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Research paper

Extraction and Determination of Nickel (Ii) Via Cloud Point Method

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Abstract

Spectrophotometric study for ion pair complex of Ni(II) ion extracted into cloud point extraction larger appear wave length of maximum absorbance was λ=660 nm, for extracted Ni(II) ion as ion pair association ,used laboratory synthesized azo derivative 2-(4-hydroxy phenyl azo)-4-Benzen naphthol(HPAB).The study about Determination the optimum condition for extraction and determination was pH =10 in presence 100 µg Ni(II) in 10 ml aqueous solution ,0.6 mL of Triton x-114 and temperature of heating was 70 c° at heating time equal to 30 minute, stoichiometry shows the ion pair complex extracted was 1:1 (Ni-HPAB) +2;2Cl_, in addition to electrolyte effect study as well as spectrophotometric determination of Ni(II) in different samples .

Keywords: *Cloud point, solvent extraction, Nickel(II), author.*

1. Introduction

Sensitive extraction method used for separation and determination Ni(II) involved coupling solvent extraction with cloud point extraction method, so it is worth mentioning there is previous studies about extraction Ni(II) ion, by cloud point extraction method for extraction and determination Cd+2 and Pb+2 with two azo derivative compound (BTABP) and (BPADPI) [1]. In research for extraction and determination Nickel(II)from acidic medium by used crown ether DBBC6 by application cloud point extraction method in the presence Triton x-100 [2], another application for cloud point method for extraction and determination Cobalt (II) by using cloud point method coupled with solvent method and by using 2,4- dimethylpentan-3-one[4]. Joind cloud point extraction (CPL) method with flame atomic absorption spectrophotometer for separation and determination of trace levels of silver by using Triton x-114and dimethyl dithiocarbamate (DDTC) as complexing agent, the calibration curve was linear in the range of 1-500 ng mL-1 will a limit of detection (LOD) at 0.3 ng mL-1[5]. Separation and determination of trace amount of copper with cloud point extraction CPE method by using complexing agent 1,2-dimethyanthraquinone -3- sulfonic acid sodium salt so that was added surfactant octylphenoxypolyethoxyethanol, the determination limit was 1.07 ng mL-1 [6]. Preconcentration by cloud point extraction CPL method and graphite furnace atomic absorption spectrometry (GFAAS) for determination of Nickel(II) in water samples with using Triton x-114 as surfactant and 1-phenyl- 3-methyl -4-benzoyl-5-pyrazolone (PMBP) as complexing agent, the experimental process under optimum condition appear detection of limit (LOD) 0.12 ng mL-1 [7]. Incorporation graphite furnace atomic absorption spectrometry with cloud point extraction method for isolation and determination Pb, Cd, Cu and Ni in environmental surface water [8]. Extraction and determination of cadmium(II) in different samples by onium technique by using 2,4-dimethyl pentan-3-one in 0.5M HCl acidic medium so the onium species giving wave length for maximum absorbanceλMax=260 nm [9]. Joined cloud point extraction (CPL) method with solvation technique for separation, Preconcentration and determination of La(III) by using 2,4-dimethyl pentan-3-one and in order to determination La(III) in aqueous solution used 8-Hydroxyquinoline and safranim dissolved in chloroform so for cloud point extraction used Tween-20 and Tween -80 as surfactant[10].By joined CPL method with onium technique in order to separation and spectrophotometric Determination of Co(II),by using (N-benzoyl-L-arginine ethyl ester hydrochloride)(N-BAEH) with Tritonx-100, the experimental studies shows λ=294nm[11]. Extraction and determination of Molybdenum((VI) by incorporation cloud point extraction(CPL) method with liquid ion exchange methods by using 2-(benzenethiozoylazo)-4-benezen naphthol as complexing agent by using Tritonx-100 as surfactant[12]. Cloud point extraction CPL method used for separation and determination trace amount of Lead(II) in Deferent water samples by used 4-(pyridylazo) resorcinol as chelatscomplexing agent at pH =6 in present Tritonx- 114 so this application give detection of limit LOD(1.15µg L-1) [13]

1.1. Experimental

For spectrophotometric studies and absorbance measurements, used Biochrom double beam spectrophotometer model (Biochrumlibra 560) (A Harvard Bio science company Cambridge UK), so for heating used water bath with regulator (Hamburg – 90) England and electrical balance company limited, Dool, CE, HR200, Japan, Stock Solution Ni^{2+}ion in concentration 1000µg/mL prepared by dissolved 0.0405g of$NiCl_2$ $6H_2O$ in 10ml distilled water in volumetric flack µn presence 0.5 ml of conc. Hydrochloric acid HCL and another solution prepared by dilution with distilled water.

1.2. The general method

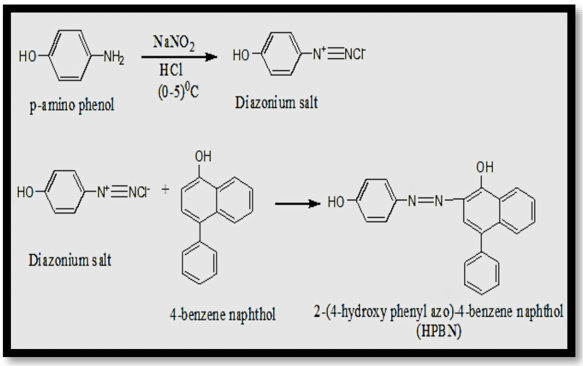
10ml aqueous solution contain 100µg Ni^{2+}ion or any other quantity with 5X10-5M or HPAB at pH=10and in presence 0.5ml of Tritonx – 114, heat this solution for suitable temperature and time until formationcloud point layer, then separated this layer and dissolved in 5ml ethanol and measure the absorbance of alcoholic solution against blank prepared at the same manner in absence Ni^{2+}ion at wave length of maximum absorbance for ion pair association complex extracted to CpL (λ_{max}) 660nm, on well on the aqueous phase treated according to DMG spectrophotometric method [14] and return to calibration care to determine the remainder quantity of Ni^{2+}ion in aqueous solution at extracted the subtraction thisquantity from the original quantity of Ni^{2+}ion which is already in the aqueous solution to determine the transfer quantity of Ni^{2+}ion to the CpL in order to formation an association complex afterward calculated distribution ratio.

$$D= \frac{[Ni^{+2}]_{CPL}}{[Ni^{+2}]aq}$$

2. Results anddiscussion

2.1. Synthesized HPAB

The organic reagent [2-(4- Hydroxy phenyl azo)-4 – benzenenaphthol HPAB laboratory synthesized according to the procedure detailed in [16]:



2.2. Spectrophotometric study

After extracted Ni^{2+}ion as ion association complex toCpL according to the general method and dissolved CpL in 5 ml ethanol the spectrum of alcoholic solution isillustrated in fig (1)

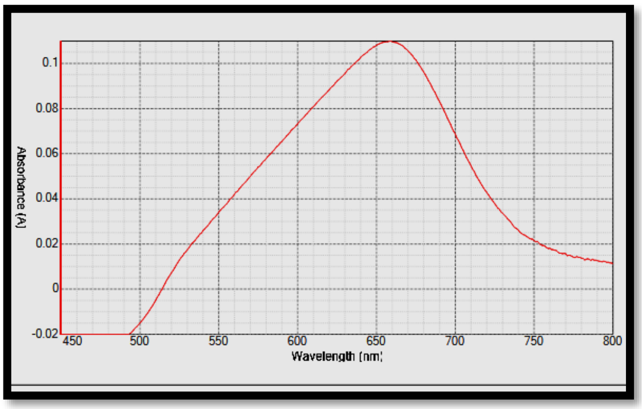


Fig. 1:UV-VIS absorbance spectrum of ion pair association complex of Ni^{+2}.

The spectrum the of ion pair association complex extracted to CpL appear wave length of maximum absorption wasλ_{max}= 660 nm

2.3.Effect of pH

10mL aqueous solution contain 100µg $Ni^{2+}$and 0.5mL of Tritonx -114 and $5X10^{-5}$MHPAB at different pH, heat these solutions at suitable temperature and time until formation cloud point layer CpL and Complete the works in the general method the result were as in figures 3,4.

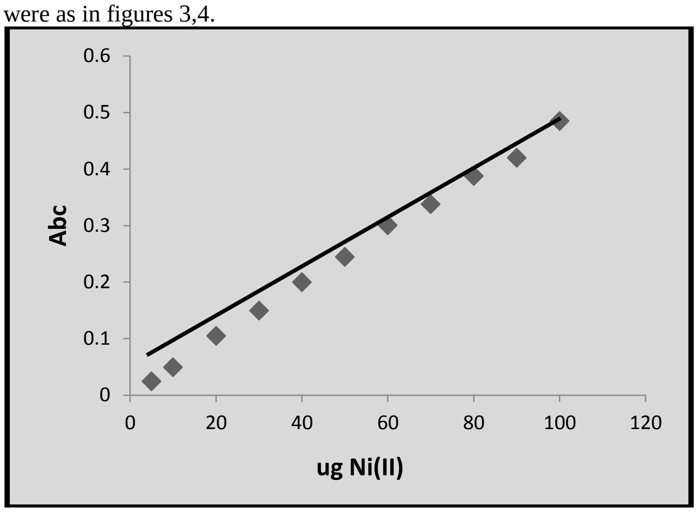


Fig. 2: Calibration curve for determination Ni+2 ion remain in the aqueous phase after extraction

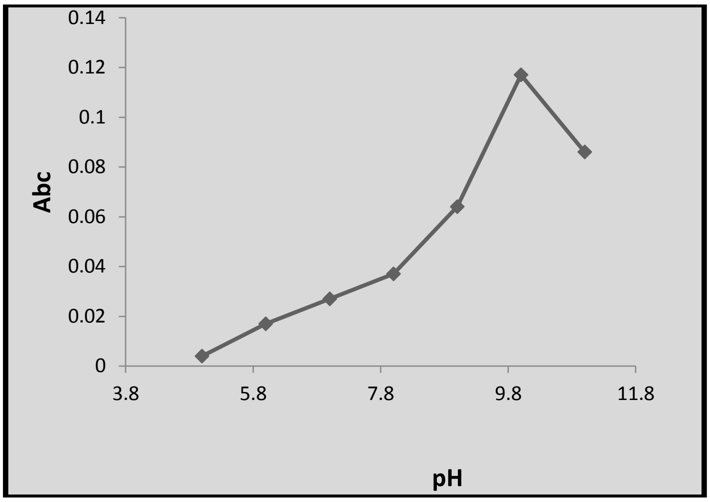


Fig. 3: Effect of pH on formation and stability of ion pair complex

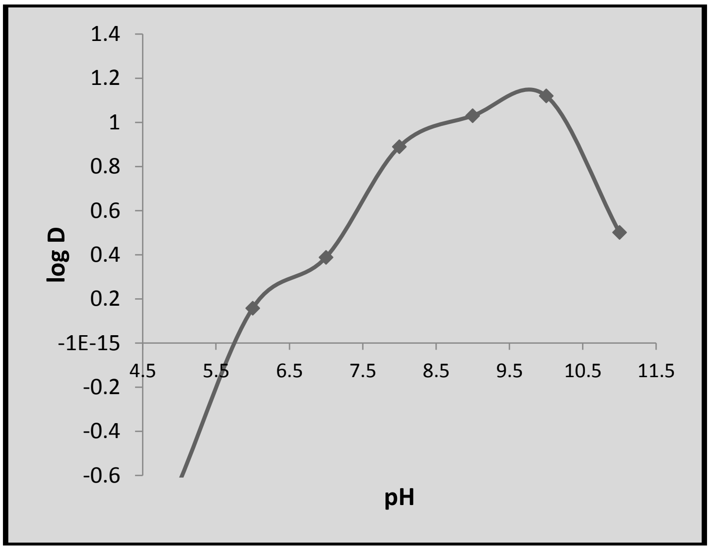


Fig. 4:D=F pHof ion pair complex extracted

The results shows pH=10 was the optimum pH give higher extraction efficiency because at this pH we get best thermodynamic equilibrium for formation ion pair associationcomplex and increase extraction efficiency in acidic medium decrease binding between HPAB and Ni^{2+}ion by reason of prolongation the position of donerelectron so that at more basic medium there is a decline in extraction efficiency also because of the formation stable compound with hydroxide ion.

2.4. Effect ofNi^{2+}concentration

A series aqueous solutions 5ml in volume contain increasing quantities at pH=10 with 0.5ml Triton X-114 and $5X10^{-5}$MHPAB heat there solutions at suitable temperature and time until formation cloud point layer CpL complete the procedure as in the general method the result were illustrated in the figures (5,6)·

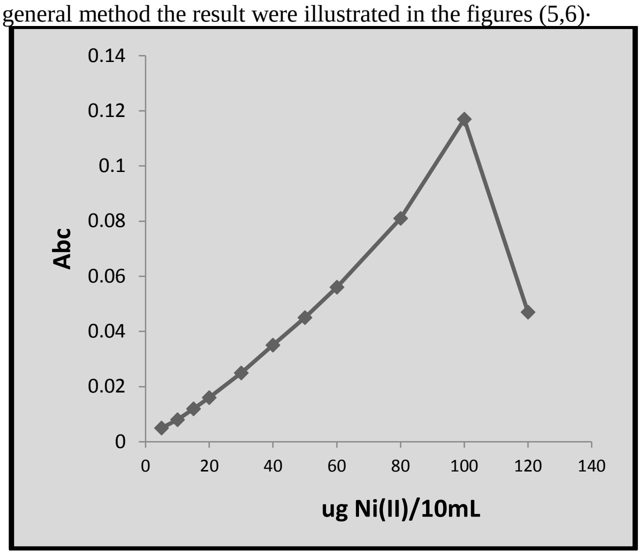


Fig. 5: Metal ion concentration

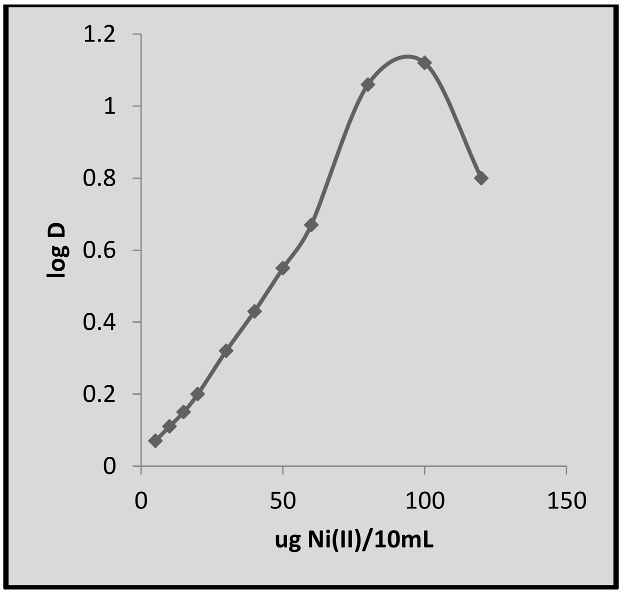


Fig. 6: Metal ion concentration

2.5. On formation and stabilityof ion pair association complex

The results shows 100 µg Ni^{2+}in 10mLaqueous solution was the optimum concentration give higher extraction efficiency because the metal ion concentration is the thermodynamic parameter any concentration less than optimum value effect to decrease extraction efficiency so that concentrations more than optimum value give decline in extraction efficiency by effect of mass action law.

2.6. Effect of surfactant volume

A series of 10mL aqueous solution contain 100µgNi^{+2}at pH=10 in present of 5X10-5 M HPAB with increasing volume at TritonX-114 and heating the solution for suitable temperature and time until formation CpL and complete the work as in the general method the result were illustrated in fig 7, 8.

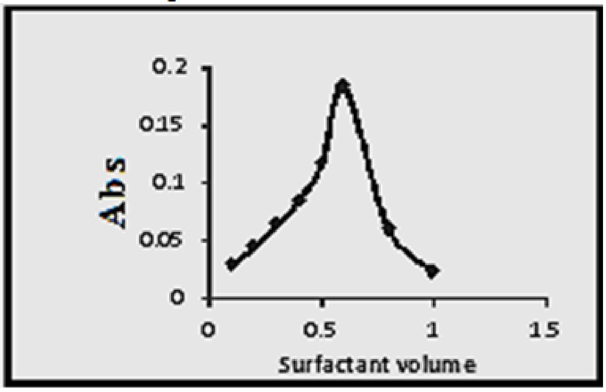


Fig. 7: Effect of Surfactant Volume on Formation CPL

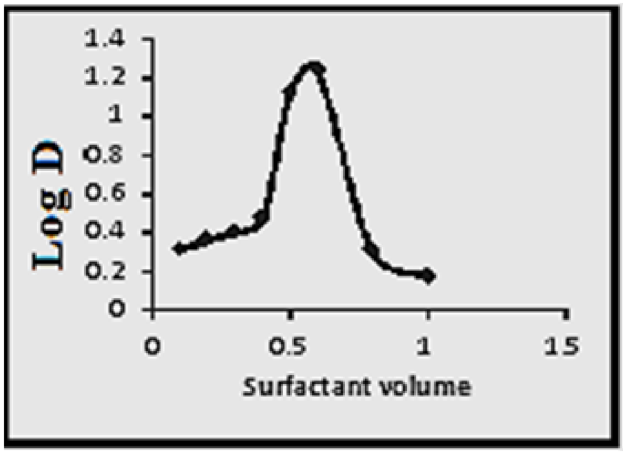


Fig. 8: Effect of surfactant volume on extraction CPL

The optimum volume of Tritonx-114 was 0.6Ml which is giving best cloud point layer able to extract higher concentration of ion pairs association complex, any volume more that optimum value effect to increase diffusion of micelles and decrease the extraction efficiency.

2.7. Effect of temperature

Aqueous solutions 10mL in volume contain 100 µg Ni^{2+}at pH=10 in presence $5X10^{-5}$mL HPAB and 0.5 mL of TritonX-114 heating the solution for different temperature andconstant time and then separated cloud point layer CpL and dissolved in 5ml ethanol and measure the absorbance of alcoholic solution atλ_{max}=660nm against blank prepared at same manner in absence Ni^{2+}ion and the aqueous phase treated according to DMG method according to the general method the result were an in fig 9, 10;

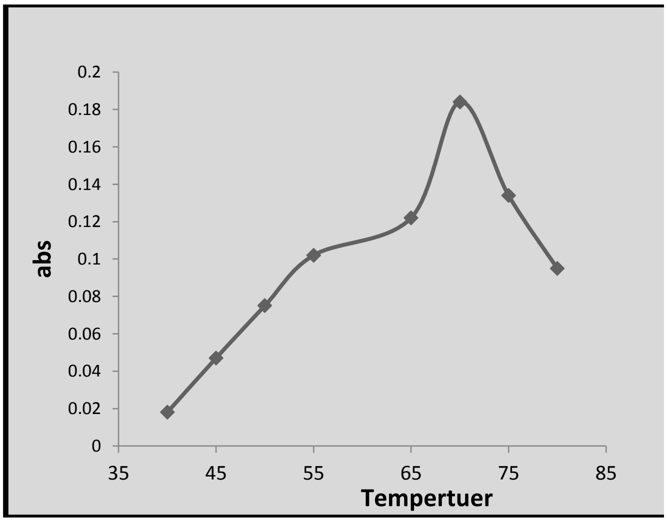


Fig. 9: Effect of temperature on the Formation cloud point layer and Extraction ion pair association complex

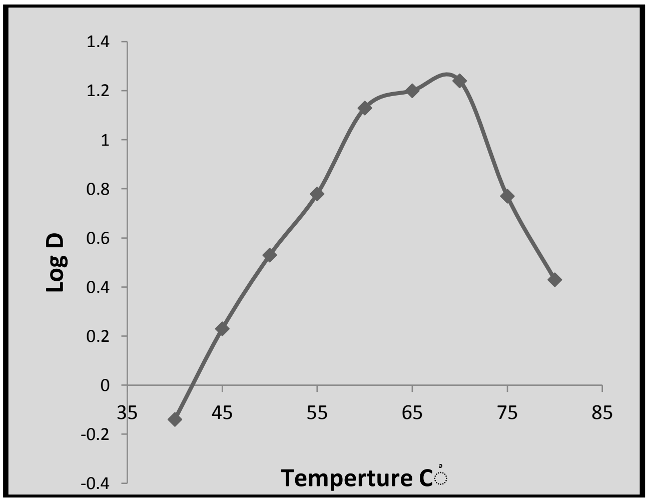


Fig. 10: Effect of temperature on theextraction efficiency and D-value

After calculated extraction constantK_{ex}at each temperature according to the relation below, The result demonstrate in fig (13)

$$K_{ex}= \frac{D}{[Ni^{+2}][HPAB]}$$

The results demomtrate in fig(11)

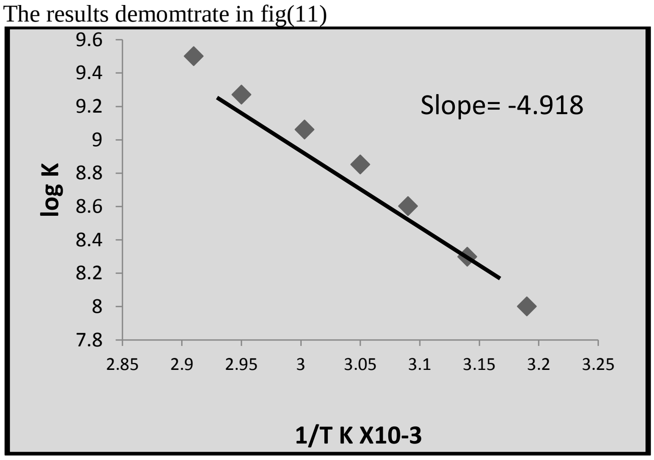


Fig. 11: Kex= f T K

Thermodynamic Data for extraction was;
ΔH_{ex}=0.0921 KJ mol^{-1}
ΔG_{ex}=-59.780 KJ mol^{-1}
ΔS_{ex}=174.55 J mol^{-1}K^{-1}

2.8.Effect of heating time

Aqueous solutions 5ml in volume contain 100μg Ni^{2+}at pH=10 in the presence 0.5 ml TritonX-114 and 5X10^{-5}HPAB heating these solution at 70°c for different time while formats cloud point layer CpL complex the worle as in the general method the result are illustrated in fig (12,13);

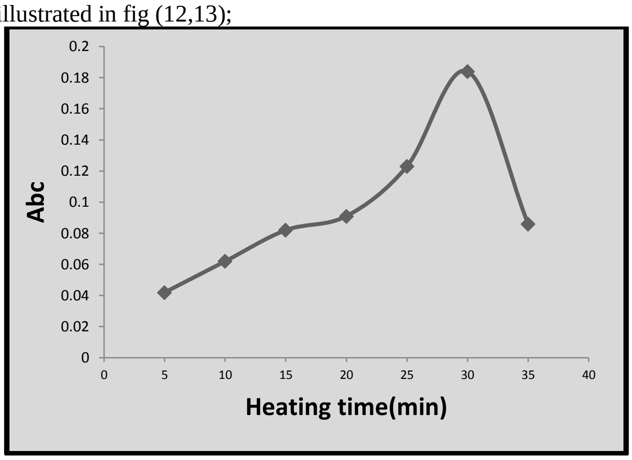


Fig. 12: Effect of heating time on Formation cloud point layer and extraction

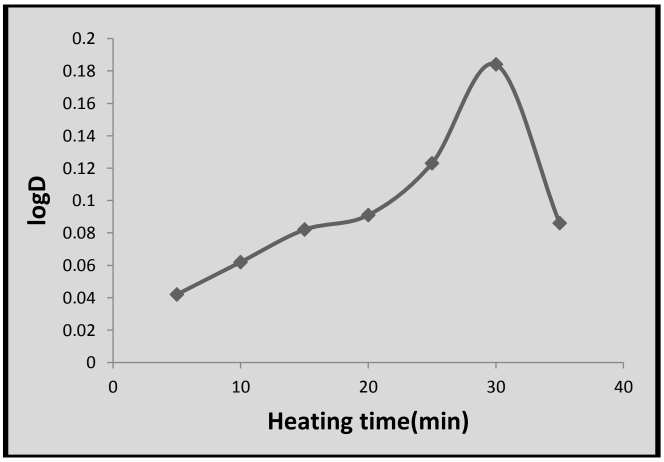


Fig. 13: Effect of heating time onextraction efficiency and D-value

The result shows optimum heating time was 30(min.) at this time we get higherkinetic energy for formation and stability cloud point layer for extraction at this time reach the favorite equilibrium to formation CpL with best dehydration and micelles aggregation heating time less than optimum not allow to formation cloud point layer with good properties so that heating time more than optimum effect increase diffusion of micelles and decrease CpL formation

2.9. Effect of electrolyte

According to the general method extracted Ni^{2+}ion at optimum condition and in the presence different concentration of many electrolyte in a series aqueous solution for each electrolyte the results were an in fig 16,17;

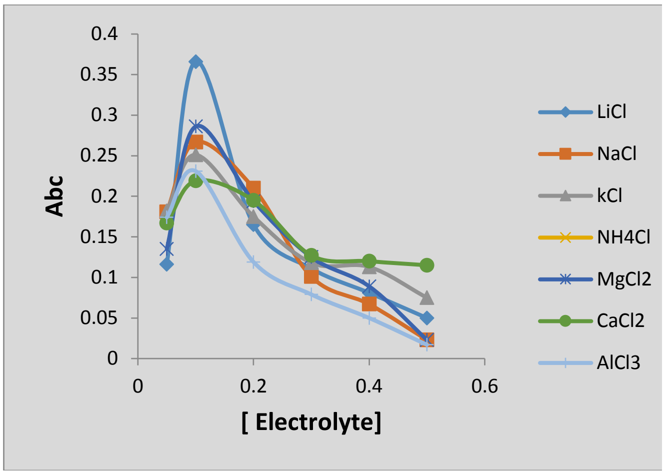


Fig. 15: Effect electrolyte on complex Formation

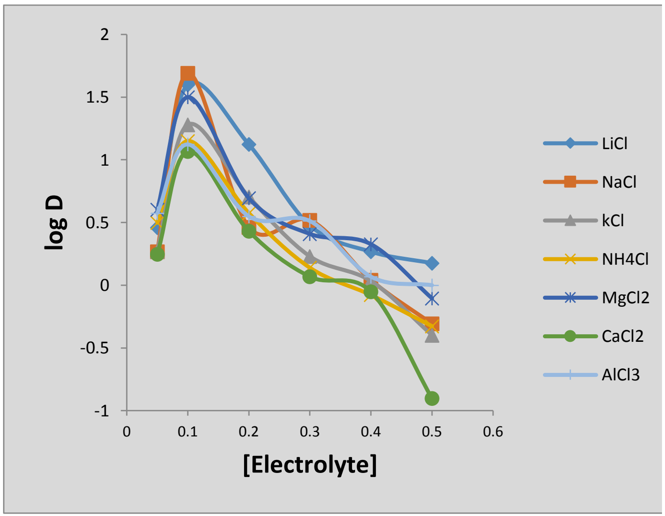


Fig. 16: Effect electrolyte on extraction efficiencyAnd D-Value

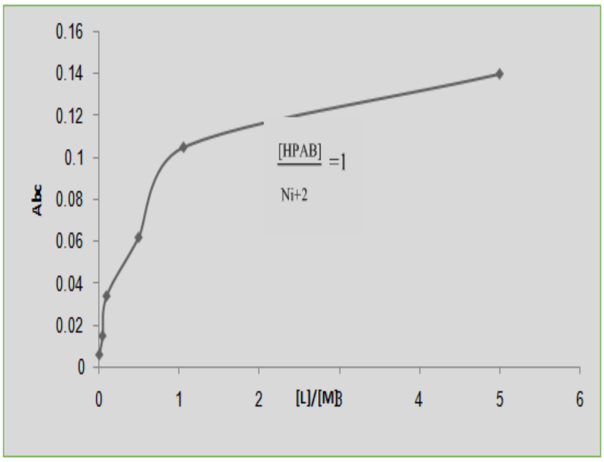


Fig. 17: Mole ratio method

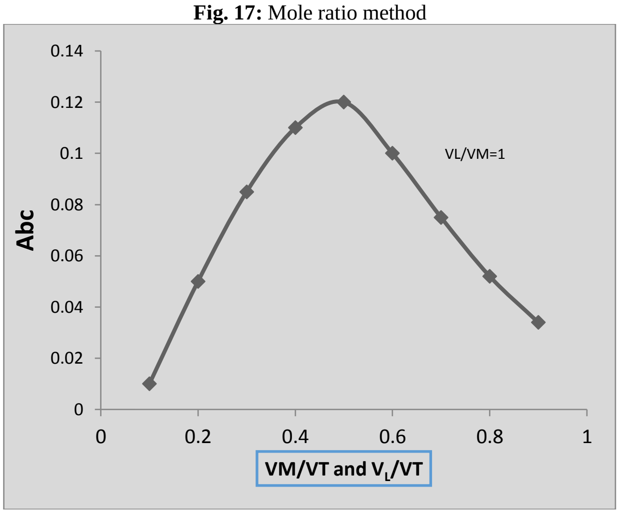


Fig. 18: Continuous variation method(Job method)

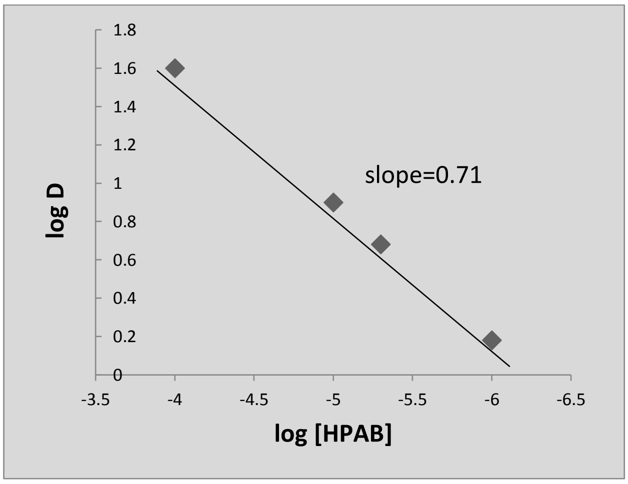


Fig. 19: Slope analysis method

The results shows the presence of electrolyte in aqueous solution effect to increase extraction efficiency because the electrolyte help to withdrawing the water from the hydration shell of the metal ion $Ni^{2+}$and destroy the hydration shell so that help to increase dehydration to increase aggregation of micelles and give CpL with good properties to give good extraction for ion pair complex

2.10. Stoichiometry

In order to determine the more probable structure of ion pain complex extracted into cloud point layer (CpL), used four spectrophotometric method with in mole into, continuum variation method, slope analysis method and slope ratio method the results are illustrated in figure (18-22).

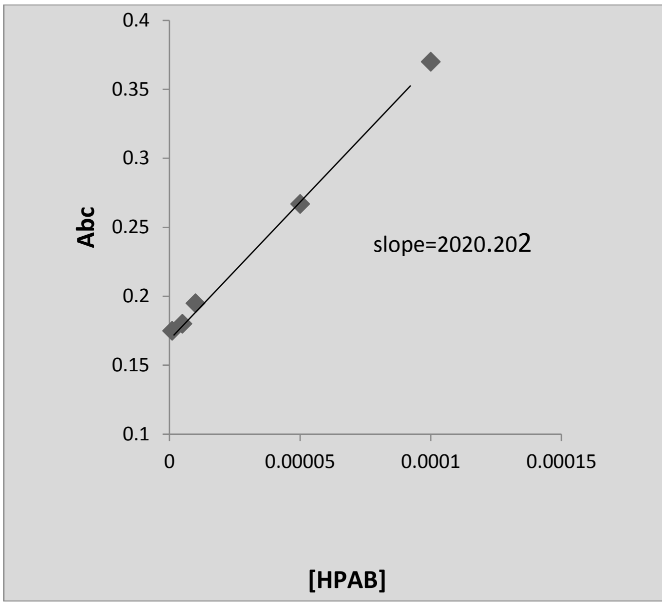


Fig. 20: Slope ratio method of HPAB concentration

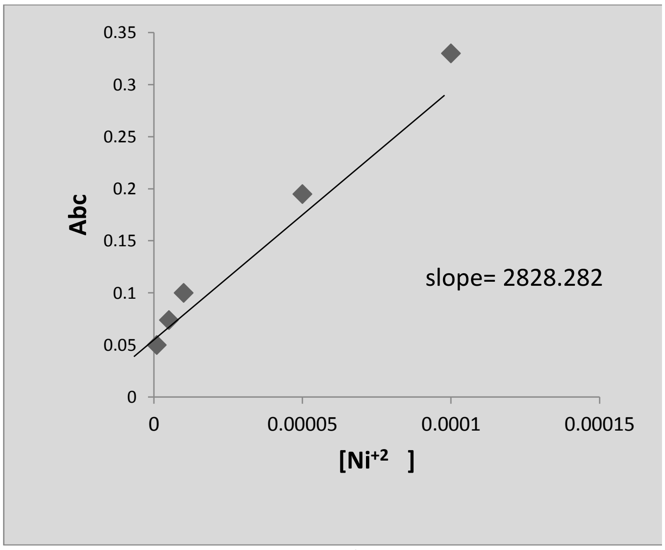
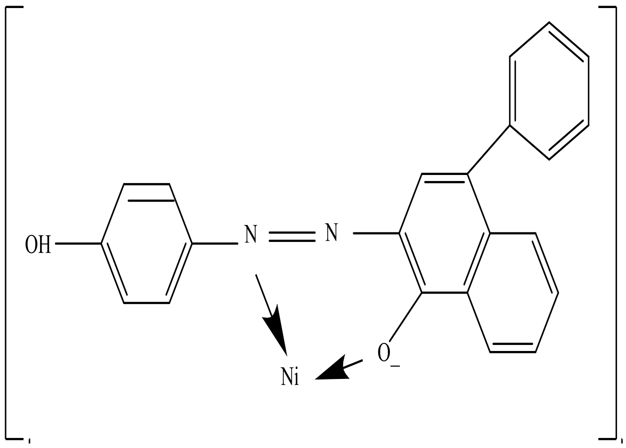


Fig. 21: Slope ratio method of Ni+2 concentration

2.11. Concentration

$$\frac{2828.282}{2352.352} = 1.2 \text{-} \sim 1$$

From these four spectrophotometric method we conclude the more probable structure of ion pair association complex extracted was



2.12. Interference effect

Table 1: Extracted Ni^{2+} from 10mL according to the general method at optimum condition and in the presence 0.01m from foreign metal cations the results are illustrated

Interferences	Abs.λ_{max}=660	D	E%
Co^{2+}	0.019	0.818	44.99
Fe^{3+}	0.036	1.326	5701
Cu^{2+}	0.088	3.167	76.00
Hg^{2+}	0.156	7.0	82.5
Zn^{2+}	0.102	3.444	77.50
Ag^{+}	0.077	1.50	60

The result shows there in an interference for all metal catoin with Ni^{2+}ionextraction, so these ions give different interferences at optimum conditions for extraction Ni^{2+}ion according to different ability to formation ion pair association complex with HPAB at then condition.

2.13. Effect of anion

A series of aqueous solutions contain 100µg Ni^{2+}ion at pH-10, 0.1M NaCl, 0.6mL Tritonx-114, $5X10^{-5}$m HPAB in the presence 0.1M of different anion, then complete the work as in the general method, the results are illustrated in Table (2)

Table 2: Effect of Onion on Extraction Efficiency

Onions	Abs	D	E%
Carbonate	0.246	11.5	92
Oxalate	0.172	7.0	20
Permanganate	0.068	0.471	0.693
Perchlorate	0.178	6.69	86.99
Nitrate	0.156	3.35	77.01
Sulfate	0.169	2.704	73.00

The result are illustrated that the presence each one of the onions give decline in the extraction efficiency of Ni^{2+}ion because these onion effect as masking gents prevent the binding with HPAB and formation ion pair association complexes with different effect according to it's different behavior in aqueous solution

2.14. Spectrophotometricdetermination

In order to determination Nickel (II) in different sample by application the general method at optimum conditions and preparing calibration carve for this purpose as in fig (22)

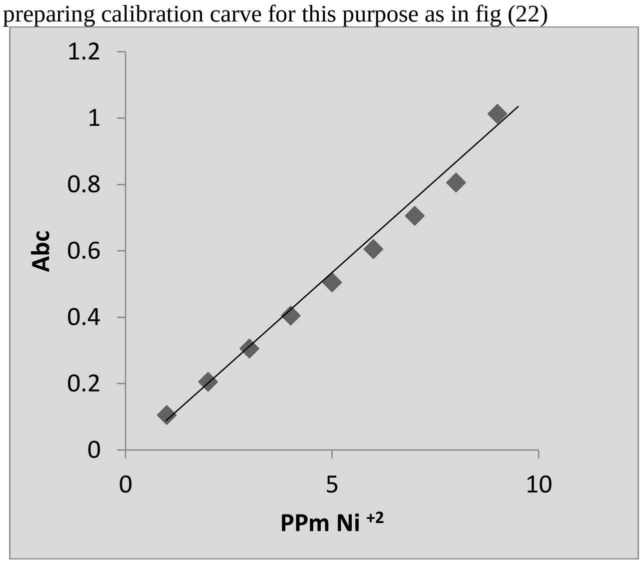


Fig. 22: Calibration curve for spectrophoto metric determination of nickel (II)

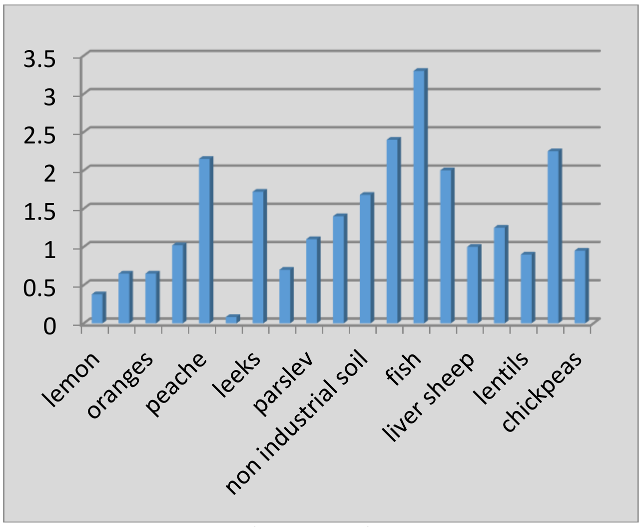


Fig. 23: Application of Nickel(II) of environmental samples

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