

Uptake of Palladium Using Liquid Emulsion Membrane Technique

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Abstract

The waste stream generated at the reprocessing facility (first cycle raffinate-Hi level waste) contains Palladium as one of the fission products. The recovery of Palladium from the waste stream reduces the activity burden and lends its use for catalytic applications.

This paper deals with its recovery from the waste stream generated at various stages of nuclear fuel cycle using liquid emulsion membrane technique. Complexants chosen were based on the chelating ability with metal ions with high stability constant in the waste stream. Complexants 8-hydroxy quinoline, Thio salicylic acid, dithiazone, cupferron, 1-Nitroso-2-naphthol, Quinalizarin were chosen as complexants. The effect of surfactant concentration, complexant concentration, agitating speed, and emulsification time on the emulsion stability have been studied. Treat ratio, the ratio of the emulsion phase to the external phase and the impeller speed on the removal of Palladium have been studied. Results show that stable emulsion could be prepared by using 1.5 ml of surfactant span 80 in 50 ml emulsion, with impeller speed 3000 RPM, for duration of 20 minutes using complexants of strength of 70-200 moles/m³. Effective recovery of Pd could be achieved by using strippant of pH 1-3.7 in the emulsion phase at 250-300 RPM of impeller speed.

Keywords: emulsion, surfactant, complexants, permeation

1. Introduction

Conventional liquid extraction suffers from the drawbacks of being equilibrium limited and need for a separate stripping operation. Liquid emulsion technique has been recognized as promising technology as it overcomes these drawbacks of the conventional liquid extraction. In general, the liquid emulsion membrane is prepared by forming an emulsion of two immiscible phases and then dispersing the emulsion into a third phase. The transport of dissolved material through the liquid membrane is termed as permeation. Since the liquid film offers much higher diffusivities and area exceeding 10,000 m²/m³, the fluxes of the liquid membranes are very high. Furthermore liquid membranes can be tailor made for specific applications.

LM technology can carry out extraction and stripping process simultaneously and have benefit of non equilibrium mass transfer and uphill effect where solute can be moved from low to high concentrated solution (Franken, 1997; Gu et al., 1994; Giaikwad, 2004). The main types of liquid membrane systems include Emulsion Liquid Membrane (ELM) (Li et al., 1997), Supported Liquid Membrane (SLM) (Bloch et al., 1967), Bulk Liquid Membrane (BLM), Flowing Liquid Membrane (FLM) (Teramoto et al., 1989), Supported Emulsion Liquid Membrane (SELM) (Fouad et al., 2008; Sonawane et al., 2007), Hollow Fibre Contained Liquid Membrane (HFCLM) (Gabelman et al., 1999) and Supported Liquid Membrane with Stripping Dispersion (SLMSD) (Basualto et al., 2003; Gu et al., 2006) etc. The potential advantages of LM techniques over traditional separation techniques and solid membranes techniques are lower capital and operational costs, lower energy and extractant consumption and higher concentration factors and fluxes. However LM techniques have not been adopted to large scale industrial processes yet (Zhang et al., 2000; Danesi et al., 1987; Gu et al., 2003; Lin et al., 2004; Neplenbroek et al., 1992; Bechiri et al., 2008; Zha et al., 1995) primarily due to lack of emulsification and deemulsification steps in ELM and SELM processes and membrane resistance in BLM and HFCLM processes etc. (Ren et al., 2007).

2. Experimental

2.1 Materials

The membrane phase used is a homogeneous mixture of Xylene as organic solvent, span 80 as surfactant. AR grade extractants 8-hydroxy quinoline, Thio salicylic acid, dithiazone, cupferron, 1-Nitroso-2-naphthol, Quinalizarin used were procured from Ms. E. Merck. The internal stripping phase is aq. Solution of Hydrochloric acid.

2.2 Preparation of Emulsion

The emulsion was prepared in a glass vessel of 100 mm dia. 25 ml of the organic phase was prepared by adding surfactant span 80 in water to a 25 ml of Xylene. 10 ml of the internal strip phase HCl was added drop wise to the organic phase and the contents were simultaneously stirred by a motor fitted with a turbine impeller of 2 cm dia. The RPM of the impeller was measured using Digital Tachometer with totalizer/counter of make Metravi NCTM-1000. Excellent dispersion was observed and a milky white emulsion was obtained.

2.3 Effect of Parameters and Emulsion Stability

2.3.1 Role of Surfactant Strength on the Emulsion Stability

Since the surfactant plays an important role in the stability of the emulsion, the effect of surfactant strength on the emulsion stability was studied by adding 25 ml of 0.8 to 12 % V/V (0.2 ml to 3 ml of surfactant span 80 in 25 ml of water) in 25 ml of Xylene. The so prepared solution was stirred at 3000 RPM for 20 minutes and the emulsion so prepared was studied for the stability by finding out the time needed for the dispersion to take place. Figure 1a shows the emulsion stability time as function of strength of surfactant.

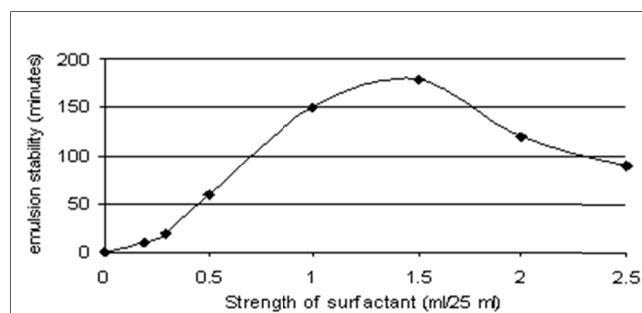


Figure 1a. Role of surfactant strength on the emulsion stability

2.3.2 Role of Agitation Time and Agitation Speed

Since stable emulsion needs optimum agitation speed at optimum time to withstand external variation in parameters, experiments on the study of agitation speed and agitation time was carried out by subjecting the solution for preparing the emulsion (aqueous phase containing the surfactant span 80 in water added to Xylene) to different agitation speed varying from 1000-4000 RPM for 20 minutes duration. Figure 1b shows the variation of emulsion stability time as function of speed of agitator.

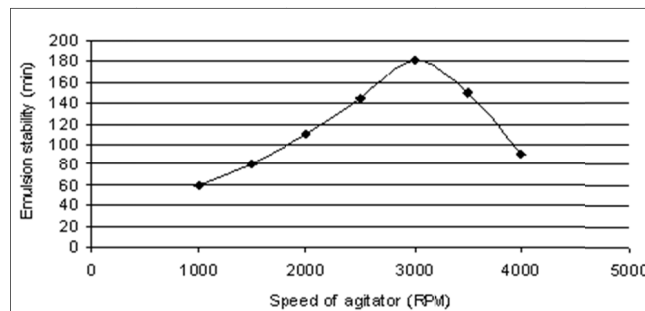


Figure 1b. Effect of agitator speed on the emulsion stability

The experiment was repeated and the emulsion prepared at different agitation times (5 to 60 minutes) were studied for their emulsion stability time as function of agitation time. Figure 1c shows the emulsion stability duration as function of agitation time.

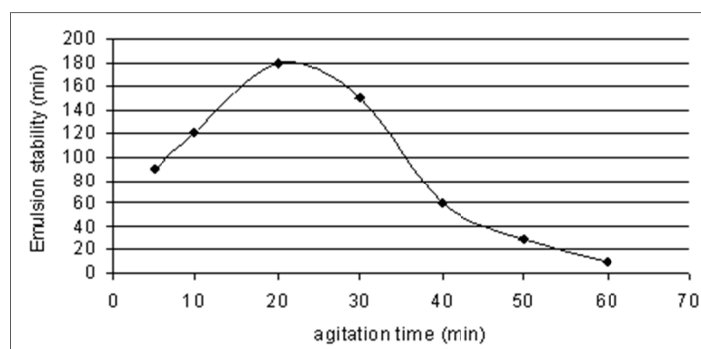


Figure 1c. Effect of agitation time on the emulsion stability

2.3.3 Role of Complexant Strength on the Stability of Emulsion

The complexant included in the emulsion when added to the solution containing the Palladium ion to be recovered extracts Palladium in to emulsion phase. Even though the higher strength of complexant may aid more recovery of metal ion, the limiting factor in the complexant strength is the emulsion stability due to complexant. Hence it is imperative to study the emulsion stability time as function of complexant strength included in the emulsion. The emulsion stability time was studied by preparing emulsion using 25 ml of aqueous solution 6% V/V of surfactant span 80 in 25 ml of Xylene with different complexants of strength 0.07 to 0.2 moles/L at 3000 RPM for 20 minutes. Figure 1d shows the emulsion stability time as function of complexant strength. In all the cases, the emulsion stability was studied from the time when the dispersion takes place as the criterion.

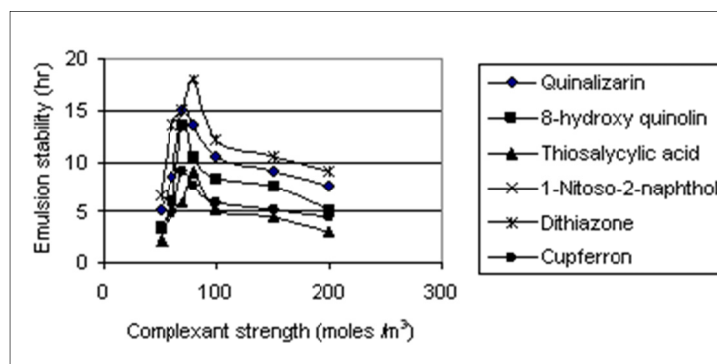


Figure 1d. Effect of complexant strength on emulsion stability

2.4 Effect of Operating Parameters on Synthetic Effluent

The emulsion prepared by using 25 ml of aqueous solution of 6% V/V of surfactant span 80 and 100 moles/m³ of the complexants viz. 0.4349 g 1-nitroso-2-naphthol, 1.583 g dithiazone, 0.3629 g 8-hydroxy quinoline, 0.3049 g thiosalicylic acid, 0.3875 g cupferron, 0.6804 g quinalizarin in 25 ml of Xylene at impeller speed of 3000 RPM for 20 minutes was used for the experiment on the uptake of Palladium from the aqueous solutions.

2.4.1 Effect of pH of the Strippant Solution

Since the pH of the strippant included in the emulsion is an important parameter for successful recovery of Palladium from the organic phase of emulsion to the aqueous phase of the emulsion, 50 ml of the so prepared emulsion was added to 50 ml of Palladium solution of strength 0.09 mM with the strippant pH varying from 1.1 to 7.2. The mixed mass was stirred at 250 RPM. The emulsion phase was separated, demulsified using sodium

chloride for the recovery of Palladium. The aqueous phase was analyzed for Palladium by AAS. Figure 2 shows the % uptake of Palladium a function of pH of the strippant.

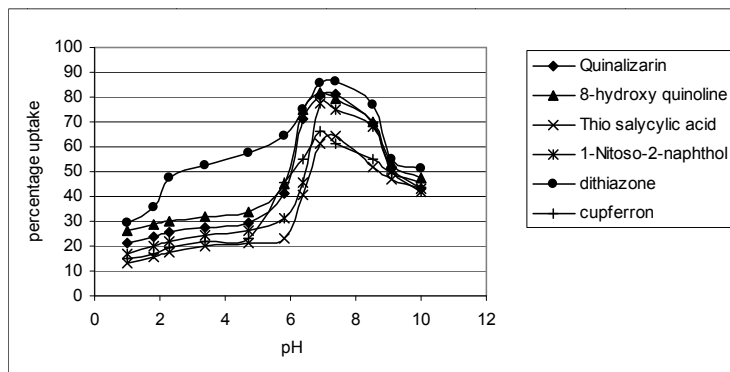


Figure 2. Uptake of Palladium as function of pH of strippant

2.4.2 Kinetics of Uptake of Palladium

To find out the kinetics of removal of Palladium, 50 ml of emulsion prepared using different complexants with the stripping solution at the optimum pH was added to 50 ml of Palladium solution of strength 0.09 mM and the solution obtained was stirred at 250 RPM. The emulsion and aqueous phases were separated and the aqueous phase was analysed for Palladium at interval of 15 minutes. Figure 3 shows the % removal of Palladium as function of time.

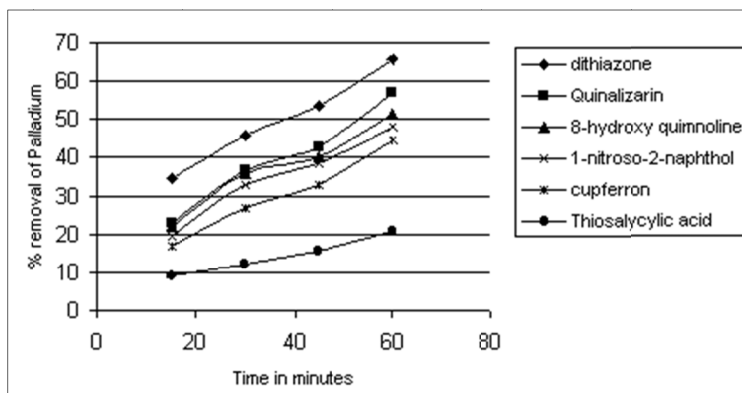


Figure 3. Kinetics of uptake of Palladium

2.4.3 Effect of Extractants on the Uptake of Pd

The extractant helps in thinning the membrane phase by reduction in the viscosity of the membrane phase. This will help in the transport of the solute ions for extraction. But beyond some concentration of extractant over a certain limit decreases the stability of the emulsion. Hence a compromise has to be made on the extractant strength which was done by adding 50 ml of emulsion prepared by including different extractants at 0.07 mole/L to 0.2 moles/L. to 50 ml solution of Palladium of strength 0.09 mM at 1:1 Treat Ratio at the optimum pH of the strippant for each extractant by stirring the mix at 250 RPM for 1 hour. The emulsion and aqueous phases were separated and the strength of Palladium was analyzed in the aqueous phase. Figure 4 shows the uptake of Palladium as function of strength of different complexants.

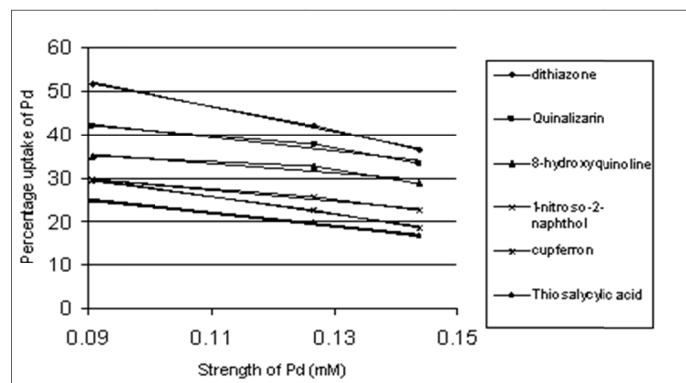


Figure 4. Uptake of Pd as function of different strength of Pd

2.4.4 Effect of Treat Ratio

Since the complexant present in the emulsion takes up the metal ion from the aqueous solution phase and emulsion membrane provides good surface area for permeation to take place, the volume ratio of emulsion to the aqueous phase containing the metal ion to be extracted called the treat ratio is a very important parameter. Experiments on the uptake of Palladium using emulsion membrane were carried out at optimum pH in the stripping solution for different complexants at different emulsion to solution volume ratio (treat ratio), 1:1, 1:2 and 1:3 keeping the aqueous solution containing the Palladium as 50 ml and the emulsion containing various complexants at 50, 100 and 150 ml giving rise to the treat ratio as 1:1, 1:2 and 1:3. After stirring at 250 RPM, the emulsion and aqueous phases were separated, and the aqueous phase was analysed for left over Palladium by AAS. Figure 5 shows the % uptake of Palladium as function of treat ratio.

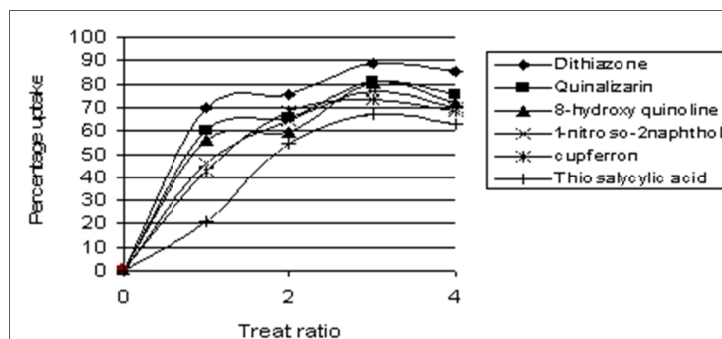


Figure 5. Uptake of Pd as function of treat ratio

2.4.5 Effect of Agitation Speed

After adding the emulsion to the aqueous solution from which the metal to be extracted, the permeation of the metal ion to the emulsion phase for complexation needs effective mixing of the two. Mixing beyond optimum RPM also breaks the emulsion phase which disrupts the extraction and hence the efficiency of the uptake comes down. The effect of agitation speed on % uptake palladium in the effluent with Palladium of strength 0.1436 mM of Pd was studied. Figure 6 shows the % uptake of Palladium as a function of RPM of the impeller.

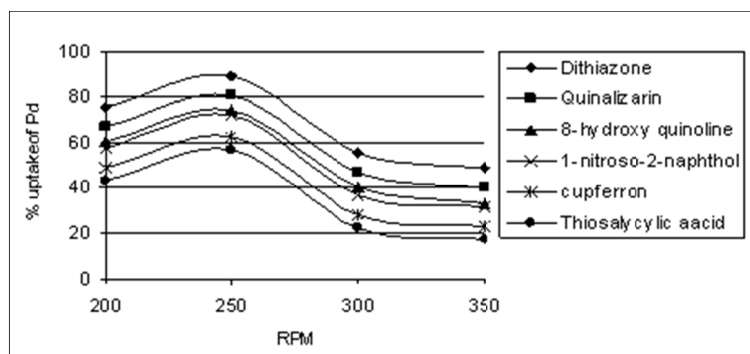


Figure 6. Optimization of RPM in the uptake of Pd

2.4.6 Effect of Feed Concentration

Since the uptake of the metal involves permeation and complexation of the metal ion, the extent of the uptake of the metal ion depends upon the initial strength of the feed. The % uptake of the metal ion was studied by taking 50 ml of the feed solution containing Palladium of strength 0.08186 mM to 0.14367 mM and 150 ml of the emulsion containing the complexants of strength 0.18-0.20 moles /L. The % uptake of the metal ion as a function of feed strength for various complexants is shown in Figure 7.

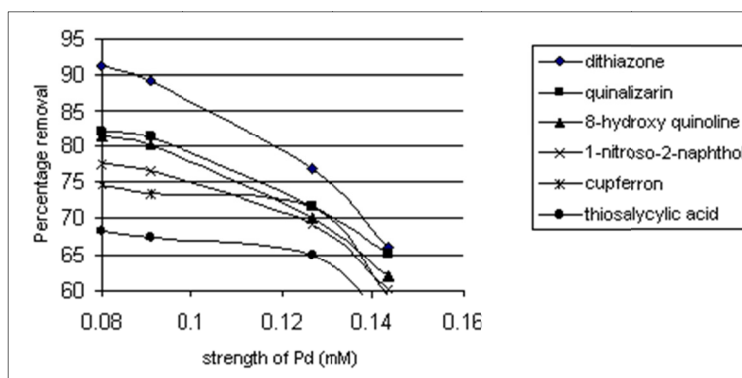


Figure 7. Uptake of Pd as function of strength of Pd

3. Results and Discussion

Membrane breakage and membrane swelling are the causes for the emulsion instability. This is measured by the time required for the emulsion to remain stable beyond which dispersion takes place. The various factors involved in the membrane instability are complexant concentration, surfactant concentration, mixing time and speed of agitation for the preparation of emulsion.

3.1 Stability of Emulsion

3.1.1 Strength of Surfactant

A stable emulsion needs proper surfactant in proper strength. From Figure 1a, it is observed that the emulsion stability measured in minutes is found to increase from 0.8 % of the surfactant to 6% giving rise to emulsion stability duration from 10 minutes to 180 minutes and then it starts coming down to 90 minutes at 1% V/V. Hence the surfactant strength was fixed as 6% V/V for the preparation of the emulsion.

3.1.2 Speed of Agitator and Agitation Time

From Figure 1b, it is observed that increasing mixing time from 5 to 20 minutes the emulsion stability was from 90 to 180 minutes and from mixing time of 20 minutes to 60 minutes the emulsion stability time was from 180 minutes to 10 minutes. Hence the mixing time for emulsion was optimized as 20 minutes.

From Figure 1c, it is observed that for mixing time of 20 minutes, the emulsion stability time was increased from

60 to 180 minutes from 1000 RPM to 3000 RPM and then it comes down from 180 minutes to 90 minutes from RPM of 3000 to 4000 indicating the optimum agitation time of 3000 RPM for the preparation of stable emulsion.

3.1.3 Complexant Strength

Figure 1d indicates that membrane with high concentration of complexant decreases the stability but with low concentration of complexant decreases the solute transport rate. Hence an optimum complexant concentration was taken in order to maintain stability and solute transport. From Figure.1d it is observed that with the complexants quinalizarin, Thiosalicylic acid, 1-nitroso-2-naphthol the stability of the emulsion was found to be very high at 80 moles/m³ whereas with the complexants 8-hydroxyquinoline, dithiazone and cupferron the stability of the emulsion was found to be high at 70 moles/m³. By repeated trials, a good emulsion could be prepared which contains 6 % (v/V) surfactant, 70-80 moles/m³ extractant prepared at 3000 rpm at mixing time of 20 minutes.

3.2 Effect of Operating Parameters

3.2.1 Effect of pH of the Strippant Solution

From Figure 2, we observe that using dithiazone when the strippant PH was between 1 to 3, the % uptake was 64.5-65.6 whereas from pH 3 to 4.6, the % uptake was 65.6-55.1. Using quinalizarin when the strippant PH was between 1 to 2.5, the % uptake was 54.8-56.9 whereas from pH 2.5 to 4.6 the % uptake was 56.9 to 41. Using 8-hydroxy quinoline, when the strippant PH was between 1 to 2.5, the % uptake was 49.3 to 51.6 whereas from pH 2.5 to 4.6, the % uptake was 51.6 to 36. Using 1-nitroso-2-naphthol when the strippant PH was between 1 to 1.5, the % uptake was 46.7 to 47.8 whereas from pH 1.5 to 4.6 the % uptake was 47.8 to 27.8. Using cupferron when the strippant PH was between 1 to 1.5, the % uptake was 38.9 to 44.5 whereas from pH 1.5 to 4.6, the % uptake was 44.5 to 23.3. Using Thiosalicylic acid, when the strippant PH was between 1 to 1.5, the % uptake was 19.8-20.6 whereas from pH 1.5 to 4.6 the % uptake was 20.6-11.8.

Hence the optimum pH of the strippant included in the emulsion for the various complexants are as follows:

For dithiazone the pH of the strippant is optimized as 3, for quinalizarin and 8-hydroxy quinoline the pH of the strippant is optimized as is 2.5, for 1-nitroso-2-naphthol, cupferron and thiosalicylic acid the pH of the stripping solution was 1.5.

3.2.2 Kinetics of Removal of Palladium

From the Figure 3 on the kinetic studies of removal of Palladium using emulsion technique, we observe that with dithiazone, % removal ranges 34.7-65.6, with quinalizarin it ranges 22.8 to 56.9 with 8-hydroxy quinoline it ranges 21.9 to 51.6, with 1-nitroso-2-naphthol it ranges 19.3 to 44.5 and with thiosalicylic acid it ranges 8.9 to 20.6 in a period of 15 to 60 minutes.

3.2.3 Effect of Strength of Complexant in the Emulsion

From Figure 4, we observe the % removal of Pd from 0.09 mM solution using emulsion containing different complexants of strength 0.07 to 0.2 moles /L at treat ratio of 1:1 at optimum pH of the strippant as function of moles of complexants. It is seen that the % removal of Pd was maximum at 0.15 moles/L for dithiazone 8-hydroxy quionoline and 0.18 moles /L for quinalizarin, 1-nitroso-2-naphthol, cupferron and thiosalicylic acid.

3.2.4 Effect of Treat Ratio

The treat ratio defined as the ratio of the volume of emulsion phase to the aqueous phase containing the metal ion to be recovered plays a dominant role in the uptake of metal since more emulsion phase provides more complexant for the metal to permeate the membrane phase and hence get recovered. It is observed from Figure 5, that as the treat ratio is increased from 1:1 to 1:3 the % recovery increased from 69.6 to 89.1, 60.1 to 81.4, 55.7 to 80.1, 45.9 to 76.7, 42.5 to 73.4, 21.3 to 67.4 for dithiazone, quinalizarin, 8-hydroxy quinoline, 1-nitroso-2-naphthol, cupferron, and thiosalicylic acid due to larger availability of the complexant. With further increase in the membrane phase, the % recovery was not improved. Since large interfacial area between external and emulsion phase is required which gets reduced due to the formation of a dense miscelle interfacial layer at the membrane phase which resists solute transport. Hence the treat ratio for the maximum recovery of Pd was fixed at 1:3 in all the cases.

3.2.5 Effect of Agitation Speed

For permeation of the metal ion and subsequent reaction with the complexant present in the emulsion, sufficient agitation of the mixture is needed. If the agitation time and agitation speed is more than the optimum level there is likelihood of the breakage of the membrane and leakage of the metal ion back to the solution phase. It is

observed from this Figure 6 that as the speed is increased from 200 to 400 rpm the % solute recovered from the external phase changed. For dithiazone it increased from 65.4 to 69.6 at RPM 200 to 250 and then it dropped to 48.6 at 350 RPM. For Quinalizarin it increased from 55.9 to 60.1 at RPM 200 to 250 and then it dropped to 40.1 at 350 RPM. For 8-hydroxy quinoline it increased from 51.1 to 55.7 at RPM 200 to 250 and then it dropped to 33.3 at 350 RPM. For 1-nitroso-2-naphthol it increased from 42.9 to 45.9 at RPM 200 to 250 and then it dropped to 31.86 at 350 RPM. For cupferron it increased from 39.8 to 42.5 at RPM 200 to 250 and then it dropped to 22.9 at 350 RPM. For Thiosalicylic acid it increased from 19.8 to 21.3 at RPM 200 to 250 and then it dropped to 17.3 at 350 RPM, at a specified treat ratio (1:3) using complexant at the optimum strength The agitation speed of 250 rpm is found to be the most effective to recover Pd^{2+} from external. The drop in % of metal ion recovery beyond 300 RPM is attributed due to either de emulsification induced by higher shear of impeller or due to leakage from internal stripping phase.

3.2.6 Effect of Feed Concentration

As seen from Figure 7, we observe that as the feed strength is increased from 0.08 to 0.1437 mM, the % removal of Pd was found to decrease from 91.2 to 66, 82 to 65.1, 81.3 to 62, 77.6 to 60.2, 74.5 to 59.3, 68.2 to 55.4 using dithiazone, quinalizarin, 8-hydroxy quinoline, 1-nitroso-2-naphthol, cupferron and thiosalicylic acid respectively.

In all the cases the order of removal of Pd follows the sequence

dithiazone>quinalizarin>8-hydroxy quinoline>1-nitroso-2-naphthol>cupferron>thiosalicylic acid

It is observed that increase in feed phase concentration decreases the % solute recovered. This confirms that this technique is more effective to treat dilute streams.

4. Conclusions

The complexants chosen have Nitrogen, Oxygen and Sulphur for enabling complexation with Palladium. When the complexants are emulsified using span 80 as surfactant the emulsion formed could allow permeation of Palladium by providing good surface area /volume and hence the recovery of Pd was found to be effective. The order of uptake of Palladium by different complexants follow the sequence:

dithiazone>quinalizarin>8-hydroxy quinoline>1-nitroso-2-naphthol>cupferron>thiosalicylic acid

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