal ions in their equimolar mixture. These metal ions were separated from their synthetic mixture. The results showed that cobalt and nickel could be extracted at 64.09% and 100% respectively from their equimolar mixture.

Keywords – Emulsion Liquid Membrane, Nickel, Cobalt, Span80.

I. INTRODUCTION

Solid or liquid industrial waste often contains heavy metals, which entail a significant pollution risk due to their high toxicity and environmental dispersion.

In the industrial sector, it is often the case that waste is difficult to recover. Storing this waste in controlled landfill sites or facilities does not guarantee the total absence of risk of soil and groundwater pollution from the infiltration of toxic metals.

In the field of hydrometallurgy, the extraction and preconcentration of metals is a major economic challenge.

The complexity and difficulty associated with effluents containing heavy metals has prompted researchers to explore a number of possible solutions.

Traditional methods for extracting metal ions from environmental matrices include the following processes: adsorption, precipitation, ion exchange, solvent extraction and electrochemical recovery, reverse osmosis, nanofiltration [1], ultrafiltration, precipitation of metal hydroxides followed by sludge filtration, fixation on ion exchange resins, chemical and biological oxidation, electrochemical techniques (electrodialysis, electroflotation, etc.) and solvent extraction techniques, which have included traditional liquid-liquid extraction and liquid membrane extraction.

The latter, which are currently the most widely used treatment techniques, may prove ineffective due to legislative, technical or economic constraints.

Emulsified Liquid Membrane Extraction (ELME) is a simple process that offers many advantages for the recovery of metal ions from solid or liquid effluents.

The MLE technique allows the selective separation of metal ions and continuous operation without the need for frequent regeneration, combined with low chemical impact and minimal energy consumption, these advantages have made the technique highly competitive economically [2].

II.MATERIALS AND METHOD

II.1. protocol and determination of cobalt (II) and nickel (II) ions

The solution to be determined was an equimolar mixture of Ni(II) and Co(II). These two metals were quantified using the UV-visible absorption spectrophotometry technique.

II.1.1. For nickel (II)

We begin by preparing the solutions required for the determination of Ni^{2+} ions using an arsenazo (III) solution with a concentration of 10^{-3} M. An aqueous nickel(II) solution is then prepared at 100ppm.

Quantification of nickel (II) was obtained using the UV-Visible absorption spectrophotometry technique. The absorbance of the Arsenazo(III)- Ni(II) complex is monitored at the characteristic wavelength.

II.1.2. FOR COBALT (II)

Similarly, solutions were prepared for the determination of Co^{2+} ions using the 10^{-3}M PAR concentration. The cobalt metal solution used is 100ppm. Solutions with lower concentrations are obtained by diluting this stock solution.

PAR forms a stable complex (PAR-Co) with Co (II) at pH=10, for which a pH=10 buffer solution was prepared.

Quantification of the cobalt(II) is obtained using the UV-Visible absorption spectrophotometry technique. The absorbance of the PAR-Co(II) complex is monitored at the characteristic wavelength.

II.2 Preparation of the emulsion

The membrane intended to extract the metal is made up of an extractant, Aliquat336 (N-methyl-N, N-dioctylotan-1-ammonium chloride), in a volume of 3.75 ml of hexane diluent, with the surfactant being SPAN 80. The purification solution chosen is nitric acid HNO_3 0.5M, which is added dropwise to the membrane phase. The emulsions are created using a VORTEX. Stirring was maintained at 1800 rpm for 20 minutes.

II.3. The extraction procedure

The membrane is dispersed in an Erlenmeyer flask containing 31.25 ml of the solution to be treated (feed solution), by mechanical stirring at 210 rpm for 30 minutes.

Once the extraction is complete, the membrane phase and the metal phase are separated using a separating funnel, and a volume of 100 µl of the feed phase is taken after extraction for UV-visible assay.

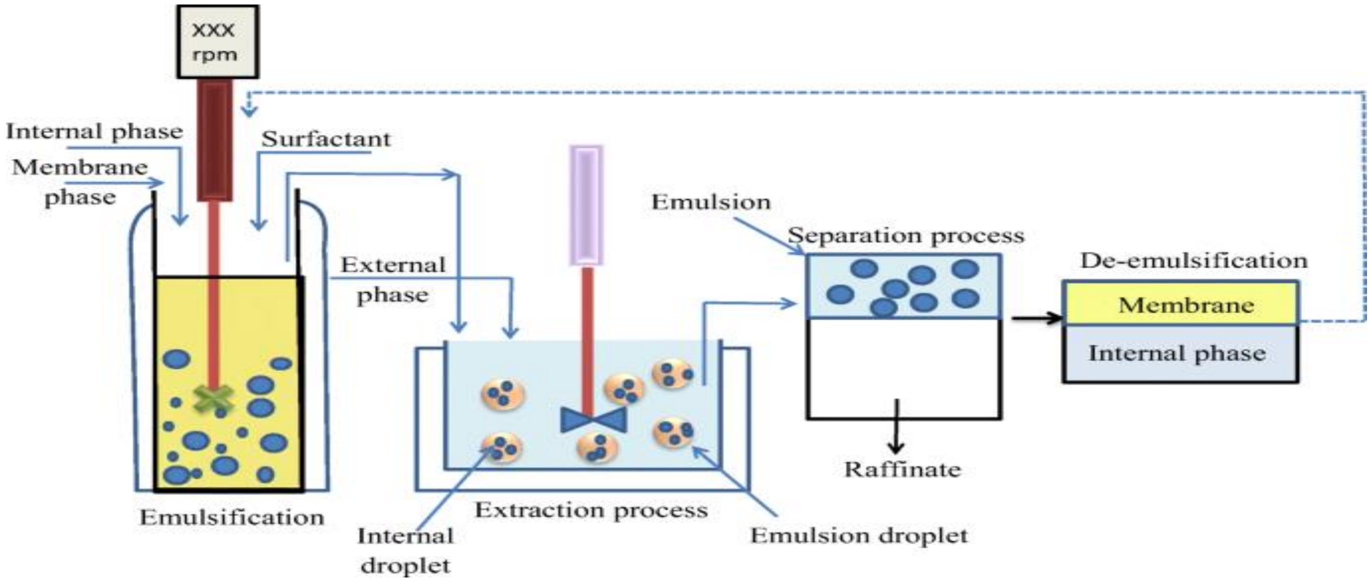

Figure 1. Emulsion Liquid Membrane ELM

II.4. Optimisation of the determination of Ni(II) and Co(II) by visible complexometry

The complexing agent Arsenazo(III) is widely known and commonly used for the spectrophotometric determination of various metal ions [3], in particular nickel(II). The determination of cobalt(II) ions with PAR (4-(2-pyridylazo)-resorcinol) by UV-Visible absorption spectrophotometry is a rapid and sensitive method [4]. Arsenazo(III) forms a stable complex with the Ni(II) ion (stoichiometry 1/6), at neutral pH, absorbing at a characteristic wavelength of 615 nm (see Figure 2). PAR also forms a stable complex with the Co(II) ion with the same 1/6 stoichiometry, absorbing in the visible at 512 nm (see Figure 2).

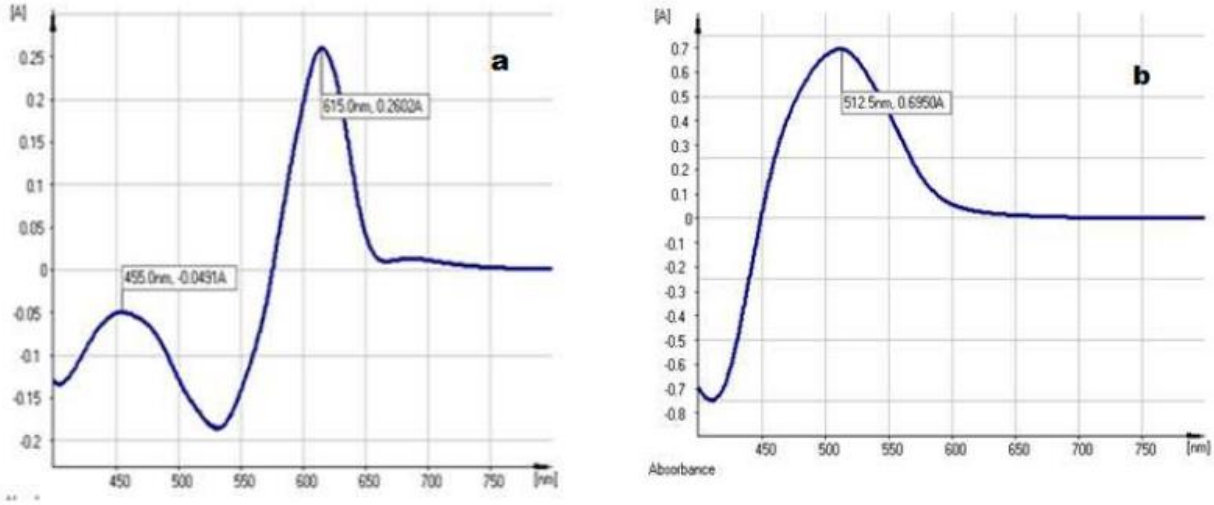

Figure2: Visible absorption band of ions ; (a) : Ni(II) and (b) : Co(II)

II.5. Study of the extraction of Ni(II) and Co(II) by MLE

The expression used to calculate the extraction yield (R) is as follows:

$$\boldsymbol{R}\ (\%) = \frac{\boldsymbol{C_i - C_f}}{\boldsymbol{C_i}} \times \mathbf{100}$$

C_i: the initial concentration of Ni(II) or Co(II) before extraction.

C_f: the concentration of Ni(II) and Co(II) after extraction.

1. Effect of extractant concentration

The choice of carrier plays a crucial role in the emulsified liquid membrane (ELM) extraction process. The carrier or extractant must form a complex with the metal, being soluble in the organic membrane phase and insoluble in the internal (purification) and external (feed) aqueous phases [5].

In this experiment, nickel (II) and cobalt (II) ions were extracted with 0.5% m/m SPAN 80 while varying the concentration of Aliquat 336 extractant from 0.1 to 3% m/m.

The results are shown in Figure 3.

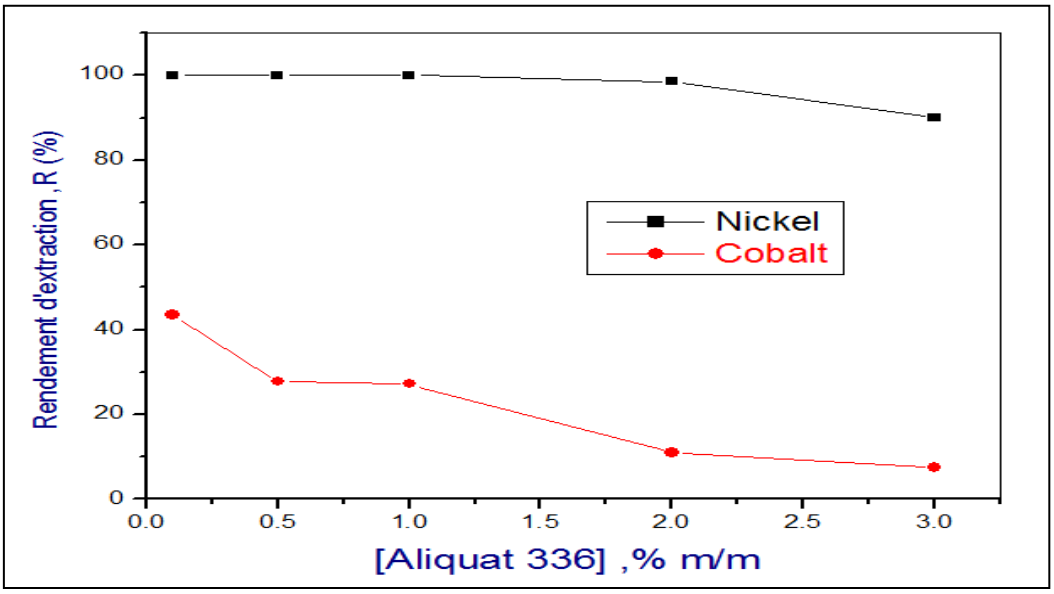

Figure 3: Ni(II) and Co(II) extraction yield as a function of aliquate concentration336

The figure shows that the extraction yields for Ni(II) reached 100% and remained virtually constant, while a maximum yield of 43.56% was observed for Co(II) ions at a content of 0.1% m/m, with extraction subsequently decreasing.

However, it has been found that by exceeding the Aliquat336 contents required for the extraction of Co(II) and Ni(II), extraction by MLE does not become advantageous [6]. This is due to the increase in the viscosity of the liquid membrane, which leads to the formation of large emulsion globules that become unstable. Also, when the threshold concentration of Aliquat336 is exceeded, the emulsion droplets swell, diluting the purification phase and consequently reducing the extraction yield [6,7,8,9].

We can therefore say that 0.1% m/m is the optimum quantity of aliquat336 for this extraction.

2. Influence of pH on MLE extraction

The extraction of Ni(II) and Co(II) ions by the MLE technique involves steps that take place at the interface between the metallic phase and the liquid membrane. In addition, the process necessarily involves a simultaneous back extraction step at the interface between the membrane and the internal phase. At this stage the extractant is regenerated and the metals are pre-concentrated.
The pH values were taken from 1 to 9, and the results are shown in Figure 4.

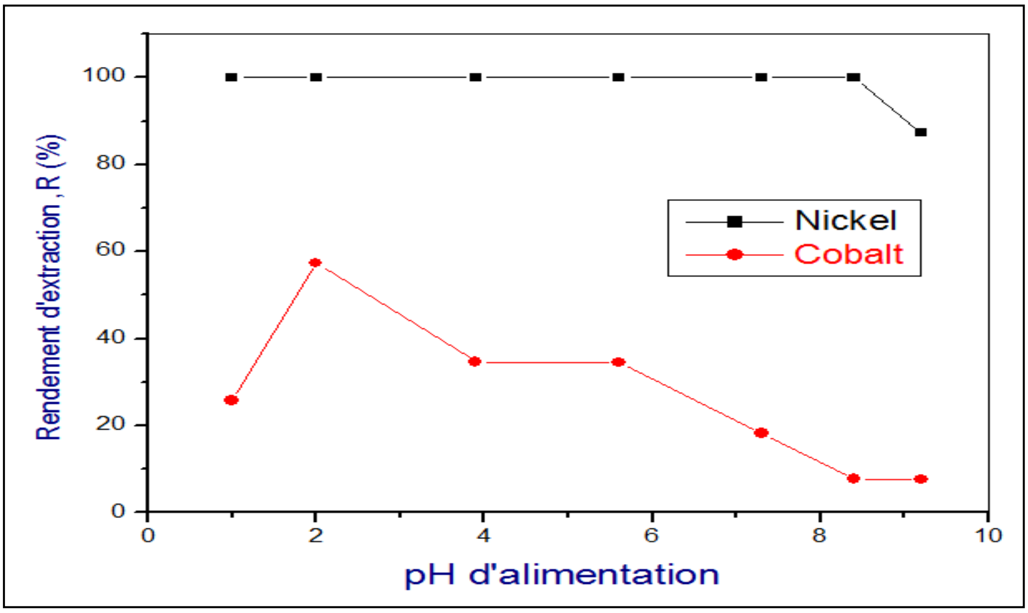

Figure 4: Ni(II) and Co(II) extraction yield as a function of pH of the feed phase

From this curve, the extraction of Co(II) and Ni(II) ions changes with increasing pH of the feed phase.

The extraction of Ni(II) is much more advantageous, the extraction yield is and remains practically constant (100%), but at pH equal to 9 the extraction yield decreases to 87.26%, this decrease can be explained by the predominance of Ni(II) species (see figure 4) which shows that at pH = 9 the hydroxide form $Ni(OH)_2$ appears, the latter is in the form of a precipitate (green), i.e. consumption of the free form of Ni(II), and therefore a decrease in its concentration, and consequently a decrease in the extraction yield.

For cobalt(II), the extraction yield reached a maximum of 57.40% at pH = 2. The theory shows that Aliquat336 can be affected by hydronium ions by disrupting the quaternary amine ion pair [10].

3. Effect of feed phase concentration on MLE extraction

Variation in solute concentration affects the transport driving force and mass transfer rate via the emulsified liquid membrane [10]. The influence of the feed phase concentration (external phase) on the extraction of Co(II) and Ni(II) is shown in Figure 5.

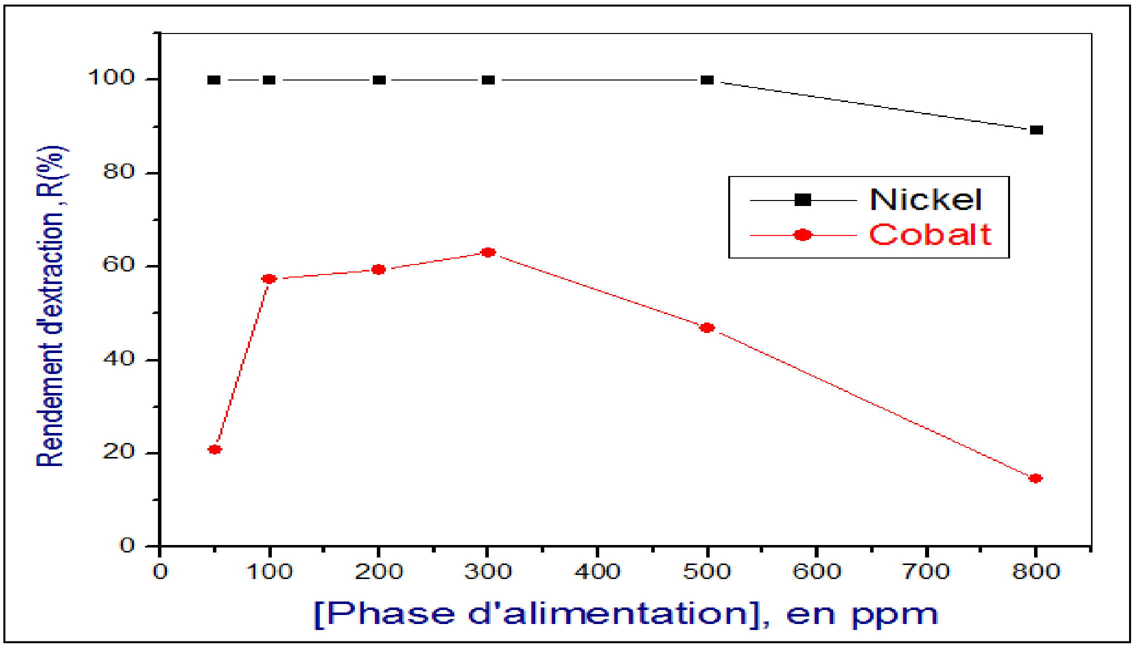

Figure 5: Ni(II) and Co(II) extraction yield as a function of feed phase concentration

The figure shows that the Co(II) extraction yield increases with the increase in Co(II) ions in the external phase. This is clearly observed for a concentration of 300 ppm where a maximum of 63.01% is reached after 30 minutes stirring, under the same emulsion conditions. This is due to the increase in the driving force (cobalt ion concentration gradient) when the concentration in the external phase increases from 300 ppm. When the external phase is further concentrated (500 ppm), extraction yields weaken and become virtually independent of the feed phase concentration. This phenomenon is attributed to the possibility of swelling of the emulsion globules due to the difference in strength. These conclusions are similar to those found by other authors [11,12,13].

The extraction yield remains at a maximum (100%) in the initial Ni(II) concentration range between 50 and 300 ppm. For the extraction of Nikel (II), this becomes very interesting in terms of hydrometallurgy and recycling plans. We note that the extraction yields of Ni(II) and Co(II) ions decrease for high feed concentrations. This may be due to saturation of the extraction interface [13]. The extraction mechanisms for each metal using Aliquat336 are given.

$$Ni^{2+}_{(aq)} + R_4NCl_{(0)} + 2\ NO_3^-{}_{(aq)} \rightleftarrows Ni(NO_3)_2 .\ R_4NCl_{(0)}$$

$$Co^{2+}_{(aq)} + R_4NCl_{(0)} + 2\ NO_3^-{}_{(aq)} \rightleftarrows Co(NO_3)_2 .\ R_4NCl_{(0)}$$

4. Effect of surfactant concentration on MLE extraction

The surfactant plays a very important role in the design and in the MLE extraction process. The choice of surfactant is a crucial parameter. It must behave as an emulsifier [14,15].

Figure 6 shows the influence of the SPAN 80 surfactant concentration in the membrane phase as a function of the nickel(II) and cobalt(II) extraction yield.

The best Co(II) extraction yield was 64.09%, with SPAN 80 present at 0.1% w/w, in the concentration range between 0.05 and 2% w/w (0.05; 0.1; 0.5 ; 1.5 and 2% w/w), which can be explained by the increased stability of the emulsion globules. Also, the increase in surfactant concentration leads to the formation of small emulsion globules, thus increasing the contact surface area and consequently the extraction yield [1,2].

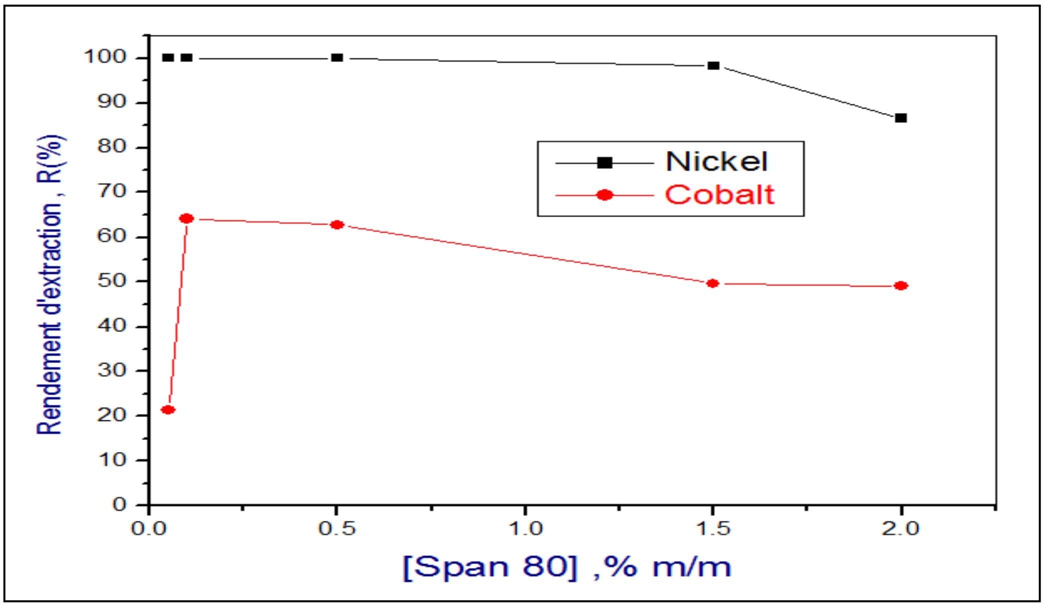

Figure 6: extraction yield of Ni(II) and Co(II) as a function of span80 concentration

However, above this concentration, the presence of an excess of surfactant molecules at the organic-purification interface generates a high interfacial occupation, causing a reduction in extraction efficiency, which will hinder the transfer of ions to the purification phase [16].

We can therefore say that 0.1% w/w is the optimum quantity for this extraction.

III.CONCLUSION

The study of the extraction of the two metal ions by Aliquat 336, using the SPAN 80 surfactant, gave a maximum extraction yield of 43.56% and 100% for Co(II) and Ni(II) respectively. This was obtained with surfactant quantities of 0.5% m/m and an extractant quantity of 0.1% w/w.

Varying the pH of the feed phase on the extraction of Ni(II) and Co(II) in their equimolar mixture gave a maximum extraction yield of 57.40% at a pH equal to 2, varying the pH values from 1 to 9.

By varying the initial concentration of the feed phase, the extraction of Co(II) and Ni(II) reached 63.01% and 100% respectively, from 300 ppm cobalt(II) and nickel(II).

The study of the effect of the SPAN 80 surfactant concentration on the extraction of these two metal ions gave a yield of 64.09% and 100% for Co(II) and Ni(II) respectively at a SPAN80 content of 0.1%w/w.

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