An Extractive Studies on Behaviour of Lead (II) From Acetate Media by Cyanex 301: A Green Approach

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Abstract - Herein we have developed a reliable solvent extraction method of lead(II) with 5×10^{-3} M cyanex 301 in kerosene from 1 M ammonium acetate solution. Physicochemical parameters like impact of ammonium acetate concentration, cyanex 301 concentration, equilibrium time, diluents study, and loading capacity were optimized for the quantitative extraction of lead(II). Lead(II) was stripped with 2 M HNO_3 from the organic phase and determined spectrophotometrically with PAR. To extend the applicability and utility of this method, diverse ion study, multicomponent mixture separation, extraction of lead(II) from galena ore, and solder alloy were also investigated.

Keywords: Lead(II), cyanex 301, extraction, acetate media, kerosene

INTRODUCTION

Lead is a toxic element for human beings and it is available everywhere in nature. When it is absorbed, it is engaged with extensive half-life and harms on kidney malfunctions, brain damage, anemia, and heart implant devices.[1-4] Lead is employed for cable sheathing, rechargeable batteries, alloys, stabilizer, radiation sheeting, ammunition and shot, pigments, pesticide production, rolled and extruded products.[5] Because of the adverse impact of lead on the living organisms and environment, enormous application of lead, it is necessary to determine,[6-8] removal[9-11] and recovery [12-14] of this heavy toxic element from contaminated environmental matrices and industrial effluents makes significance in different industrial and environmental studies.

It is significant to separate lead from various sources by environmentally friendly approaches. Many strategies have been developed for the separation of lead. Among which, solvent extraction is a more reliable strategy widely employed for separation, purification, and removal of various target metal ions. Various extractant have been used for the extraction of lead such as di-(2-ethyl-hexyl) phosphoric acid (HDEHP),[15] Cyanex 302,[16] tributylphosphate and bis(2,4,4-trimethylpentyl)phosphinic acid.[17] Bis-4,4'(5')-[ter.-butylcyclohexano]-l8-crown-6,[18] 2-(Dibutylcarbamoyl)benzoic Acid,[19] Acetic acid,[20] Fluosilicic Acid.[21] EDTA,[22] di(2-ethylhexy1)phosphoric acid (D2EHPA) and 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester (HEHEHP). [23] Weiyi et al extracted the lead from drinking water by using point-of-use filters [24] but this method requires faucets of point-of-use filters.

As a result, the robustness of the above methods work is concerned; those require either mineral acid media, high concentration of extractant, high time of extraction etc. Whereas, in the proposed system, the extraction was carried out in 1.0 M ammonium acetate and cyanex 301 concentrations were 5×10^{-3} M, indicating the system is comparatively eco-friendly and a step ahead in the direction of green chemistry.

The main goal of the proposed study was to develop a greener and precise method for the extraction of lead. The hard works have been made to optimize the extraction method. The beauty of the proposed method is less use of extractant concentration, recovery of

solvent, and does not require too much of instrumentation.

Experimental

Apparatus and reagents

Optical absorption measurements were carried out by using a visible spectrophotometer (Chemito 215D), a digital pH meter (Model LI-120, Elico, India) with glass and calomel electrodes was used for pH measurement and a digital flame photometer (PI, Modelno. 041, India) was used for determination of alkali and alkaline earth metals.

A stock solution of lead nitrate was prepared by dissolving 1.59 g of lead nitrate (AR grade Merck Specialties Private Limited, Mumbai, India) in 100 mL distilled deionized water and standardized gravimetrically.[25] A solution containing 100 µg/mL of lead(II) was prepared by appropriate dilution of the standard stock solution. A stock solution of ammonium acetate (4.0 M) was prepared by dissolving 30.08 g of ammonium acetate extra pure (Thomas Baker private Limited, Mumbai, India) in 100 mL distilled deionized water. Cyanex 301 (American Cyanamide Co) was used without further purification.

General procedure

To an aliquot of solution containing 50 µg/mL lead(II), 1 M ammonium acetate solution was added in a total volume of 10 mL. The solution was transferred to a separating funnel and equilibrated with 10 mL of 5 $x10^{-3}$M cyanex 301 in kerosene as a diluent, for 10 minutes on a wrist action flask shaker. The two phases were allowed to settle and separate. Lead(II) was stripped from the organic phase by shaking with 10 mL of 2.0 M HNO_3. Lead(II) in the aqueous phase was determined spectrophotometrically with PAR [26-28] at 520 nm using a calibration graph.

RESULTS AND DISCUSSION

Extraction of lead(II) as a function of ammonium acetate concentration with cyanex 301.

The optimum concentration for the quantitative extraction of lead(II) was ascertained by extracting lead(II) with $5x10^{-3}$ M cyanex 301 in kerosene as a diluent. In these experiments, the concentration of cyanex 301 in the organic phase was fixed at $5x10^{-3}$ M and the concentration of ammonium acetate was varied from $1x10^{-3}$ to 4.0 M as shown in (Figure 1). It was found that there was a quantitative extraction of lead(II) from $5x10^{-1}$ to 4.0 M ammonium acetate concentration. For further extraction studies, 1.0 M of ammonium acetate was selected and used.

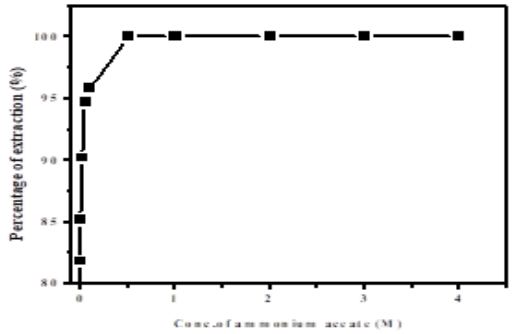

Figure 1. Extraction of lead(II) as a function of ammonium acetate concentration

Effect of varying concentration of cyanex 301

To ascertain the optimum concentration of cyanex 301 require for the quantitative extraction of lead(II), the concentrations of cyanex 301 were varied in the range of $1x10^{-3}$ to 1.0 M by using $5x10^{-1}$ M of ammonium acetate at as shown in (Figure 2). It was found that the extraction of lead(II) increases from 87 % at 1×10^{-3} M to 93.42% at $2x10^{-3}$ M and was quantitative from 4 x 10^{-3} M to 6 x 10^{-3} M, then extraction decreases at higher concentration. Hence, further extraction studies of lead(II) was carried out using 5 x 10^{-3} M cyanex 301 in kerosene.

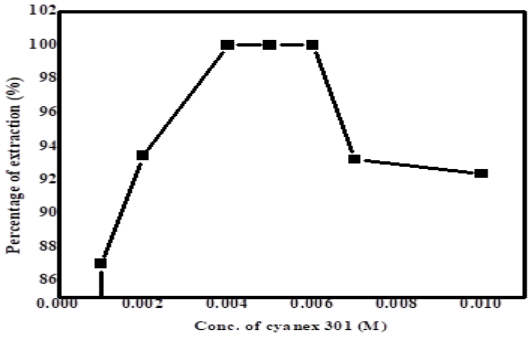

Figure 2. Effect of varying concentration of cyanex 301

Time of equilibration and choice of stripping agents

The extraction time was optimized by performing the equilibration for various periods (1-20 min). The results showed that with 10 minutes of equilibration there was quantitative extraction of lead(II). So, 10 minutes of equilibration time was used. After extraction, back extraction was carried out to separate lead(II) from the organic phase, therefore various stripping agents were tested. The back extraction of lead(II) from the organic phase was carried out using

HNO_3 H_2SO_4, HCl, $HClO_4$ as stripping agent.The concentrations of strippants were varied from 0.1-8.0 M. Back extraction of lead(II) was quantitative with 1.0–8.0 M nitric acid, 7.0–8.0 M sulphuric acid, 2.0-8.0 M hydrochloric acid, 0.1-8.0 M perchloric acid, 5.0 -8.0 M hydrobromic acid. Whereas all strippant are efficient strippant for lead(II) except acetic acid as shown in (Figure 3). Among all strippant nitric acid, hydrochloric acid, perchloric acid are most efficient strippants for lead(II). Further back extractions were carried out with 2.0 M HNO_3.

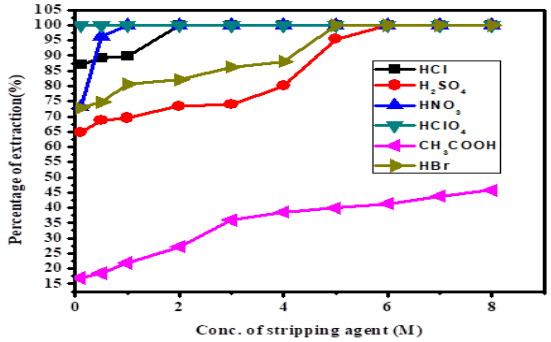

Figure 3. Choice of stripping agents

Effect of diluents on the extraction of lead(II)

In the extraction of lead(II) the influence of solvent is very important. The polarity of solvent seems to be the most decisive factor. Lead(II) extraction was carried out from 1.0 Mammonium acetate using $5x10^{-3}$ M cyanex 301 with various solvents, such as carbon tetrachloride, xylene, toluene, chloroform, kerosene, tetrachloroethane, n–dodecane as shown in (Table 1). The phase volume ratio was maintained at unity. It was observed that there was quantitative extraction with kerosene while carbon tetrachloride, xylene, toluene, chloroform, tetracholroethane & n-dodecane were found to be inefficient diluents. Kerosene was the cheapest and best diluent among tested and gives clear cut separation was achieved. Hence, kerosene was selected as a diluent for further study.

Table 1. Effect of diluents on the extraction of lead(II)

Diluents	Dielectric constant	% Extraction	Distribution ratio(D)
Chloroform	4.80	63	1.70
Carbon tetrachloride	2.24	76	3.1
Toluene	2.38	47	0.1
Kerosene	2.02	100	∞
Xylene	2.30	73	2.7
n-Dodecane	2.00	17	0.2
Dichloroethane	10.50	55	1.22
Tetrachloroethane	8.20	76	3.1

Effect of varying concentration of lead(II)

Lead(II) was extracted with 10 mL of $5x10^{-3}$ M cyanex 301 from1.0M ammonium acetate medium using kerosene as a diluent. The concentration of lead(II) was varied from 50-2000 µg /mL. It was found that 10 mL of $5x10^{-3}$ M cyanex 301 solutions was adequate to extract lead(II) quantitatively up to 700 µg /mL of sample solution as shown Figure 4.

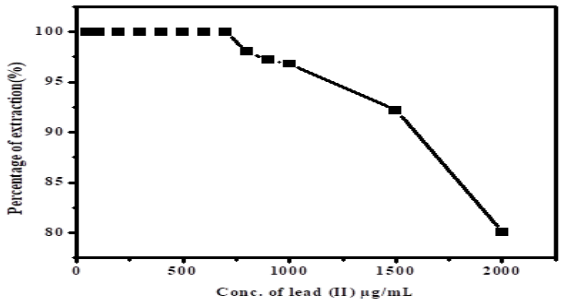

Figure 4. Effect of varying concentration of lead(II)

Applications

Study of diverse ion effect on extraction of lead(II)

Lead(II) was extracted in the presence of a wide range of foreign ions. The tolerance limit was set such that the amount of foreign ions required causing±2% error in the recovery of lead(II). The Zinc(II), cadmium(II) iron(II), zirconium(IV), copper(II) and cobalt(II) was co-extracted along with lead(II). Some d-block cations showed a low tolerance limit except manganese (II).But a most of the p-block and f-block cations showed a low tolerance limit. Most of the s-block and anions of organic and inorganic acids showed a low tolerance limit except manganese(II) and NO_3^-. The tolerance limit (mg) of various cations and anions are listed in (Table 2).

Table 2. Study of diverse ion on extraction of lead(II)

Ion	Added as	Tolerance limit (mg)	Ion	Added as	Tolerance limit(mg)
Li^+	LiCl	10	Tl^{3+}	$Tl(NO_3)_3.3H_2O$	5
Na^+	NaCl	8	La^{3+}	$La(NO_3)_36H_2O$	4
K^+	KCl	1	Y^{3+}	$Y(NO_3)_3$	5
Cs^+	CsCl	6	V^{4+}	$VOSO_4.H_2O$	5
Be^{2+}	$BeSO_4.4H_2O$	4	Th^{4+}	$Th(NO_3)_4.6H_2O$	0.5
Mg^{2+}	$MgCl_2.6H_2O$	5	Mo^{6+}	$(NH_4)_6Mo_7O_{24}4H_2O$	2.5
Ca^{2+}	$CaCl_2$	0.1	Cl^-	HCl	10
Sr^{2+}	$Sr(NO_3)_2$	1	Br^-	HBr	10
Ba^{2+}	$Ba(NO_3)_2$	2.5	SCN^-	NaSCN	10
Co^{2+}	$CoCl_2.6H_2O$	Co-extract	ClO_4^-	$HClO_4$	10
Ni^{2+}	$NiCl_2.6H_2O$	2.5	CH_3COO^-	CH_3COOH	10
Mn^{2+}	$MnCl_2.4H_2O$	12	SO_4^{2-}	H_2SO_4	0.01>
Zn^{2+}	$ZnCl_2$	Co-extra	Tartarate	Tartaric acid	0.2
Cd^{2+}	$(CH_3COO)_2CdH_2O$	Co-extra	EDTA	EDTA	0.1
Ce^{3+}	$Ce(NO_3)_3$	3	Oxalate	Oxalic acid	0.5
Sn^{2+}	$SnCl_2.2H_2O$	1	Citrate	Citric acid	0.5
Fe^{3+}	$FeCl_3.6H_2O$	Co-extra	Zr^{4+}	$Zr(NO_3)_4.4H_2O$	Co-extra
Cr^{3+}	$K_2Cr_2O_7$	0.075	NO_3^-	HNO_3	15
Cu^{2+}	$CuSO_4$	Co-extra	Sb^{3+}	$SbCl_3^{2-}$	0.01

Separation of lead(II) from multicomponent mixtures

The mixture containing lead(II), Iron(III), and beryllium(II)/ lanthanum(III), vanadium(IV) in 1.0 M ammonium acetate was extracted with $5x10^{-3}$ M cyanex 301 in kerosene. Under this set conditions lead(II) and iron(III) were extracted leaving behind beryllium(II), lanthanum(III), vanadium(IV) in the aqueous phase. From the organic phase, iron(III) was stripped with 0.5 M H_2SO_4 while lead(II) remained in the organic phase. Finally, lead(II) was stripped with 2.0M HNO_3. Also, the mixture containing lead(II), copper(II), and lanthanum(III), cerium(III), thallium(II) in 1.0 M ammonium acetate was extracted with $5x10^{-3}$ M cyanex 301 in kerosene. Under this set condition lead(II) and copper(II) were extracted leaving behind lanthanum(III), cerium(III), lanthanum(III), thallium(II) in the aqueous phase. From the organic phase copper(II) was first stripped with 1.0 M H_2SO_4 while lead(II) remained in the organic phase. Finally, lead(II) was stripped with 2.0 M HNO_3. The results show that good separation was achieved by using this method and recovery is often greater than 98 % as shown in (Table 3).

Table 3. Separation of lead(II) from multicomponent mixtures

No.	Mixture	Taken µg/mL	Found µg/mL	*Recovery percentage	Extractant	Strippant
1	Pb(II)	50	49.5	99.0	CX 301+AA	2.0M HNO_3
	Fe (III)	50	49.8	99.6	CX 301+AA	0.5M H_2SO_4
	Be(II)	50	49.9	99.8	Aq. Phase	-
2	Pb(II)	50	49.5	99.0	CX 301+AA	2M HNO_3
	Fe(III)	50	49.8	99.6	CX 301+AA	0.5M H_2SO_4
	La(III)	50	49.7	99.7	Aq. Phase	-
3	Pb(II)	50	49.5	99.3	CX 301+AA	2.0M HNO_3
	Fe(II)	50	49.8	99.6	CX 301+AA	0.5M H_2SO_4
	V(IV)	50	49.8	99.6	Aq. Phase	-
4	Pb(II)	50	49.7	94.4	CX 301+AA	2.0M HNO_3
	Cu (II)	50	49.5	99.0	CX 301+AA	1.0MH_2SO_4
	Ce(III)	50	49.9	99.8	Aq. Phase	-

5	Pb(II) Cu(II) Ba(II)	50 50 50	49.6 49.8 49.9	99.2 99.6 99.8	CX 301+AA CX 301+AA Aq. Phase	2.0M HNO_3 1.0MH_2SO_4 -
6	Pb(II) Cu(II) Tl(II)	50 50 50	49.8 49.8 49.5	99.6 99.6 99.0	CX 301+AA CX 301+AA Aq. Phase	2.0M HNO_3 1.0MH_2SO_4 -

Determination of lead(II) in real sample

Real sample of galena ore (PbS) (Geological sample)

Known weight of galena ore (PbS) was heated up to 700 ^{0}C for 2 hrs to remove organic matter. The PbS was treated with concentrated nitric acid and heated up to moist dryness. The solution was filtered through Whatmann filter paper number 41 and diluted to 100 mL with distilled deionized water. An aliquot sample solution was analyzed as per the general procedure and lead(II) content was determined. The amount of lead(II) found in Galena ore by general procedure was 57.9 % and the reported value of lead in galena ore was 60 %.

Real sample of solder alloy

The amount of lead(II) found in solder alloy by general procedure.Given the known weight of solder alloy was heated to remove organic matter. The solder alloy was treated with concentrated nitric acid and heated to moist dryness. The solution was extracted with distilled deionized water. At this stage, lead was converted into lead nitrate ($PbNO_2$) and tin was separated as metastannic acid. The solution was filtered through Whatmann filter paper number 41 and diluted to 100 mL with distilled deionized water. An aliquot sample solution was analyzed as per the general procedure and lead(II) content was determined. The amount of lead(II) found 38.0% in solder alloy and the reported value of lead in solder alloy was 40.0 %. Also by the same general procedure, the amount of lead (II) in gunmetal alloy was 1.70 % and the reported value of lead in gunmetal alloy was 2.31 %.

CONCLUSIONS

From investigation, it is concluded cyanex 301 is a potential extractant for the extraction of lead(II) from ammonium acetate medium. The proposed method has advantages such as easy phase separation, less equilibration time and single extraction is sufficient for quantitative extraction of lead(II). Cyanex 301 extracts lead(II) very rapidly, equilibrium was reached within 10 minutes. The method permits the separation of lead(II) from other elements. The method was extended to the determination of lead in real samples. The present investigation aims to demonstrate a simple, selective with cheap method and has good reproducibility (approximately ± 2%).

Declarations

Competing interests

The authors declare that they have no competing interests.

Funding

Not applicable.

Availability of data and materials

Not applicable

Code Availability

Not applicable

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