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EXTRACTION OF PLATINUM(IV) VIA LIQUID EMULSION MEMBRANE CONTAINING CYANEX-923 FROM BROMIDE MEDIA

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ABSTRACT

Transport behavior of Pt(IV) from bromide media through a liquid emulsion membrane has been studied using Cyanex-923 as a mobile carrier. Platinum(IV) was quantitatively extracted from external bromide (feed phase) media and stripped in perchloric acid as an internal (strip) phase. The important variables governing the permeation of Pt(IV) through LEM studied are membrane viscosity, concentration of mobile carrier, treatment ratio, speed of agitation, and internal (strip) phase concentration.

The optimum condition for extraction of Pt(IV) via liquid emulsion membrane are—*internal phase*: 4.0 mol dm^{-3} HClO_4 ; *organic phase*: 12% span-80, 7% 1-octanol, and 0.8% Cyanex-923 in xylene; *feed phase*: 10 ppm of Pt(IV) in 5.0 mol dm^{-3} HBr in presence of $0.005 \text{ mol dm}^{-3}$ SnCl_2 .

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Key Words: Liquid emulsion membrane; Platinum(IV); Cyanex-923; Hydrobromic acid; Extraction; Enrichment

INTRODUCTION

Platinum possessing the characteristic physical and chemical properties is widely used in electronic industries and as a catalyst in various reaction processes. Due to the scarcity and high cost, its separation and recovery from industrial resources and wastewater treatment is essential.^[1]

Numerous extractants of different types^[2–11] are known for conventional liquid–liquid extraction of Pt(IV) metal ion. Similarly, centrifugal partition chromatography^[12] and sulfur-containing extractants in solvent impregnated resins^[13] are used for the extraction of noble metals from chloride media. However, relatively little work has been conducted on extraction via liquid emulsion membrane because of slow extraction kinetics and difficulties in stripping^[14,15] of noble metals.

Li introduced the use of liquid emulsion membrane technique in 1968,^[16] which gives selective extraction of metal ions by permeation through membrane.

Membrane separation is achieved by hydrophobic liquid membrane, which separates two aqueous phases. The technique is energy-efficient, requires a small quantity of reagent, which permits the use of costly and highly selective carrier extractant than in solvent extraction. It can be used on continuous flow basis. It has high a distribution coefficient and its simultaneous extraction and stripping operation is very attractive to move metal ions from low concentration to a higher concentration. However, the pilot plant study showed that the membrane technique is cheaper than the solvent extraction.^[17,18]

Through the last few years, liquid emulsion membrane has emerged as a potential alternative to the conventional liquid–liquid extraction. Supported liquid membrane having TOA as a carrier study for extraction of Ir(IV)^[19] and Pt(IV),^[20] where 1-octanol was used to improve the stripping of Pt(IV). However, Pd(II) was separated from Pt(IV) from thiocyanide feed phase through a selective solid supported liquid membrane containing Cyanex-471X^[21] as a carrier. Liquid emulsion membrane having K⁺ dicyclohexane-18-crown-6 as a carrier^[22] was studied for the transport of Ag, Au, and Pd.

Recently, Cytec Industries Inc. in Canada have manufactured a series of organo phosphine compounds which are marketed under the trade name of “Cyanex” including trialkylphosphine oxide [Cyanex-923] having “O” as donor atom.

Nowadays, growing interest is observed in studying the effect of media on the extraction of platinum(IV). Thus, different media like bromide,^[23] iodide,^[24]



and thiocyanide^[25] were used instead of traditional chloride media. The Pd(II) extraction was found to be more favorable in HBr media than in HCl media with Di-*n*-octyl sulfide^[26] (DOS) as an extractant in cyclohexane, similarly extraction of Au(III)^[27] and Zn(II)^[28,29] in HBr media was found to be more effective than in the HCl media. In a novel separation scheme, chloro complexes are converted to the bromo form, since the bromo form complexes undergo aquation to a lesser extent and are more easily extractable than the chloro⁻ complexes. Thus the use of bromide^[30] has opened a new path for separation of platinum metals.

In our previous study, we have found that the distribution coefficient (*D*) of Pt(IV)^[31] and Ir(III)^[32] varied considerably by the use of HBr media, instead of HCl as an extracting media.

The present work deals with the study of the extraction of platinum(IV) from hydrobromic acid media using liquid emulsion membrane having Cyanex-923 as a carrier.

EXPERIMENTAL

Apparatus and Reagents

A G.B.C.-911A UV/Visible spectrophotometer was used for absorbance measurements. The extractant Cyanex-923 kindly supplied by Cytec Industries Inc.^[33,34] and surfactant sorbitan monooleate (Span-80) was obtained from Mohini Organic Pvt. Ltd. were used without further purification. The stock solution of Pt(IV) was prepared by dissolving K₂PtCl₆ in hydrobromic acid and standardized by known methods.^[35] The required concentration of this solution was prepared by further dilution. The SnCl₂ solution was prepared in 2.0 mol dm⁻³ HCl. All other chemicals used were of analytical grade.

PROCEDURE

Emulsion was prepared by dropwise addition of internal aqueous phase (stripped phase perchloric acid) to a stirred solution of organic phase containing, extractant (Cyanex-923) in xylene, surfactant (Span-80), and 7% 1-octanol at an O/A phase ratio of 1 for 30 min with six blade turbine impeller in 2 dm³ glass vessel at 3000 rpm stirring speed. The stable milky emulsion was obtained. Emulsion was dispersed in the feed phase of platinum metal ion in bromide media, which was stirred in 500 dm³ glass vessel at 500 rpm for 10 min.

The samples were withdrawn at different time intervals and the aqueous phase was analyzed. At the end of the experiment, the emulsion was separated



from the aqueous phase by a separating funnel and break at 80°C, where stripped phase was analyzed spectrophotometrically.

All the experiments were carried out at 25°C. The initial concentration of Pt(IV) was maintained at 10 mg dm^{-3} throughout the experiments (unless otherwise stated) and the metal content in the equilibrated aqueous phase was determined by the stannous chloride^[36] method at 403.0 nm. The amount of metal present in the organic phase was calculated by a mass balance. The distribution coefficient D was calculated as the ratio of the equilibrium concentration of Pt(IV) in organic phase to the aqueous phase.

RESULTS AND DISCUSSION

Influence of Hydrobromic Acid Concentration

The bromide complexes are generally weaker or less stable^[28] than chloride complexes in aqueous solution and they can be extracted easily, probably due to the large molecular size or large aquophobic^[29] tendency in addition to less electronegativity. Therefore the bromide media was employed for the extraction of Pt(IV) rather than the traditional chloride media.

The extraction of Pt(IV) with Cyanex-923 in xylene was found to have a maximum of 56.3% in the range $4.5\text{--}5.0 \text{ mol dm}^{-3}$ HBr. The optimum conditions of LEM experiment are given in Table 1. At lower and higher concentrations of the feed phase, the increase in emulsion swelling may be due to the osmotic pressure difference between the internal and external phase. During stripping, the third phase formation was observed due to higher concentrations of the mineral acids and was avoided by the addition of 7% of 1-octanol.^[19,20] The

Table 1. Optimum Condition for Extraction of Pt(IV) with Cyanex-923 from Bromide Media

1. Internal (strip) phase	4.0 mol dm^{-3} HClO_4
2. Organic phase	12% (v/v) Span-80, 7% (v/v) 1-octanol and 0.8% (v/v) Cyanex-923 in xylene
3. External (feed) phase	10 ppm Pt(IV) in 5.0 mol dm^{-3} HBr in the presence of $0.005 \text{ mol dm}^{-3}$ SnCl_2
4. Treat ratio	1:5 (emulsion to feed phase)
5. Speed of stirring (during emulsion preparation)	3000 rpm
6. Speed of stirring	500 rpm
7. Time	7.0 min

quantitative extraction of Pt(IV) was found possible only in the presence of SnCl_2 (Fig. 1).

Influence of Stannous Chloride

The effective influence of the liquid membrane system was studied for Pt(IV) extraction from 5.0 mol dm^{-3} of HBr by varying the concentration of SnCl_2 from 1.0×10^{-4} to $1.0 \times 10^{-3} \text{ mol dm}^{-3}$ with 12 span-80, 7% 1-octanol, and 0.8% Cyanex-923 in xylene and 4.0 mol dm^{-3} HClO_4 as an internal (strip) phase. The quantitative extraction was observed in the presence of $0.005 \text{ mol dm}^{-3}$ of SnCl_2 (Fig. 1).

Influence of Membrane Viscosity

Membrane viscosity plays an important role in controlling permeation of metal through liquid membrane and stability of membrane.^[37,38] The nonionic span-80 surfactant having a HLB value of 4.3, was used since it was suitable for the preparation of W/O emulsion. The viscosity of membrane depends upon the concentration of surfactant.

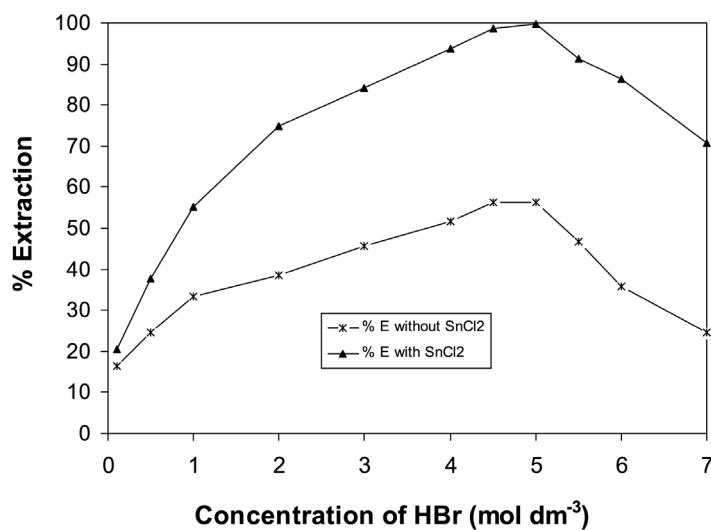


Figure 1. Influence of external acid concentration (feed phase) on extraction of Pt(IV) with and without SnCl_2 .

Effect of membrane viscosity was studied by preparing three different membranes having 8, 12, and 15% span-80 (Fig. 2). In the first type (8% span-80) of membrane, the initial extraction rate is significantly faster than the others due to its low viscosity, but with the longer contact time the emulsion breaks and leakage of internal phase was observed. However, in the third type of membrane, (15% span-80) the mass transfer seems to resist due to its high viscosity. So optimum extraction and stability was obtained by using 12% span-80.

Effect of Equilibrium Time

The influence of equilibration time was studied for 1–10 min. The results obtained showed that the extraction equilibrium of Pt(IV) was achieved within 7 min, however, with further increase in equilibrium period, an abrupt decrease in extraction was observed, which causes a decrease in the stripping, that may be due to emulsion instability (Table 2).

Influence of Reagent Concentration

Concentration of mobile carrier (extractant) was studied for 0.5, 0.8, and 1.2% of Cyanex-923 (Table 3). The extraction was found to increase with the increase in the reagent concentration, but its stripping (enrichment) was found to

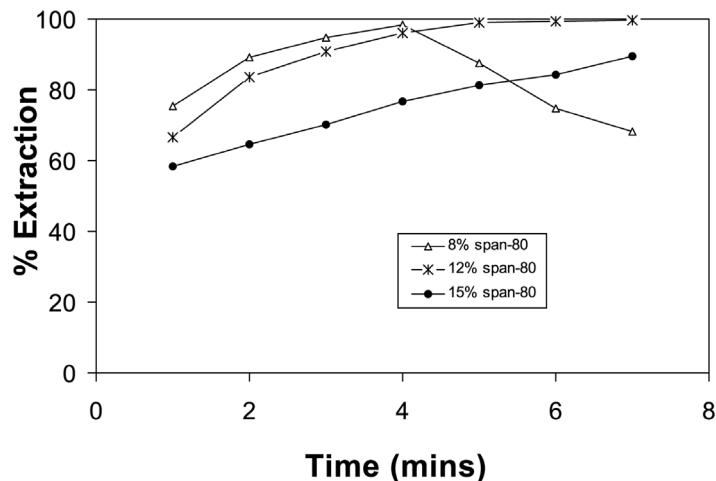


Figure 2. Influence of concentration of surfactant on extraction of Pt(IV).

**Table 2.** Effect of Time on Extraction of Pt(IV)

Contact Time (min)	External (Feed) Phase (mg dm ⁻³)	Percentage Extraction	Internal (Stripped) Phase (mg dm ⁻³)	Enrichment
0	10.0	—	—	—
1	3.33	66.7	17.8	2.67
2	1.64	83.6	19.7	2.36
3	0.91	90.9	22.3	2.45
4	0.38	96.2	26.9	2.80
5	0.11	98.9	29.3	2.96
6	0.07	99.3	32.3	3.25
7	0.02	99.8	38.6	3.87
8	0.02	99.8	38.6	3.87
9	0.13	—	—	—
10	0.42	—	—	—

decrease, so minimum reagent concentration was recommended to obtain maximum enrichment.

Influence of Treatment Ratio

The treat ratio (TR) is the ratio of emulsion phase volume to the external feed phase volume (V_e/V_f) which shows (Fig. 3) the mass transfer and enrichment dependence upon the TR. The experiments were performed for the TR of 1:2, 1:5, and 1:10. However, lower TR is always favorable, because with less emulsion, maximum enrichment can be achieved.

Transport of aqueous phase from outer phase (external phase) to inner phase (internal phase) increases as the treatment ratio increases, which may be due to the increase in osmotic pressure. Thus, extraction in TR 1:5 is higher than 1:2. However, in TR 1:10, initially the extraction is higher due to high transport of aqueous phase, but after that emulsion becomes unstable and breaks extraction decreases.

Influence of Speed of Stirring

During the preparation of emulsion, the stability of emulsion depends upon the speed of agitation. As the speed increases, there is an increase in the stability and thus the viscosity, so extraction may decrease, but the effect was negligible as compared to other parameters. Similarly, breaking of emulsion becomes difficult.



Table 3. Influence of Reagent Concentration on Extraction of Pt(IV)

Time (min)	0.5% Cyanex-923		0.8% Cyanex-923		1.2% Cyanex-923	
	External Phase (mg dm ⁻³)	Internal Phase (mg dm ⁻³)	External Phase (mg dm ⁻³)	Internal Phase (mg dm ⁻³)	External Phase (mg dm ⁻³)	Internal phase (mg dm ⁻³)
0.0	10.0	—	10.0	—	10.0	—
1.0	5.5	14.78	3.33	17.82	2.45	5.90
2.0	3.10	21.53	1.64	19.75	0.83	14.75
3.0	1.85	25.75	0.91	22.30	0.18	22.62
4.0	0.83	28.53	0.38	26.90	0.02	26.96
5.0	0.60	30.50	0.11	29.35		
6.0	0.48	32.5	0.07	32.35		
7.0	0.45	32.5	0.02	38.70		

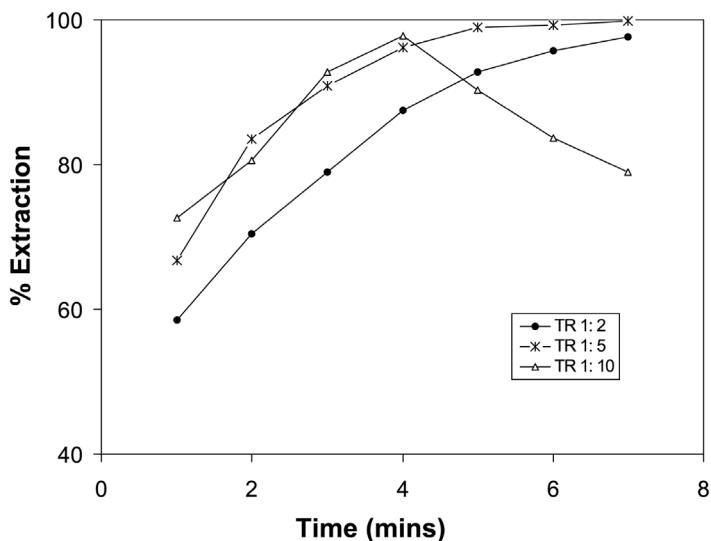


Figure 3. Influence of TR on extraction of Pt(IV).

The speed of stirring during the extraction process may affect the extraction, so it was studied at different speeds of 100, 300, 500, 700, and 1000 rpm. It was found that as the stirring speed was increased, the extraction was also increased up to 500 rpm, above that, the shear and transport of water^[39] was increased, causing a breakage of emulsion and thus extraction was decreased (Fig. 4).

Influence of Internal Acid Concentration

The acid concentration of internal phase was important for enrichment of metal. According to liquid–liquid extraction study, the perchloric acid was the best stripping agent for Pt(IV).^[7] Thus study was performed in the range 0.1–7.0 mol dm⁻³ HClO₄. Stripping (enrichment factor) was increased as acid concentration increased and maximum at 4.0 mol dm⁻³, above which it decreases because of swelling and breaking of the membrane (Fig. 5).

Influence of Diluents

The effect of the diluents was studied with extraction of Pt(IV) from 5.0 mol dm⁻³ HBr in the presence of 0.005 mol dm⁻³ SnCl₂ with 0.8% Cyanex-

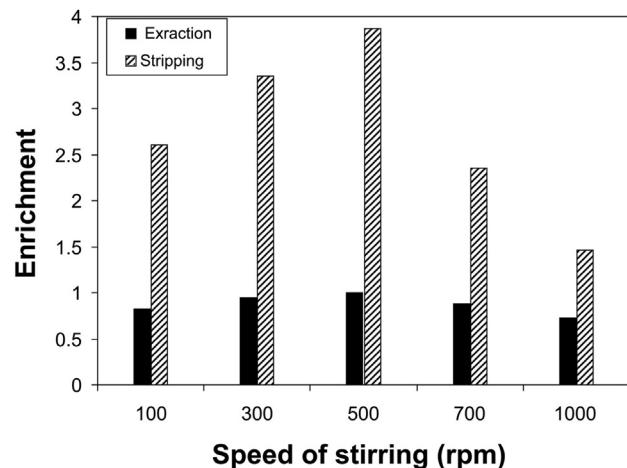


Figure 4. Influence of speed of stirring on extraction of Pt(IV).

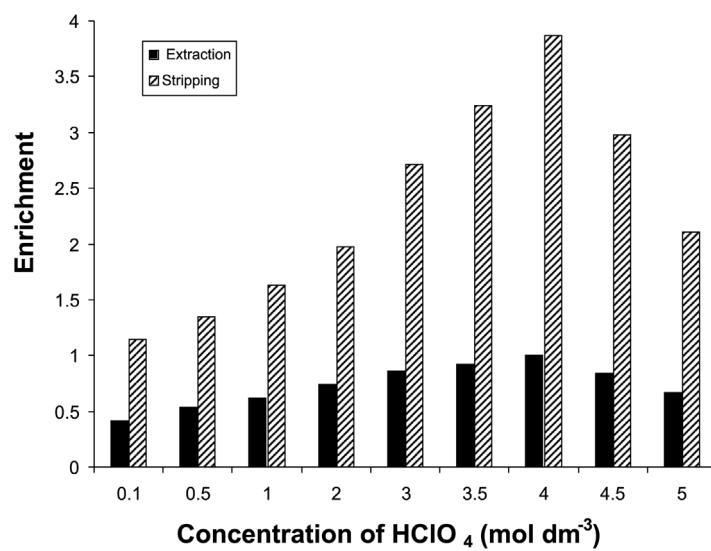


Figure 5. Influence of internal (strip) phase concentration on extraction of Pt(IV).



Table 4. Influence of Diluents on Extraction of Pt(IV) from 5.0 mol dm^{-3} HBr in Presence of $0.005 \text{ mol dm}^{-3}$ SnCl_2 with 0.8% Cyanex-923 in Liquid–Liquid Extraction

Diluents	% Extraction ^a	% RSD
Toluene	100.0	0.00
Xylene	99.47	0.23
<i>n</i> -hexane	93.70	0.41
Cyclohexane	99.23	0.36
Chloroform	92.80	0.22
Carbon tetrachloride	93.90	0.21

^a Means of triplicate values.

923 by convention liquid–liquid extraction, however, the quantitative extraction was observed in toluene and xylene while *n*-hexane, cyclohexane, carbon tetrachloride, and chloroform do not favor quantitative extraction (Table 4). Thus LEM experiments were performed by using xylene as a diluent.

CONCLUSION

Liquid emulsion membrane of Cyanex 923 is successfully applied for the extraction of Pt(IV) from the bromide media. The optimum conditions for maximum recovery of Pt(IV) from its dilute solution were found out by studying the different parameters. The mobile carrier Cyanex-923 was effectively used for the extraction of Pt(IV) through liquid emulsion membrane from bromide media where SnCl_2 facilitate the quantitative extraction of Pt(IV), while HClO_4 was found as the most effective stripping agent.

Use of membrane facilitates the recovery of Pt(IV) in high concentration as compared to the solvent extraction. However, the concentration of Cyanex-923 used was less as compared to the other reagent like TOA.

NOMENCLATURE

[]	concentration (mol dm^{-3})
<i>D</i>	distribution ratio
%E	percentage extraction
%S	percentage stripping
aq	aqueous phase
org	organic phase



TR	treat ratio (emulsion phase/feed phase)
En	enrichment (stripped metal conc./extracted metal conc.)

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