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Studies on the Recovery of Uranium from Phosphoric Acid Medium Using Synergistic Mixture of 2-Ethyl Hexyl Hydrogen 2-Ethyl Hexyl Phosphonate and Octyl(phenyl)-N,N-diisobutyl Carbamoyl Methyl Phosphine Oxide

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Studies on the Recovery of Uranium from Phosphoric Acid Medium Using Synergistic Mixture of 2-Ethyl Hexyl Hydrogen 2-Ethyl Hexyl Phosphonate and Octyl(phenyl)-N, N-diisobutyl Carbamoyl Methyl Phosphine Oxide

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Abstract: This paper describes the extraction of uranium from aqueous phosphoric acid medium using 2-ethyl hexyl hydrogen 2-ethyl hexyl phosphonate (PC88A) and octyl (phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide (CMPO) individually as well as their synergistic mixture in different diluents. The extraction parameters such as variation in concentration of either of the extractants, concentration of H_3PO_4 and uranium in the aqueous phase are investigated to optimize the extraction conditions. Results indicate that the synergistic mixture, 0.9 M PC88A + 0.1 M CMPO in xylene, can be used for the extraction of uranium from the phosphoric acid medium. The loaded uranium from the synergistic organic phase can be stripped using 0.5 M solution of $(NH_4)_2CO_3$. This synergistic mixture is used to recover uranium from a typical wet process phosphoric acid sample and the recovery is found to be better than 90%.

Keywords: Solvent extraction, synergism, uranium, wet process phosphoric acid

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INTRODUCTION

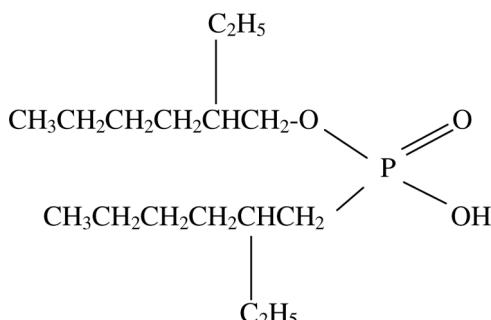
Uranium plays an important role in the generation of nuclear power but has limited primary resources. To sustain the nuclear power its secondary resources are being explored worldwide. Among the secondary resources, natural phosphates are found to contain several tens to hundreds parts per million of uranium depending upon the origin of the phosphate rocks (1-3). Geochemical data show that sedimentary phosphate deposits of marine origin contain higher uranium than those of igneous origin (4). Besides geochemical origin, the geographical locations also influence the uranium content in phosphate rocks. During the sulphuric acid digestion of phosphate rocks for the production of wet process phosphoric acid (WPA), most of the uranium (<90%) dissolves in phosphoric acid leaving a minor portion (<10%) in the solid residue (5). Among the various separation techniques solvent extraction is the most widely used technique and many solvent systems have been used for the recovery of uranium from WPA (6-8). Among these systems, di-2-ethylhexyl phosphoric acid (D2EHPA)-tri-n-octyl phosphine oxide (TOPO) is the most popular and proven system for uranium recovery from the phosphoric acid medium (9,10). In an attempt to select an alternate solvent system for uranium recovery from the phosphoric acid medium, the results using the D2EHPA-tri-n-butyl phosphate (TBP) system are reported to be very encouraging (11,12). Among the other extractants, 2-ethylhexyl hydrogen 2-ethylhexyl phosphonate (PC88A) is gaining importance in the separation of rare earths (13) and has many properties similar to D2EHPA. Octyl(phenyl)-N, N-diisobutylcarbamoyl methyl phosphine oxide (CMPO), a neutral organophosphorous reagent has been used for the extraction of actinides from high level liquid waste (HLLW) generated in nuclear industries (14). The extraction of uranium from the phosphoric acid medium using these extractants individually as well as with their synergistic mixture is the subject of the present investigation which has not been studied so far. Based on the results carried out under different experimental conditions, a flow Scheme has been proposed and used to recover uranium from a typical WPA samples.

EXPERIMENTAL

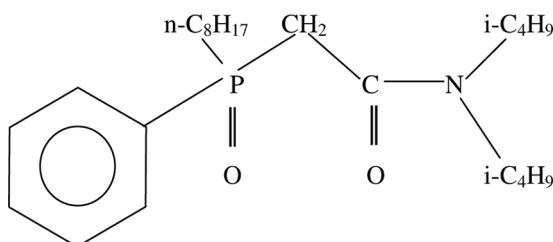
Reagents

Commercial grade 2-ethylhexyl hydrogen 2-ethylhexyl phosphonate (PC88A) (purity>95%) from Daihachi Chemical Industry Co. Ltd. Japan was used as supplied. Indigenously synthesized and purified

octyl-(phenyl)-N,N-diisobutylcarbamoyl methylphosphine oxide (CMPO) (15) by the Bio-Organic Division of this center was used. Structural formulae of these reagents are presented below:



2-ethyl hexyl hydrogen 2-ethyl hexyl phosphonate (PC88A)



Octyl(phenyl)-N,N-diisobutylcarbamoyl methylphosphine oxide (CMPO)

Stock solution of uranium (1 g/L) in phosphoric acid medium was prepared from uranyl nitrate hexa hydrate using the method reported earlier (16). All other reagents used were of analytical grade.

²³³U radiotracer was separated and purified from THOREX streams using the method described elsewhere (17) and converted into phosphoric acid medium. To study the extraction behavior of uranium, suitable aliquots from this radiotracer were spiked in the feed solution during extraction studies.

The wet process phosphoric acid sample obtained from the Uranium Extraction Division (URED) of this center was used for uranium recovery. The H_3PO_4 concentration of the sample was $\sim 5\text{ M}$.

Analysis

Uranium-233 in the aqueous/organic phase was assayed by α -scintillation counter using Ag activated ZnS detector. The efficiency of the detector as

standardized using ^{239}Pu was $30 \pm 2\%$. Uranium concentrations in WPA sample as well as in other aqueous streams, generated during batch experiments, were analyzed by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) after diluting them appropriately.

All the experiments were carried out at room temperature i.e. $\sim 25^\circ\text{C}$ in duplicate and mean values are reported. The mass balance for all the batch experiments was found to be generally within $\pm 5\%$.

Batch Experiment

Suitable aliquots from the stock solution of uranium were used to prepare feed solutions during the batch extraction studies. In these studies, a fixed volume of the aqueous phase (generally, 2 mL to 20 mL) at a desired concentration of H_3PO_4 containing known concentration of uranium spiked with tracer activity of ^{233}U was contacted with equal volume of the extractant.

To optimize the equilibration time the batch extraction was carried out separately for different interval of time varying upto 1 h using the desired organic phase at an organic to aqueous phase ratio of 1:1. The feed solutions used in these studies were spiked with tracer activity of ^{233}U that contained 300 mg/L uranium at 0.5 M H_3PO_4 . After contact the phases were separated and assayed for alpha activity.

Preliminary experiments were carried out using a different concentration of PC88A and CMPO separately. The concentration of PC88A in these extractions was varied from 0.03–0.9 M and that of CMPO from 0.01–0.5 M in xylene. The aqueous phase concentration of uranium in these cases were maintained at 300 mg/L while the concentration of phosphoric acid was varied up to 4.5 M.

Under similar feed conditions the extraction of uranium was also studied using the solvent mixture of 0.15 M PC88A + 0.1 M CMPO in xylene. In a separate set of experiments the extraction behavior of uranium was studied using the same concentration of PC88A and CMPO in commonly available diluents viz. kerosene, xylene, toluene, benzene, hexane, chloroform, and carbon tetrachloride.

The extraction of uranium using the solvent mixture of PC88A and CMPO was also studied from an aqueous phase containing different concentration of uranium in the range 50–500 mg/L at 0.5 M H_3PO_4 medium.

The selection of a reagent for uranium stripping from the organic phase was carried out using commonly used stripping agents viz. HNO_3 (8 M), HCl (12 M), H_2SO_4 (10 M), $(\text{NH}_4)_2\text{CO}_3$ (2 M), citric acid (1 M), urea (3 M), sodium salt of EDTA (0.01 M) and water. After

assaying the uranium in both the phases by radiometry, the extraction/ stripping was expressed in percentage or distribution ratio (D) using the standard method. Synergistic coefficients were also calculated using D_U values as follows:

$$\text{Synergistic coefficient} = \text{Log} [(D_{1,2})/(D_1 + D_2)] \quad (1)$$

Where $D_{1,2}$ is the distribution ratio of uranium using the synergistic mixture during extraction and D_1 , D_2 are the distribution ratios of uranium using the individual extractant.

The extraction of uranium from the WPA sample was carried out under different experimental conditions. In one case extraction was carried out as such without feed conditioning whereas in other experiments the acidity of the aqueous feed was reduced to 0.5 M with respect to the wet process phosphoric acid either by partial neutralization with NaOH or by diluting it with water.

RESULTS AND DISCUSSION

Figure 1 gives the extraction of uranium by PC88A and CMPO as a function of time. These results indicate that a contact time of 20 minutes is enough to reach the equilibrium for both the systems. Based on these results a contact time of about 30 minutes was maintained in all the extraction experiments so as to ensure the equilibrium.

Table 1 gives the extraction behavior of uranium from the phosphoric acid medium using varying concentrations of these extractants individually. Results show low extraction of uranium by PC88A ($D_U \sim 10^{-1}$) which increases with increasing concentration of the extractant. The extraction of uranium by CMPO is observed to be lower (by an order of magnitude) under experimental conditions and increase in the D_U values is not remarkable with its increasing concentration especially above 0.1 M of CMPO.

The extraction behavior of uranium as carried out using varying concentration of phosphoric acid in the range of 0.5 to 4.5 M using 0.15 M PC88A in xylene and 0.1 M CMPO in xylene and results are given in Table 2.

From the results it is seen that as the concentration of H_3PO_4 in the aqueous phase increases the D_U values decrease in the case of PC88A which explains its cation exchange behavior. Low D_U values from the entire phosphoric acid range studied suggests poor extraction of uranium by CMPO. The extraction of uranium by an arbitrarily chosen solvent mixture (0.15 M PC88A and 0.1 M CMPO in xylene) when used, a

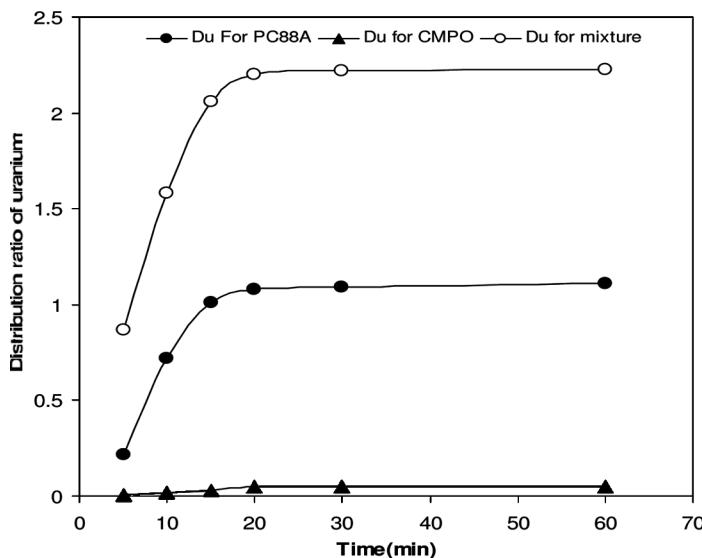


Figure 1. Distribution ratio of uranium vs time of contacts. [Feed: conc. of uranium: 300 mg/L, Conc. of H_3PO_4 : 0.5 M, Extractants: 0.15 M PC88A, 0.1 M CMPO and their mixture in xylene, Phase ratio: 1:1 in each contact, Volume of each phase: 2 mL].

Table 1. Extraction of uranium using varying concentration of PC88A and CMPO

Distribution ratio of uranium			
PC88A in xylene		CMPO in xylene	
[PC88A] (M)	D _U	[CMPO] (M)	D _U
0.03	0.06	0.01	0.018
0.09	0.16	0.02	0.025
0.15	0.47	0.05	0.031
0.30	0.57	0.10	0.042
0.45	0.72	0.20	0.044
0.60	0.86	0.30	0.044
0.90	1.09	0.50	0.046

[Feed: Conc. of uranium = 300 mg/L, Conc. of H_3PO_4 = 1 M, Contact time: 30 min., Phase ratio: 1:1, Volume of each phase: 2 mL].

Table 2. Extraction of uranium from varying concentration of H_3PO_4 medium using PC88A, CMPO and their mixture

[H_3PO_4 in feed (M)]	Extraction using 0.15 M PC88A in xylene (D _U)	Extraction using 0.1 M CMPO in xylene (D _U)	Extraction using a mixture of 0.15 M PC88A and 0.1 M CMPO in xylene (D _U)	Synergistic coefficient (S.C.)
0.50	1.12	0.051	2.230	0.279
0.75	0.86	0.045	1.170	0.112
1.0	0.47	0.043	0.678	0.121
1.5	0.29	0.037	0.426	0.115
2.0	0.11	0.031	0.224	0.201
3.5	0.02	0.025	0.160	0.514
4.5	0.01	0.022	0.060	0.301

[Feed: Conc. of uranium = 300 mg/L, Conc. of H_3PO_4 = 0.5 M, to 4.5 M Contact time: 30 min., Phase ratio: 1:1, Volume of each phase: 2 mL].

remarkable improvement in the D_U values was observed in all the cases. This synergistic enhancement may be due to the participation of the neutral donor in the extraction of metal ion by replacing the water of hydration and making the complex more hydrophobic. Such mechanism has been reported in literature in case of β -diketone with neutral extractant (18) and in solvent system using D2EHPA and TBP in the extraction of uranium from H_3PO_4 medium (11,12). The extraction time required for uranium extraction using a synergistic combination as studied from 1.0 M H_3PO_4 medium is also shown in Fig. 1, which clearly indicates that 30 minutes contact time is more than enough to reach the final equilibration.

One of the parameters that influence the extraction is the nature of diluents used during extraction. The results from an experiment where common diluents were used for studying the extraction behavior of uranium from varying concentration of phosphoric acid using an arbitrarily chosen solvent combination as extractant are given in Table 3. The diluents used in the present study belong to diverse classes like aromatic, alicyclic and aliphatic. The synergistic enhancement does not follow classwise behavior. Diluent parameters such as dipole moment, dielectric constants etc. are also not useful in describing the observed extraction behavior. Xylene and kerosene are found to have higher extraction of uranium ($D_U \sim 2$). The enhanced D in the case of xylene can be explained based on the increasing branching on the benzene which enhances the solubility. Kerosene contains a mixture of paraffins with branched chain

Table 3. Effect of diluents on the extraction of uranium from phosphoric acid medium using a synergistic combination of PC88A and CMPO

[H ₃ PO ₄] (M)	Distribution ratio of uranium (D _U)						
	Kerosene	Xylene	Toluene	Benzene	Hexane	CHCl ₃	CCl ₄
0.50	2.08	2.23	1.48	1.07	1.27	1.65	1.12
0.75	0.98	1.11	0.92	0.75	0.86	0.95	0.91
1.0	0.62	0.68	0.58	0.53	0.51	0.51	0.48
1.5	0.38	0.41	0.34	0.19	0.18	0.22	0.21
2.0	0.20	0.24	0.17	0.16	0.17	0.19	0.16
3.5	0.16	0.16	0.14	0.11	0.10	0.15	0.13
4.5	0.04	0.06	0.03	0.02	0.07	0.08	0.02

[Feed: Conc. of uranium = 300 mg/L, Conc. of H₃PO₄ = 0.5 M, to 4.5 M Extractant mixture: 0.15 M PC88A + 0.1 M CMPO in various diluents. Contact time: 30 min., Phase ratio: 1:1, Volume of each phase: 2 mL].

hence it may be following the similar behavior. Since kerosene is a mixture of different carbon chain compounds, its extraction behavior may not be useful for understanding the extraction mechanism. Hence, further studies were carried out with xylene alone which is also being used on a plant scale (12). Other diluents are not industrially used and hence were not considered further. The decreasing trend in D_U values with increasing concentration of H₃PO₄ in aqueous phase using all the diluents is also similar.

In an attempt to optimize the solvent composition the extraction of uranium was studied from 1.0 M phosphoric acid using varying concentration of either of the extractant. The results are given in Table 4.

The distribution ratio (D_U) values obtained indicate synergism when the solvent combination of 0.15 M PC88A contained CMPO above 0.05 M. Below 0.05 M of CMPO the extraction may be controlled by PC88A only. The similar increasing trend in the distribution ratio of uranium (D_U) is observed when the concentration of CMPO is kept 0.1 M in xylene and the concentration of PC88A is varied upto 0.90 M in xylene. These results show that 0.90 M PC88A and 0.1 M CMPO in xylene is the most suitable synergistic combination for uranium extraction at a concentration level that is generally encountered in secondary resources like wet process phosphoric acid.

Figure 2 depicts the extraction of uranium as carried out from its different concentration varying from 50–500 mg/L at 0.5 M H₃PO₄ medium using the synergistic mixture of PC88A and CMPO. This is the concentration range that can be encountered in secondary resources

Table 4. Effect of variation in concentration of either of the extractants on extraction of uranium

Composition of extractants mixture		Extraction by mixture of extractants (D_U)	Extraction of uranium due to synergism $D_{synergism}$
[PC88A] in xylene (M)	[CMPO] in xylene (M)		
0.15	0.01	0.231	-0.257
-do-	0.02	0.375	-0.120
-do-	0.05	0.515	0.014
-do-	0.10	0.672	0.163
-do-	0.20	0.678	0.166
-do-	0.30	0.684	0.172
-do-	0.50	0.687	0.175
0.03	0.10	0.160	0.058
0.09	-do-	0.420	0.158
0.15	-do-	0.674	0.162
0.30	-do-	1.260	0.648
0.45	-do-	1.540	0.778
0.60	-do-	1.790	0.888
0.90	-do-	2.200	1.070

[Feed: Conc. of uranium = 300 mg/L, Conc. of H_3PO_4 = 1 M, Contact time: 30 min., Phase ratio: 1:1, Volume of each phase: 2 mL].

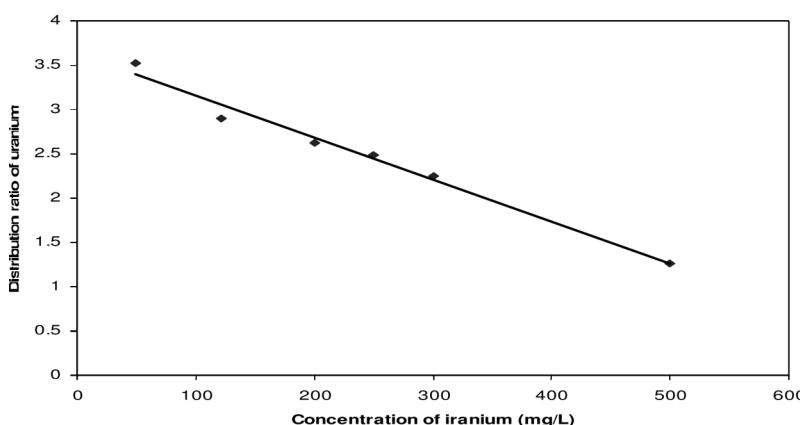
**Figure 2.** Distribution ratio of uranium by a mixture of 0.15 M PC88A and 0.1 M CMPO in xylene vs concentration of uranium (mg/L) in 0.5 M H_3PO_4 [Conc. of H_3PO_4 in feed: 0.5 M H_3PO_4 , Extractant: 0.90 M PC88A + 0.1 M CMPO in xylene, Phase ratio: 1:1, Contact time: 30 min., Volume of each phase: 2 mL].

Table 5. Extraction of uranium from phosphoric acid medium using synergistic mixture of PC88A and CMPO

Number of contacts	Conc. of uranium in organic phase (mg/L)	Cumulative extraction (%)
I	219.27	73.09
II	61.54	93.06
III	18.13	99.65

[Feed: Conc. of uranium = 300 mg/L, Conc. of H_3PO_4 = 0.5 M H_3PO_4 , Extractants: 0.90 M PC88A + 0.1 M CMPO in xylene, Contact time: 30 min., Phase ratio: 1:1 in each contact, Volume of each phase: 10 mL.

like WPA of different origin. Results show a decrease in the D_U value with increase in the concentration of uranium in the aqueous phase at a particular concentration of H_3PO_4 . This decrease in D_U can be

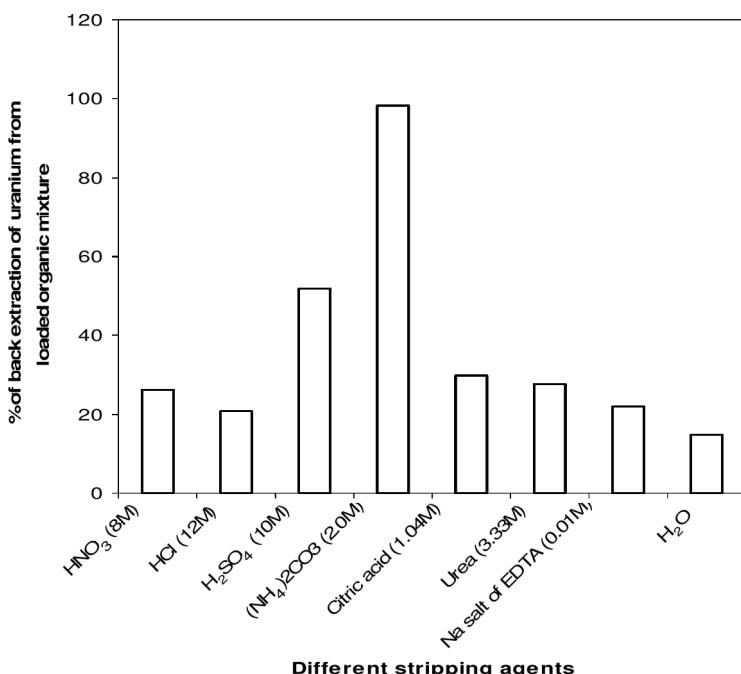


Figure 3. Back extraction of uranium from a synergistic system of 0.15 M PC88A + 0.1 M CMPO in xylene by different stripping agents. [Conc. of uranium in composite organic phase: 100 mg/L, Phase ratio: 1:1 for each contact, Volume of each phase: 2 mL, Contact time: 30 min.].

attributed to the decreased free solvent concentration at equilibrium during extraction.

Table 5 gives the results on the extraction of uranium using the optimized combination of the solvent i.e. 0.90 M PC88A and 0.1 M CMPO in xylene. The concentration of uranium used in feed is 300 mg/L at 0.50 M H_3PO_4 as this is the highest concentration reported in secondary resources of different origin. Extraction of uranium is found to be higher from the lower concentration of phosphoric acid (0.50 M). Results show >99% extraction of uranium in three successive contacts with fresh synergistic combination.

The uranium loaded composite organic phase was employed for stripping experiments using commonly used strippants. The results are shown in Fig. 3. Ammonium carbonate is found to be the most effective strippant among all which could strip uranium >99% in a single batch contact. H_2SO_4 though could strip ~50% uranium in a single contact is not suitable for industrial use. Other stripping agents used were less effective (<30% stripping in single contact) for uranium stripping. The concentration of ammonium carbonate to be used for uranium stripping from the composite loaded organic phase was optimized through an experiment carried out separately using varying concentration of

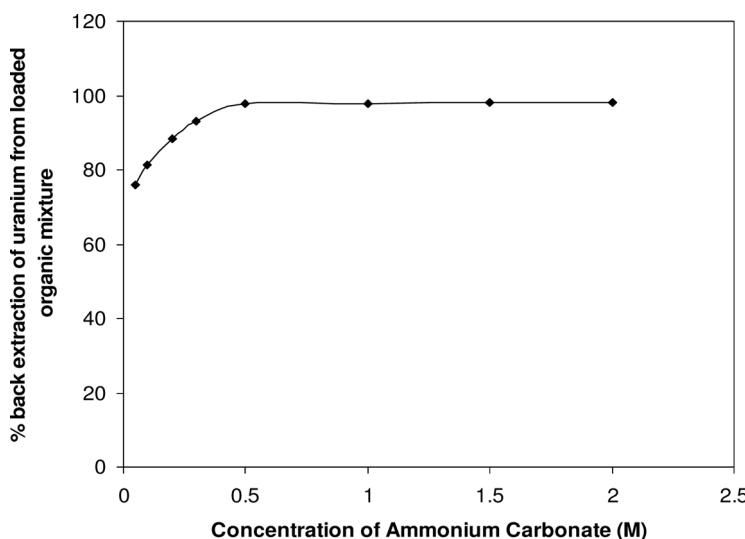


Figure 4. Optimizations of the conc. of $(NH_4)_2CO_3$ as strippant for uranium from the loaded synergistic organic phase. [Conc. of uranium in composite organic phase: 100 mg/L, Phase ratio: 1:1 for each contact, Volume of each phase: 2 mL, Contact time: 30 min. each.].

Table 6. Extraction of uranium from WPA using synergistic mixture of PC88A and CMPO

Details of the feed used	[H ₃ PO ₄] (M)	Percentage extraction In multiple contacts			Cumulative Extraction (%)	Stripping of uranium from loaded organic phase (%)
		I	II	III		
WPA sample	5.12	8.80	9.23	10.64	26.40	98.32
WPA neutralized using NaOH	0.50	52.71	54.56	59.34	91.40	99.56
WPA sample diluted with water	0.050	42.93	44.67	49.82	84.16	99.63

Cone. of uranium in feed: 300 mg/L, Extractants: 0.90 M PC88A + 0.1 M CMPO in xylene, Contact time: 30 min. each, Phase ratio: 1:1 in each contact, Volume of each phase: 2 mL].

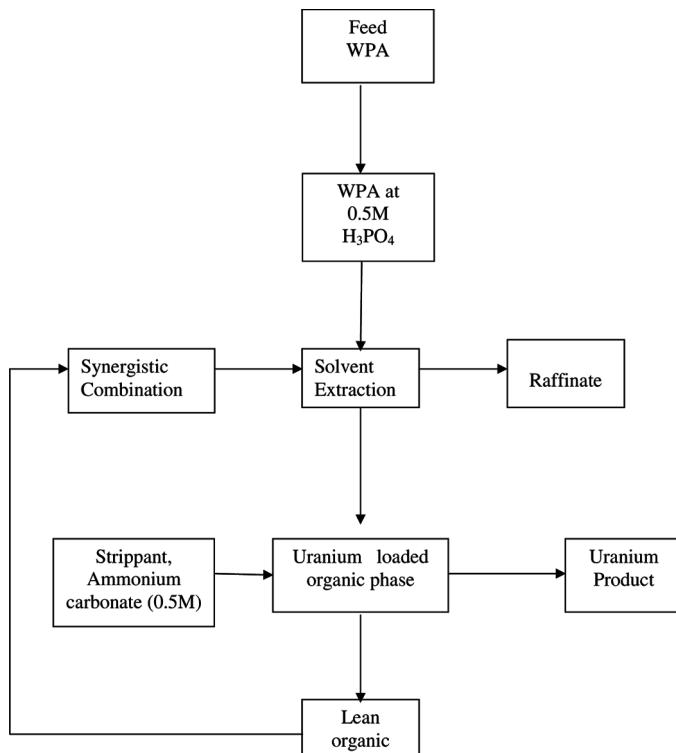


Figure 5. Proposed Flow scheme for recovery of uranium from Wet Process Phosphoric Acid.

$(\text{NH}_4)_2\text{CO}_3$ for uranium stripping. The results are shown in Fig. 4 indicating that 0.5 M $(\text{NH}_4)_2\text{CO}_3$ is enough to strip uranium most effectively.

Encouraged by the above extraction results the optimized synergistic mixture was used to recover uranium from a typical WPA sample. The concentration of uranium in the sample as analysed by ICP-AES was 182 mg/L.

Table 6 gives the results of uranium extraction and stripping as carried out under different experimental conditions. These results show that only $\sim 26\%$ uranium is extracted in three batch contacts when the sample is used as feed without conditioning. More than 90% uranium extraction is observed in three contacts when the sample is partially neutralized to 0.50 M with respect to H_3PO_4 using 10 M NaOH. When the sample is diluted to reduce the concentration of H_3PO_4 to 0.50 M prior to the extraction step, $\sim 84\%$ uranium could be extracted in three contacts. Small white turbidity was observed during the partial neutralization step

which disappeared during mixing. The uranium loaded composite organic phase from each of the extraction experiments when used for uranium stripping separately using 0.5 M ammonium carbonate solution, >98% stripping of uranium, that got loaded in the organic phase, was observed in single contact. Based on the above studies, a flow scheme (Fig. 5) is proposed for the recovery of uranium from WPA. Studies on the validation of the proposed flow-sheet using laboratory scale mixer-settlers are in progress and will be reported separately.

CONCLUSION

The studies carried out in this paper clearly indicate that a synergistic mixture of 0.90 M PC88A and 0.1 M CMPO in xylene can be employed for the extraction of uranium from its secondary resources like WPA. The extraction of uranium is found to be better when the concentration of H_3PO_4 in the aqueous feed is low (0.50 M of H_3PO_4). The uranium from the loaded organic phase can be stripped quantitatively using 0.50 M $(NH_4)_2CO_3$. The proposed scheme can be used for the recovery of uranium from WPA.

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