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Liquid-Liquid Extraction of Cd(II) Metal ion using Trialkyl Phosphine Oxide Extractant

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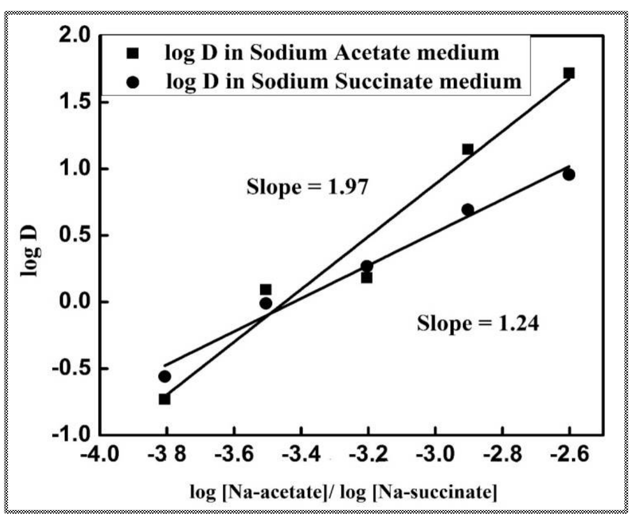
ABSTRACT

Solvent extraction method has been proposed for the extraction of Cd(II) using trialkyl phosphine oxide (Cyanex-923), in toluene from the salt of weak organic acid like sodium acetate and sodium succinate media. Various parameters such as the effect of pH, effect of Cyanex-923 concentration, effect of sodium acetate and sodium succinate concentration, effect of diluents, effect of time and stripping agent, composition of extracted species, effect of temperature, aqueous to organic volume ratio and effect of metal loading capacity. FTIR of the extracted species in the organic phase containing cyanex-923 were also studied. The proposed method was extended for extraction and separation of Cadmium(II) from spent Ni-Cd battery waste.

Graphical Abstract



Effect of sodium acetate/ sodium succinate concentration on Distribution ratio of Cd(II).

Keywords: Cyanex-923, Sodium acetate, Sodium succinate, Cd(II), Stripping, Ni-Cd battery

INTRODUCTION

Heavy metal cadmium is toxic and poisonous due to inhalation or ingestion of cadmium metal through various means like air, food, water etc. Cadmium enters the environment from the process of smelting, refining of ZnS ore and mining. Ni-Cd batteries which is widely used as rechargeable batteries and its spent which are discarded directly into environment, soil and water contributes to the discharge of toxic metal cadmium [1]. Nickel-Cadmium batteries which are mostly used in laptops, computers, video cameras and portable tools since it can deliver high current. These batteries after many cycles of use cannot further being charged and they are discarded [2]. Therefore, a large concern for the recovery and treatment of cadmium from spent Ni-Cd storage batteries.

In last decades, different acidic organophosporus extractants like Cyanex-302, Cyanex-301 and Cyanex-272 were used for extraction of cadmium [3].Other extractants that can extract Cd(II) are 2-theonyl trifluoroacetate [4], Amberlite XAD-7 coated with organophosphorus extractant Cyanex-301 [5] and Amberlite XAD-2 resin coated with extractants like Cyanex-272 and Cyanex-302 [6].

Cd(II) was extracted from various medium such as sulphate, phosphate, nitrate, chloride using different extractant such as Cyanex-272, TOPS 99, PC88A [7], bis(2-ethylhexyl) phosphoricacid [8], Cyanex-301[9], quaternary ammoniums [10], pyridyl ketoximes [11] and Cyanex-923 and Cyanex-272 [12].

The present work is aimed at exploring the effect of acetate (CH_3COO^-) and succinate ($C_2H_4(COO)_2^{2-}$) ions on the extraction of Cd(II) using Cyanex-923 in toluene. The advantages of this method over other reported methods are that the mediums used in this work are not corrosive acids such as H_2SO_4, H_3PO_4, HNO_3, HCl, but are the salts of weak acids. Further the extraction of Cd(II) was carried out from spent Ni-Cd battery waste.

MATERIALS AND METHODS

Apparatus and Reagent: A measured amount of $CdCl_2$ was dissolved in minimum quantity of hydrochloric acid and final dilution was done with double distilled. Cyanex-923 extractant was supplied by Cytec Industries Inc. Canada, was used without further purification. 4-(2-pyridylazo) resorcinol (PAR), sodium acetate, sodium succinate, different strong and weak acids, solvents and metal salts used were of AR grade. Instrument used for adjusting pH was (Elico model LI 120) and Lab UV Nexgen spectrophotometer was used for absorbance measurements. 3000 Hyperion Microscope with vertex 80 FTIR system was used to study the extracted species in the organic phase.

General Extraction Conditions: 10 µg of Cd(II) solution containing CH_3COONa and $C_2H_4(COO)_2Na_2$ was prepared respectively. The pH of the solution was then maintained at 8.0 for both the mediums with dilute HCl and NH_4OH solutions. The solutions were diluted to 10 mL with distilled water and then transferred to a separating funnel containing 10 mL of Cyanex-923 diluted in toluene. The two immiscible phases were hand shaken for about 10 min and then allowed to separate for 5 min. After the separation of two phases, the organic phase containing metal ion was stripped with 4M HNO_3 and 3M H_2SO_4 respectively. Then the amount of Cd(II) was determined spectrophotometrically at 496 nm by PAR method [13].

RESULTS AND DISCUSSION

Effect of pH and Cyanex-923 extractant concentration: Using CH_3COONa and $C_2H_4(COO)_2 Na_2$ mediums, the extraction of Cd(II) was done with Cyanex-923 in toluene at different pH values. The effect of pH on the extraction was carried out in the pH range of 1.0-9.0. It was observed that on increasing the pH, the extraction goes on increasing and it was found to be quantitative at pH 8.0 in CH_3COONa as well as in $C_2H_4(COO)_2Na_2$ medium. Therefore, all the extractions were carried out at pH 8.0 for both the mediums (Fig. 1). Extraction of Cd(II) in both the mediums was done by changing the concentration of Cyanex-923 in toluene. In CH_3COONa medium, the concentration of Cyanex-923 was varied from 7.8 x10^{-5} M to 2.0 x 10^{-2}M, whereas in $C_2H_4(COO)_2Na_2$ medium the concentration of Cyanex-923 was varied from 2.5 x 10^{-3} M to 1.5 x 10^{-2} M keeping all the other parameters like concentration of medium, extraction time, diluent and temperature constant. It was found that the extraction increased with increase in concentration of Cyanex-923. Hence, in CH_3COONa medium the concentration of Cyanex-923 was fixed at 2.5 x 10^{-3} M and in $C_2H_4(COO)_2Na_2$ medium the concentration of Cyanex-923 was fixed at 1.5 x 10^{-2} M where the extraction was quantitative (Fig. 2 and 3).

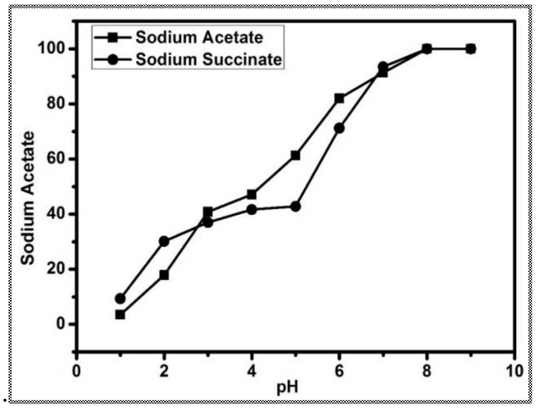


Figure 1. Effect of pH on the percentage extraction of Cd(II) in sodium acetate/ sodium succinate medium with Cyanex-923 in toluene.

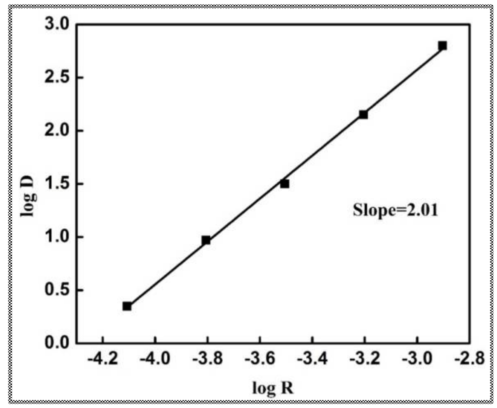


Figure 2. Effect of Cyanex-923 concentration on Distribution ratio of Cd(II) in sodium acetate medium.

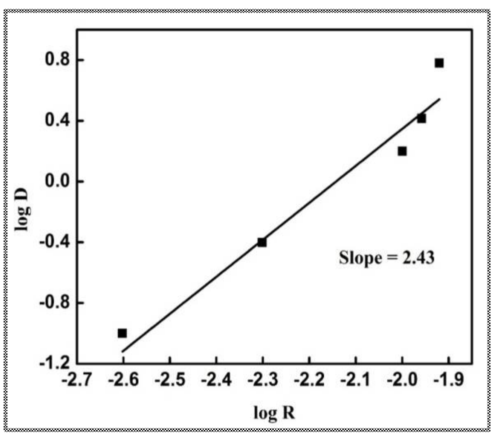


Figure 3. Effect of Cyanex-923 concentration on Distribution ratio of Cd(II) in sodium succinate medium.

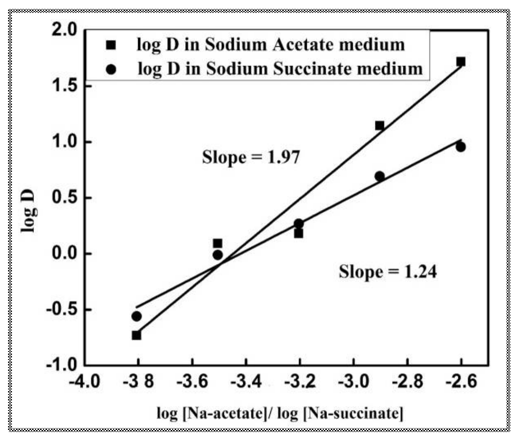


Figure 4. Effect of sodium acetate/ sodium succinate concentration on Distribution ratio of Cd(II).

Effect of sodium acetate and sodium succinate concentration: The effect of CH_3COONa concentration on the percentage extraction of Cd(II) with 2.5 x 10^{-3} M Cyanex-923 in toluene at fixed pH of 8.0 has been studied in the CH_3COONa concentration range from 1.56 x 10^{-4} M to 4.0 x 10^{-2} M. It was found that as the concentration of CH_3COONa increased, the extraction of Cd(II) also went on increasing and became quantitative in the range of 5.0 x 10^{-3} M to 4.0 x 10^{-2} M with Cyanex-923 in toluene. Hence, all the extractions were carried out in 5.0 x 10^{-3} M of CH_3COONa with Cyanex-923 in toluene. On the other hand, the effect of $C_2H_4(COO)_2Na_2$ concentration on the percentage extraction of Cd(II) with 1.5 x 10^{-2} M Cyanex-923 in toluene at fixed pH of 8.0 has been studied in the

$C_2H_4(COO)_2Na_2$ concentration range from 7.8 x 10^{-5} M to 1.0 x 10^{-2} M. As the concentration of $C_2H_4(COO)_2Na_2$ increases, extraction of Cd(II) goes on increasing and becomes quantitative in the range 2.5 x 10^{-3} M to 1.0 x 10^{-2} M. Hence all the extractions were carried out at 2.5 x 10^{-3} M of $C_2H_4(COO)_2Na_2$ with Cyanex-923 in toluene (Fig. 4).

Effect of Diluents, Time and Stripping agent: Different organic solvents like toluene, chloroform, cyclohexane, n-hexane, xylene, carbon-tetrachloride, dichloromethane, amyl alcohol, n-butanol, methyl isobutyl ketonewere employed for the extraction of Cd(II). The extraction of Cd(II) was found to be quantitative in toluene for both the mediums i.e. CH_3COONa and $C_2H_4(COO)_2Na_2$ with Cyanex-923. However, there was incomplete extraction with other solvents. Toluene was found to be the best diluent due to better phase separation (Table 1).

Table 1. Effect of diluent on the percentage extraction of Cd(II) in sodium acetate and sodium succinate medium using Cyanex-923.

Diluent	% E of Cd(II)	
	Sodium Acetate Medium	Sodium Succinate Medium
Toluene	99.9	99.9
Xylene	91.9	74.3
Chloroform	86.1	98.1
n- Hexane	80.0	20.0
Carbon tetrachloride	96.9	93.3
Cyclohexane	92.8	98.2
Dichloromethane	89.9	98.3
Amyl Alcohol	56.0	92.9
n- Butanol	91.8	78.4
Methyl Isobutyl Ketone	90.7	92.7

The effect of different periods of shaking was studied in the range from 1-15 mins. It was observed that a time period of 10 min was sufficient in both the mediums for quantitative extraction of Cd(II) with Cyanex-923 in toluene (Table 2). However, there was no adverse effect by increasing the extraction period upto 15 min.

Table 2. Effect of period of equilibration on percentage of extraction of Cd(II) in sodium acetate and sodium succinate medium.

Time (min)	% E of Cd(II)	
	Sodium Acetate Medium	Sodium Succinate Medium
1	77.0	47.8
3	96.7	85.1
5	99.4	92.0
10	99.9	99.9
15	99.9	99.9

The stripping of Cd(II) from metal loaded organic phase was done using different acids like HCl, H_2SO_4, HNO_3, CH_3COOH, $HClO_4$, HCOOH. Cd(II) was quantitatively stripped in CH_3COONa medium with 3M HNO_3 with Cyanex-923 and in $C_2H_4(COO)_2Na_2$ medium Cd(II) was quantitatively stripped with 2M HNO_3in Cyanex-923 (Table-3).

Composition of Extracted Species: On plotting the graph of log D v/s log R (R = Cyanex-923) in CH_3COONa medium at pH 8.0 (Fig. 2) and in $C_2H_4(COO)_2Na_2$ medium at pH 8.0 (Fig. 3) the probable composition of extracted species was determined. The slope found in CH_3COONa medium is 2.01 which is around 2, showing that two molecules of Cyanex-923 react with one molecule of Cd(II) ion and the slope in $C_2H_4(COO)_2Na_2$ is 2.43 which is also around 2, again it shows that two molecules of Cyanex-923 react with one molecule of Cd(II) ion. Hence, the probable stoichiometric ratio obtained for Cd(II) to Cyanex-923 in both mediums is 1:2 [14].

Table 3. Effect of stripping agent on percentage recovery of Cd(II) from metal loaded organic phase in sodium acetate/ sodium succinate with Cyanex-923 in toluene.

% Recovery of Cd(II) from metal loaded organic phase								
	Sodium Acetate Medium					Sodium Succinate Medium		
	1M	2M	3M	4M	5M	1M	2M	3M
HCl	30.7	31.3	46.9	57.7	99.3	42.6	72.1	99.8
HNO_3	83.7	97.5	99.5	-	-	67.9	99.9	-
H_2SO_4	99.0	-	-	-	-	99.9	-	-
CH_3COOH	96.6	-	-	-	-	96.4	-	-
$HClO_4$	99.4	-	-	-	-	99.0	-	-
HCOOH	28.5	82.0	-	-	-	24.9	85.4	-

The graph of log D v/s $[CH_3COO^-]$ gave a slope of 1.97, indicating that two molecules of CH_3COO^- ions react with one molecule of Cd(II) and the slope of log D v/s $[C_2H_4(COO)_2^{2-}]$ is 1.24, indicating one molecules of $(C_2H_4(COO)_2^{2-})$ ions react with one molecule of Cd (II). The cadmium complex is extracted in the organic phase by solvation mechanism. Thus the probable extracted species of Cd(II) in the organic phase is $[Cd(CH_3COO)_2]$.2Cyanex-923 and $[Cd(C_2H_4(COO)_2)]$. 2Cyanex923.

$$Cd^{2+}_{(aq)} + 2(CH_3COO^-)_{(aq)} + 2\text{Cyanex-923}_{(org)} \rightleftharpoons [Cd(CH_3COO)_2].2\text{Cyanex-923}_{(org)} \quad (1)$$

$$Cd^{2+}_{(aq)} + (C_2H_4(COO)_2^{2-})_{(aq)} + 2\text{Cyanex-923}_{(org)} \rightleftharpoons [Cd(C_2H_4(COO)_2)].2\text{Cyanex923}_{(org)} \quad (2)$$

Effect of Temperature: The extraction of Cd(II) was carried out in both mediums using Cyanex-923 in toluene at different temperatures. The extractions of Cd(II) in CH_3COONa medium with 2.5 x 10^{-3} M Cyanex-923 in toluene at pH 5.0 and with 1.5 x 10^{-2} M Cyanex-923 at pH 6.0 in $C_2H_4(COO)_2Na_2$ medium were carried out at different temperatures ranging from 303K to 343K. It was observed that with increasing the temperature the percent extraction increases and so the distribution ratio.

The Van't Hoff equation is,

$$\log D = -(\Delta H/2.303\ RT) + C \quad (3)$$

where, D represents the distribution ratio, ΔH is the enthalpy change for the reaction and C is a constant.

The slope obtained from the plot of log D v/s 1/T x 1000 in CH_3COONa medium is -2.73 and in $C_2H_4(COO)_2Na_2$ medium, it is -2.69 (Fig. 5). These slopes have been used to calculate ΔH, ΔG, ΔS (Table 4).

$$\text{Slope} = -\Delta H/2.303\ R \quad (4)$$

$$\Delta G = -RT\ln D = -2.303RT\log D \quad (5)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (6)$$

The thermodynamic parameters i.e. ΔH, ΔG, ΔS in the table 4 have been calculated by using equations 4, 5 and 6. The ΔH value obtained for CH_3COONa medium is 52.27 kJ mol^{-1} and that for $C_2H_4(COO)_2Na_2$ medium is 51.50 kJ mol^{-1}, indicating that the reaction in both the mediums is endothermic in nature.

Table 4. Thermodynamic parameters of the extraction of Cd(II) from Sodium Acetate and Sodium Succinate medium by Cyanex-923 in toluene solution.

Temp (K)	Sodium Acetate Medium			ΔH kJ mol^{-1}	Sodium Succinate Medium			ΔH kJ mol^{-1}
	Log D	-ΔG kJ mol^{-1}	ΔS J mol^{-1}		Log D	-ΔG kJ mol^{-1}	ΔS J mo^{l-1}	
303	0.059	0.342	173.6		0.005	0.029	170.0	
313	0.480	2.876	176.1		0.150	0.898	167.4	
323	0.870	5.380	178.4	52.27	0.500	3.092	69.0	51.50
333	1.000	6.375	176.1		0.820	5.228	170.3	
343	1.120	7.350	173.8		0.970	6.370	168.7	

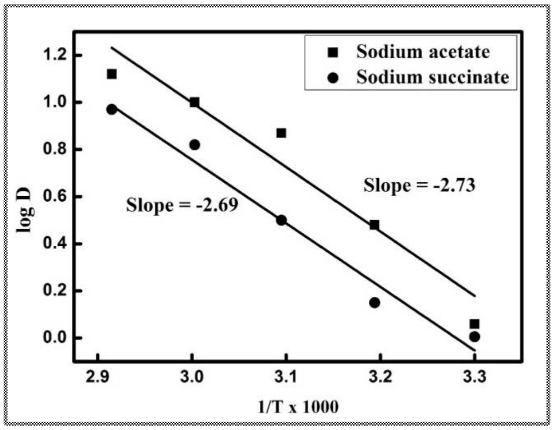


Figure 5. Effect of temperature on distribution ratio Cd(II) in sodium acetate/sodium succinate medium.

The ΔG value obtained is negative which shows that extraction of Cd(II) in both the mediums is thermodynamically favourable and the reaction is spontaneous. The positive ΔS value shows that the Cd(II) has been transferred from highly ordered water structure to the disordered state of the organic phase [15].

Effect of aqueous to organic volume ratio: The effect of different aqueous to organic volume ratios on extraction of Cd(II) in 5.0 x 10^{-3} M CH_3COONa and 2.5 x 10^{-3} M $C_2H_4(COO)_2Na_2$ medium using Cyanex-923 have been studied. Keeping the volume of organic phase same, the volume of aqueous phase containing Cd(II) was changed from 10 to 300 mL. In CH_3COONa and $C_2H_4(COO)_2Na_2$ medium, it was observed that there was increase in percentage extraction when the aqueous to organic phase ratio was varied 30:1 to 1:1. The quantitative extraction was observed from 6:1 to 1:1 in case of CH_3COONa medium while 1.5:1 to 1:1 for $C_2H_4(COO)_2Na_2$medium. Therefore, the phase ratio in both medium for the extraction of Cd(II) was kept fixed at 1:1 (Table 5)

Table 5. Effect of aqueous to organic volume ratio.

Aq : Org Vol (mL)	Sodium Acetate % E	Sodium Succinate % E
10 : 10	100	100
15 : 10	100	100
25 : 10	100	96.7
40 : 10	100	96.4
50 : 10	100	91.8
60 : 10	100	88.1
75 : 10	96.9	82.9
100 : 10	96.3	77.4
150 : 10	82.3	74.3
200 : 10	71.5	69.5
300 : 10	45.8	66.8

Metal Loading Capacity: The effect of the initial Cd(II) concentration on percentage extraction keeping all the parameters fixed has been studied. The concentration of Cd(II) was varied from 10-100 µg in both the mediums using Cyanex-923. It was observed that till 20 µg the extraction is quantitative in both the mediums i.e. CH_3COONa and $C_2H_4(COO)_2Na_2$. Further, it was observed that in both the medium the percent extraction decreased on increasing the concentration of Cd(II). Therefore, for quantitative extraction of Cd(II) in CH_3COONa and $C_2H_4(COO)_2Na_2$ medium using Cyanex-923 in toluene the loading capacity is 20 µg (Table 6).

Table 6. Metal Loading Capacity.

Amount (µg)	Sodium Acetate % E	Sodium Succinate % E
10	100	100
20	100	100
30	94.0	88.1
40	89.7	54.2
50	87.3	45.2
60	79.9	20.5
70	79.5	20.4
80	79.0	20.0
90	79.1	20.0
100	79.4	20.0

FTIR Study: Cyanex-923 in toluene loaded with Cd(II) is analyzed by FTIR spectroscopy in the range of 400-4000 cm^{-1} in both the medium. For comparison the unloaded Cyanex-923 in toluene was also analyzed by FTIR. The peak at 1161 cm^{-1} was assigned to P=O stretching vibration in Cyanex-923 which is shifted to 1143.42 cm^{-1} in Cd-loaded Cyanex-923 in CH_3COONa and in $C_2H_4(COO)_2Na_2$ media the shift was observed at 1149.63 cm^{-1}. This shift to lower band and reduction of intensity indicates that Cyanex-923 in toluene takes part in co-ordination reaction between Cd(II) and oxygen atom of P=O group [16] of Cyanex-923. In addition, all the three spectra shows some characteristic CH- stretching vibration in toluene solvent at around 2900 cm^{-1} to 2850 cm^{-1} (Fig. 6).

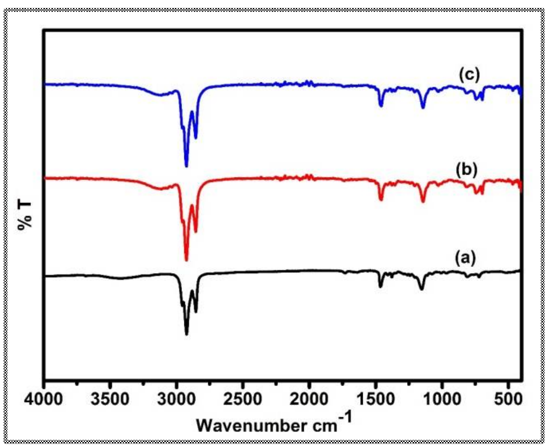


Figure 6. Comparison of FTIR spectra of the organic phase, figure 6(a) - Cyanex-923 in toluene before extraction, figure 6(b) - Cyanex-923 in toluene with Cd(II) and in acetate medium after extraction, figure 6(c)-Cyanex-923 in toluene with Cd(II) and in succinate medium after extraction.

APPLICATION

Determination of Cd(II) from spent Nickel Cadmium Battery Waste: Recovery of Cd(II) from spent Nickel Cadmium battery waste has been successfully accomplished. 0.1 g of battery waste was treated with 15 mL of concentrated nitric acid and the solution was evaporated to almost dryness.

Table 7. Nickel Cadmium Battery Waste

Sample	Sodium Acetate Medium				Sodium Succinate Medium			
	Present µg	Found µg	% Recovery	R.S.D	Present µg	Found µg	% Recovery	R.S.D
Spent Ni-Cd Battery	10	9.55	99.5	0.10%	10	9.44	94.4	0.15%

Then, the residue obtained after drying was extracted into 50mL of double distilled water and this solution was boiled, cooled and filtered. The filtrate obtained was diluted to 100 mL with double distilled water. This sample solution was found to contain 100 ppm of Cd(II) determined by atomic absorption spectrometer (AAS). The solution having 10 µg of Cd(II) were taken and its pH was maintained at 8.0 and the extraction of Cd(II) was carried out with 10ml of 2.5 x 10^{-3} M of Cyanex-923 in CH_3COONa medium and with 1.5 x 10^{-2} M of Cyanex-923 in $C_2H_4(COO)_2Na_2$medium. The extracted Cd(II) in organic phase was then stripped with 3M HNO_3in CH_3COONa medium and with 2M HNO_3 in $C_2H_4(COO)_2Na_2$ medium. The experimental results are listed in (Table-7).

CONCLUSIONS

1. The result indicates that use of salts of weak organic acid likeCH_3COONa and $C_2H_4(COO)_2Na_2$ has been successfully used in extraction of Cd(II).
2. The quantitative extraction of Cd(II) in sodium acetate was observed at 0.0025M Cyanex-923 in toluene and in sodium succinate medium it was with 0.015M Cyanex-923 in toluene.
3. Cd(II) was extracted into organic phase by solvation mechanism through formation of [Cd(acetate)$_2$]. 2Cyanex-923 in sodium acetate medium and [Cd(succinate)]. 2Cyanex923 in sodium succinate medium.
4. FTIR study of the complex after extraction in both the mediums showed co-ordination of Cd(II) and P=O group of the Cyanex-923 in toluene.
5. The extraction of Cd(II) in both the mediums is endothermic and spontaneous confirmed by temperature study.
6. The proposed method is used for the extraction of cadmium from spent Ni-Cd battery waste.

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