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Address:
Iraq-Najaf-University of Kufa
Box(190)

www.Edu_girl.Kuiraq.com

E-mail:Edu_grils@kuiraq.com

Cloud point extraction with Solvation Method for Separation and Determination of Iron (III)

Faris H. Hayder[1] and Shawket K. Jawad[2]

1 Ministry of Education , Al-Mustafa Intermediate, School for Basic Education

2 Chemistry Department, College Of Education for Girls, Kufa university-Najaf-Iraq

Email [1]: farissiraf3@gmail.com

Email [2]: shawkat.alshakarchi@uokufa.edu.iq

Abstract

Cloud point extraction (CPE) method joined with Solvation method used for Separation and Determination Iron (III), the study shows up higher extraction efficiency for Iron (III) we got it at optimum conditions, 100 µg Fe^{3+} in 10 mL aqueous solution, (1×10^{-3} M) $NaNO_3$, (1×10^{-4} M) Erythrosine, 0.5 ml Triton X-100 and heating of 85 ℃ for 15 minutes, as well as solvation species gave maximum absorbance at λmax = 543 nm, in addition to some effective experiments.

Keywords: *Iron (III), cloud point extraction, Triton X-100, dyes, Solvation method*

Introduction

Applied cloud point extraction studied use new complexing agent by reaction Fe(II) with 7-(1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1H-pyrazol-4-ylazo)-8-hydroxyquinoline-5-sulphonic acid in pH 4.5, which extracted with Triton X-114, The ascorbic acid reduction Fe(III) ion to Fe(II) ion. Procedure CPE have detection limit value 1.20 ng mL^{-1}, procedure has been studied some interference, applied CPE procedure given high efficiency.[1]

Ion pair complex Joined with cloud point extraction by using liquid ion exchange iron(III) ion pair complex formation with (4-hydroxyphenylazo)-4-Benzennaphthol in 0.3 M HCl acid in present 100 µg of Iron(III) ion per 10 mL which extracted with 0.5 mL of TritonX-100, electrolyte and interferences was studies and technique include determination Fe(III) in different samples.[2]

Methodology of liquid ion exchange was using for separates and extraction to determination Iron (III), the ion pair association complex was extraction in optimum condition by applied CPE technique was shows heating temperature in 90 °C for 15 minutes.[3]

Iron (III) ion extraction by used Cloud point extraction technique from urine samples and the samples urine measured in Flame atomic absorption, the reagent was used Benzidine and surfactant Triton X-114 the studies include influence of interferences.[4]

Organic reagent 2-methyl-8-quinolinol was derivatives from 8-quinolinol which used in the cloud point extraction procedure to extracted and determine Iron(III) from riverine water, this procedure was applied first through cloud point extraction then measured amount of Iron(III) by a graphite furnace atomic absorption spectroscopy.[5]

A laboratory experiment is described in which students determine the concentration and speciation of iron in beer samples using cloud point extraction and absorbance spectroscopy. The basis of determination is the complexation between iron and 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP) as a colorimetric reagent in an aqueous micellar solution followed by cloud point extraction for preconcentration.[**6**]

Non-ionic surfactant, Triton X-114 was used in The application of cloud point methodology, the Cations were preconcentration heavy metal ions, complexation with 8-quinolinol in an aqueous, were 1×10^{-3} M from 8-quinolinol at pH 7.0 given higher a preconcentration factor and the detection limits were 0.8 – 15 μg/L.[**7**]

Spectrophotometric determination of Iron(III) ion and Mercury(II) ion in real samples by used thiazolylazo as organic reagent [methyl phenyl thiazolyl azo]-3-methyl-4-methoxy-2-naphthol (MPTAN), this method joined with the Cloud point extraction included nonionic surfactant TritonX-100.[**8**]

Organic compounds were extraction and preconcentration with new procedure were extraction it from solid matrices (soil, coal) The factors of extraction efficiency and percent recovery for solutes phenols and polycyclic aromatic hydrocarbons were critically assessed.[**9**]

Procedure depend on flame atomic absorption spectrometry and cloud point extraction procedure system, were used as selective determination of trace iron in environmental samples using organic reagent N,N-(2,2-(ethane-1,2-diylbis(oxy))bis(ethane-2,1-diyl))bis(2-chloroacetamide)(EDBOCA) in pH 5.0 for only Fe(II) and Fe(III) ions, procedure was based on the complexation between Fe(III) ions and organic reagent EDBOCA in Triton X-114.[**10**]

Extraction method contains Triton X-114 were used in nano-determination for carbendazim (MBC) pesticide in present real samples. Methods including oxidation Fe (III) ions and with Fe (III) ions with MBC pesticide in present acidic medium. The spices were extracted into Triton X-114.[**11**]

Experimental

Apparatus

A Shimadzu double beam UV-visible spectrophotometer model uv-1700 (Japan) equipped with 1cm cell path length for study absorption spectra of complexes formed. But absorbance measurement was performed with Shimadzu single beam UV-visible model 100.02 spectrophotometer (Japan).

Reagents and Materials

All reagent and chemical material used as is from the supplier company without additional purification, the standard stock solution for reagents and samples has been prepared by using Distilled water. The stock solution of Fe(III) 1 mg/mL prepared from $FeCl_3.6H_2O$ by dissolving 0.121 gm in 100 mL of distilled water in volumetric flask and prepared Organic reagent Erythrosine solution with concentration was $1×10^{-2}$ M by dissolving 0.8798 gm in 100mL distilled water in the volumetric flask.

Comprehensive method

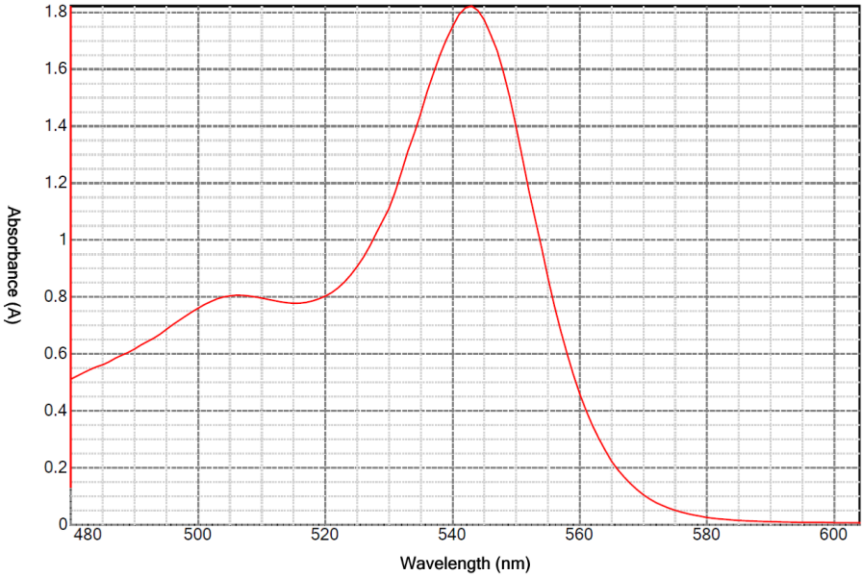
10 ml aqueous solution contain suitable and appropriate concentration of $NaNO_3$, Erythrosine with fixed quantity of Iron(III) ion and favorite volume of TritonX-100, heat the solutions in electrostatic water bath at optimum temperature and time until formation Cloud point layer (CPL), then separate the CPL from the aqueous solution and Dissolved in 5mL ethanol , the measure the absorbance of alcoholic solution at λ_{max} of Solvation species formed and extracted against blank prepared at the same manner without Iron(III) ion, as well as the aqueous solution treated according to Thiocyanate KSCN spectrophotometric method [**12**], and Return to calibration curve in Fig (2) to determine the remain quantity of Fe^{3+}ion in aqueous solution after extraction, afterward abstraction the remain quantity from the total amount in aqueous solution before extraction to determine the transfer quantity of Iron(III) ion into cloud point layer as solvation species, at latter calculate distribution ratio (D) of extraction as in relation below.

$$D=[Fe^{3+}]_{CPL} / [Fe^{3+}]_{aq}$$

Results and Discussion

Spectrophotometric study

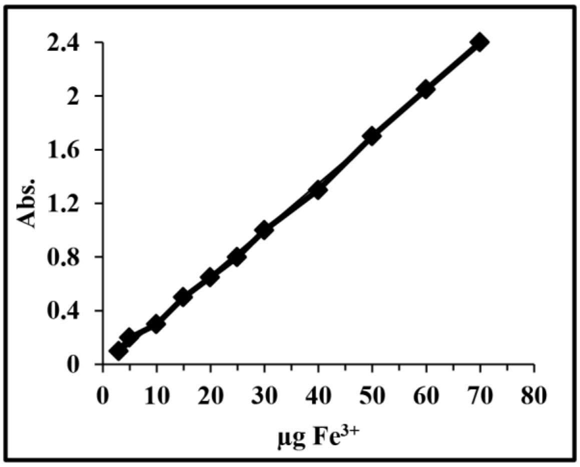
Spectrophotometric study according to joined method come out the wave length for maximum absorbance for solvation species by use Erythrosine as organic reagent was $\lambda_{max} = 543$ nm, as in Fig (1).



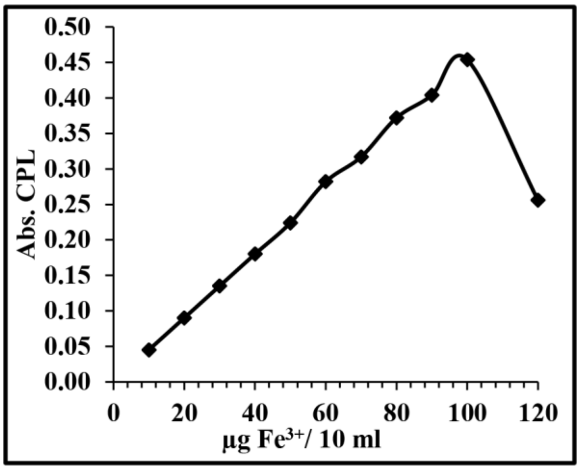
Fig(1): UV-Vis Absorption spectrum for Solvation species of Iron(III) with Erythrosine

Effect of metal ion Concentration

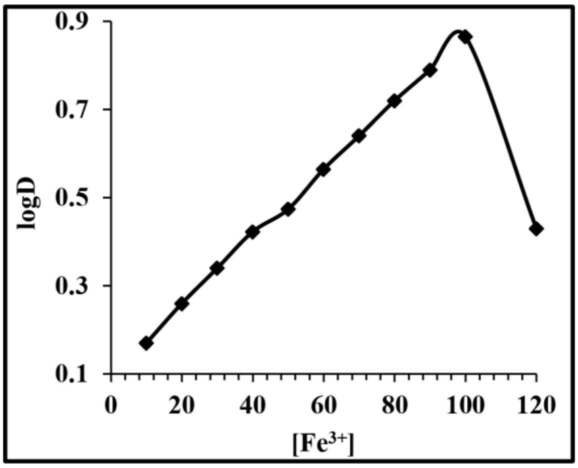
Formulate sequence 10mL aqueous solutions with rising microgram (µg) of Fe^{3+} ion, in existence of 1×10^{-3} M of sodium nitrate $NaNO_3$, 1×10^{-4} M of Erythrosine and 0.5 mL surfactant TritonX-100. Heat the solutions in electrostatic water bath at suitable temperature and time to formation CPL, then separated CPL from aqueous solution and dissolved it in 5mL ethanol and measured the absorbance of alcoholic solution at $\lambda_{max}= 543$ nm against blank prepared at the same manner without Fe^{3+}ion, as well as treated the aqueous solutions according to Thiocyanate KSCN spectrophotometric method and Calculate Distribution ratio (D) as in comprehensive method, the results was as in Fig(3,4) .



Fig(2) : Calibration Curve for determination Fe^{3+}ion in aqueous solution



Fig(3) Increasing solvation species formal and stability with increase Fe^{3+} ion Conc.



Fig(4): Increasing extraction efficiency, with increase Fe^{3+} ion concentration

The Results appear 100 µg Fe^{3+}/10 mL aqueous solution was the optimum concentration which was gave higher extraction efficiency and higher concentration of solvation species extracted into CPL, as well as unfold concentration of Fe^{3+} ion in aqueous solution was a thermodynamic data control the thermodynamic equilibrium to formation solvation species. And effect on the rate of thermodynamic formation equilibrium.

Salting out Concentration

Formulate numerous 10mL aqueous solutions consist 100 µg Fe^{3+} ion and disparate concentration of salting out $NaNO_3$, at the range (1×10^{-6}-1×10^{-2}) M, with 1×10^{-4} M Erythrosine and 0.5 mL of surfactant TritonX-100. Heat these solutions in electrostatic water bath at suitable temperature and time to form CPL which is separated from aqueous solution and dissolved in 5 mL ethanol, and then complete the experiment as in Comprehensive method, the results were as in Fig(5,6).

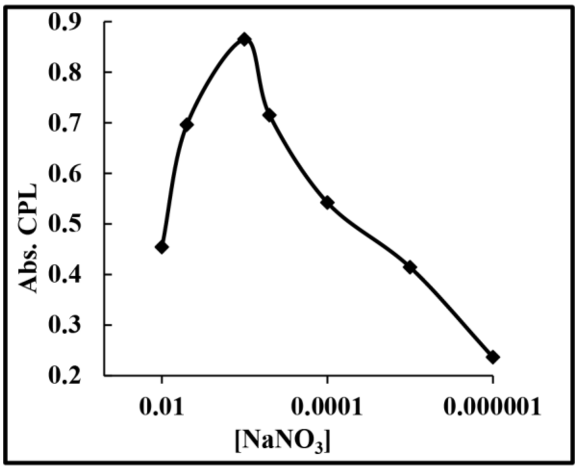
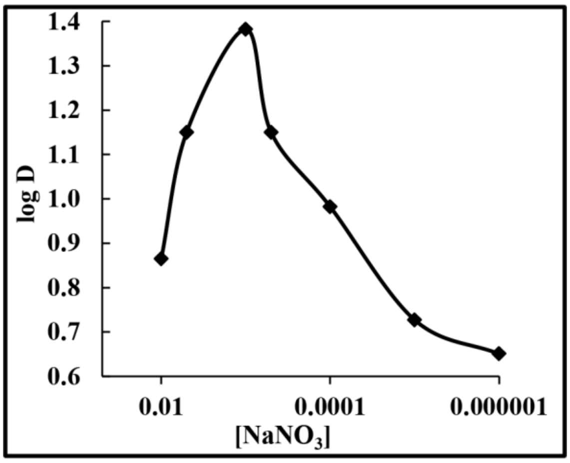


Fig (5): Effect of salting out Conc. on formation and stability of Salvation species

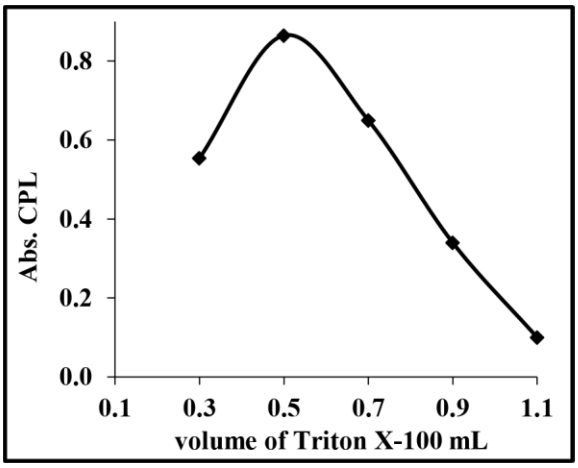


Fig(6): Effect of salting out Conc. on extraction efficiency and D value

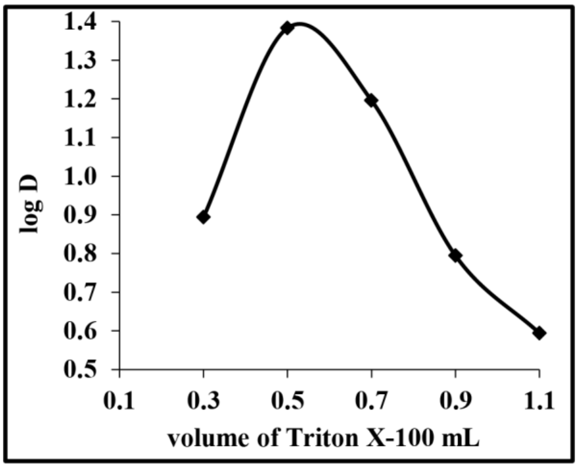
The results show 1×10^{-3} M was the optimum Conc. of Salting out $NaNO_3$, which gave higher extraction efficiency, and maximum concentration of solvation. Species extracted into CPL because it affect as a thermodynamic data control the rate of thermodynamic equilibrium , any concentration of $NaNO_3$ les or more Than optimum value brought decline in extraction efficiency and rate of thermodynamic equilibrium.

Surfactant Volume

Prepare a number of aqueous solutions 10 mL in volume accommodate 100 µg Fe^{3+} ion, 1×10^{-3} M salting out $NaNO_3$, 1×10^{-4} M Erythrosine and different volume of surfactant TritonX-100. Heat These solutions in electrostatic water bath at suitable Temperature and time to form best CPL, then separated this layer and dissolved in 5 mL ethanol and Complete the experiment as in Comprehensive method, the results were as in Fig (7,8).



Fig(7): Effect of surfactant Volume on quality of Cloud point layer formed

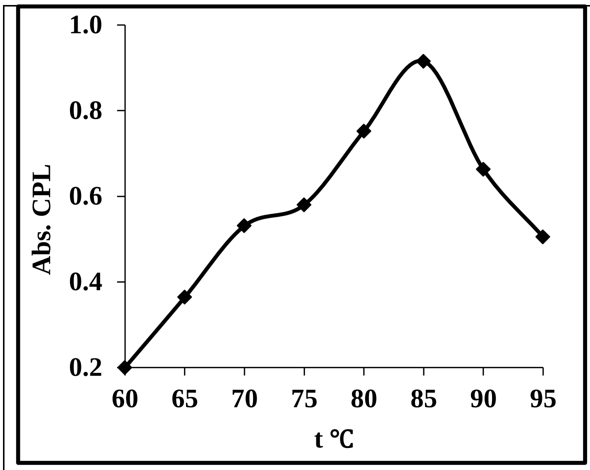
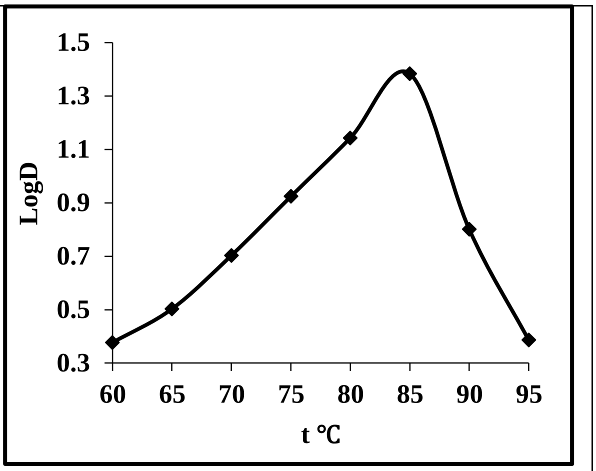


Fig(8): Effect of surfactant volume on extraction efficiency and D value

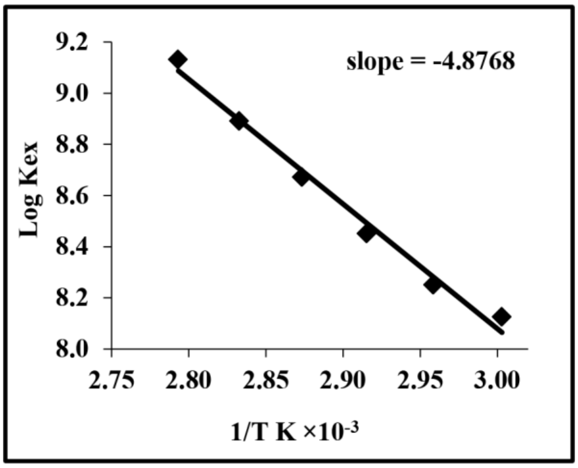
The results materialize 0.5 mL of TritonX-100 was optimum volume delivered best extraction efficiency because this volume formed best CPL to extracted maximum Concentration of solvation species.

Temperature effect

many aqueous solutions 10 mL in volume have to prepared each one contain 100 µg Fe^{3+} ion , 1×10^{-3} M salting out $NaNO_3$, 1×10^{-4} M Erythrosine and 0.5 mL TritonX-100. Heat these solutions in electrostatic water bath at dissimilar temperature for fixed time to formation CPL, then complete the experiment as in Comprehensive method the results were as in Fig (9,10).

	
Fig(9): Effect of temperature on formation cloud point layer	Fig(10): Effect of temperature on extraction efficiency and D values

The results above show 85 ℃ was the optimum temperature to give higher extraction efficiency, after calculate extraction constant Kex at each temperature the results were as in Fig(11).



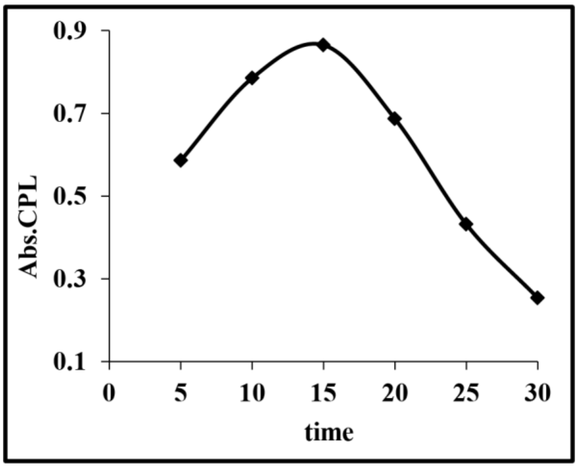
Fig(11): Kex=f temperature

Thermodynamic data for extraction Fe^{3+}ion as solvation species according to this Compact method were.

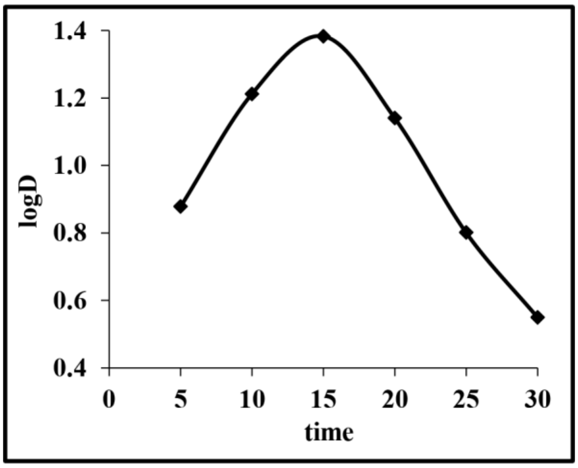
ΔHex= 0.0934 kJ.mol^{-1}
ΔGex = -68.7332 kJ.mol^{-1}
ΔSex = 175.4931 J.mol^{-1} k^{-1}

Heating time effect

Extracted 100 µg Fe^{3+}ion from many aqueous solution 10 mL in volume Contain 1×10^{-3} M $NaNO_3$, 1×10^{-4} M Erythrosine and 0.5 mL TritonX-100. Heat these solutions in electrostatic water bath at 85 ℃ for different time, and complete the experiment as in Comprehensive method, the results were as in Fig(12,13).



Fig(12): Effect of heating time on formation cloud point layer

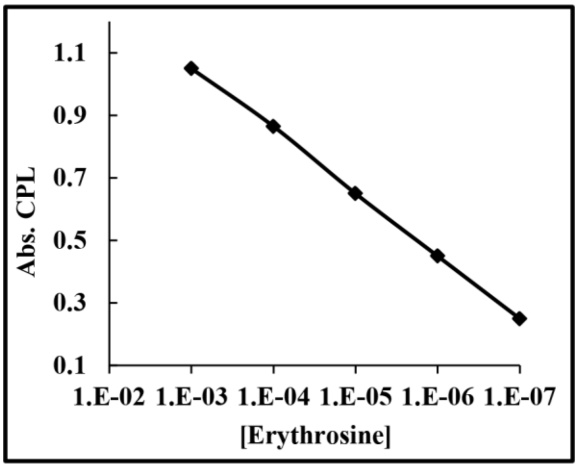


Fig(13): Effect of heating time on efficiency and D-value

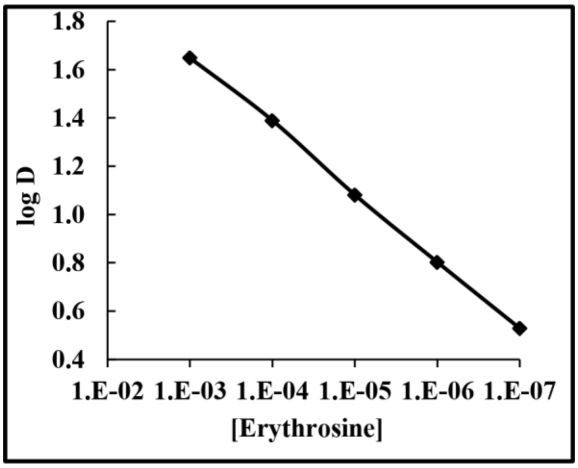
The results show 15 minutes. was the optimum heating time which is gave higher extraction efficiency because it is help to formation best cloud point layer, as well as represent the Kinetic side of extraction method.

Organic Reagent Concentration

A series of aqueous solutions 10 mL in volume have to prepared and enclose 100 µg Fe^{3+} ion, 1×10^{-3} M $NaNO_3$, 0.5 mL TritonX-100, with Different concentration of Erythrosine, heat these solutions in electrostatic water bath at 85 ℃ for 15 minutes down to Formation CPL, which separated and complete the experiment as in comprehensive method, the results were as in Fig(14,15).



Fig(14): Effect of Erythrosine Conc. on extraction stability of solvation species

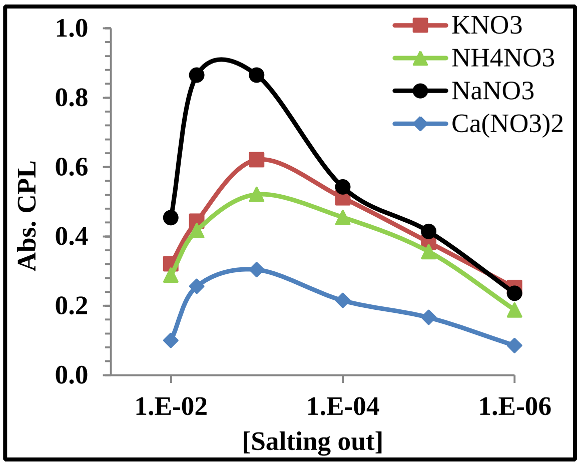


Fig(15): Effect of Erythrosine Conc. on extraction efficiency and D-value

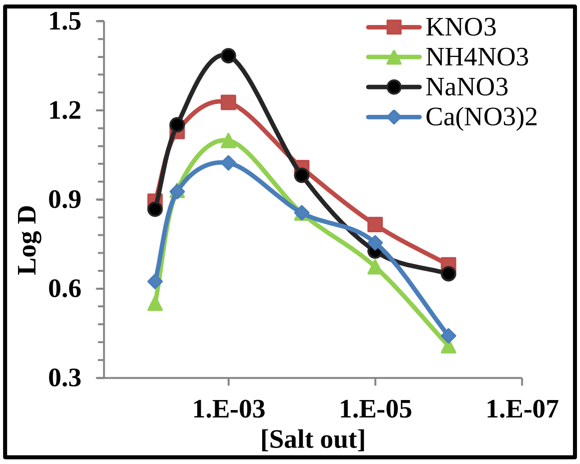
The results demonstrate increasing concentration of solvation species formation which confirms organic reagent Erythrosine is thermodynamic data control maybe extracted into CPL, thermodynamic equilibrium of formation solvation species.

Salting out kind

Three series of aqueous solutions 10 mL in volume was prepared contain 100 µg Fe^{3+} ion, 1×10^{-4} M Erythrosine, 0.5 mL TritonX-100 and Different concentration of salting out NH_4NO_3, KNO_3, $Ca(NO_3)_2$ respectively for the three series, heat all solutions in electrostatic water bath at 85 ℃ for 15 minutes until formation CPL which separated and dissolved in 5mL ethanol, then complete the experiments as in comprehensive method the results were as in Fig(16,17).



Fig(16): Effect of Salting out kind on conc. and stability of solvation species



Fig(17): Effect of Salting out kind on extraction efficiency and D values

The results shows all Salting out give higher extraction efficiency at 1×10^{-3} M, that is mean this concentration suitable for nitrate concentration to give the best thermodynamic equilibrium of solvation species formation and the higher extraction efficiency differ with different salting out as in the sequence $NaNO_3 > KNO_3 > NH_4NO_3 > Ca(NO_3)_2$.

Effect of organic Reagent Kind

Three aqueous solutions 10 mL in volume have to produce, each one contain 100 µg Fe^{3+} ion, 1×10^{-3} M $NaNO_3$, 0.5 mL TritonX-100 and 1×10^{-4} M from different organic reagent. Heat these solutions in electrostatic water bath at 85 ℃ for 15 minutes. Till provide CPL which is separated and dissolved in 5 mL ethanol. Then taken an absorption spectrum for alcoholic solution against blank prepared for each organic reagent without Fe(III) ion, as well as treated the aqueous phase as in comprehensive method the results were as in Table (1).

Table 1: effect of organic reagent on extraction efficiency

Organic Reagent	λ_{max}	**D**
Rhodamine 6G	553 nm	2.100
Salicylic acid	295 nm	2.817
2,4-dimethyl-3-pentanon	292 nm	7.737

Different organic reagent gave different extraction efficiency and different rate of thermodynamic equilibrium of formation and stability of solvation species, this different behavior go to the structure of organic reagent and its ability to formation more stable solvation species.

Interferences

Many aqueous solutions 10 mL in volume contain 100 µg Fe^{3+} ion, 1×10^{-3} M $NaNO_3$, 1×10^{-4} M Erythrosine, 0.5 mL TritonX-100 and 1×10^{-3} M from different interferences, head this solutions in electrostatic water bath at 85 ℃ for 15 minutes. Until formation CPL, which is separated and dissolved in 5 mL ethanol and complete the experiments an in Comprehensive method, the results were as in Table (2).

Table 2: effect of interferences on extraction efficiency

Interferences ion	**Abs.aq**	**D**
Co^{2+}	0.564	18.79
Cd^{2+}	0.433	12.13
Cu^{2+}	0.361	8.49
Pb^{2+}	0.214	5.18

All these nitrate electrolyte salts, behave as interference in extraction method and affect to decline extraction efficiency and rate of thermodynamic equilibrium of formation solvation species, because they foreign ions Fe^{3+} ion participation to formation solvation species for each metal ion in these nitrate salts side by side with Fe^{3+} ion and affect to decline concentration of solvation species of Fe^{3+} ion because decrease the concentration of all materials in aqueous solution from optimum value.

Conclusions

1- Method of formation solvation species depend on the suitable concentration of nitrate ion NO_3^- but not high concentration.

2. The metal in nitrate electrolyte salt effect on the formation and stability of Solvation species

3. Ketones and esters kinds it plays an important role in formation and stability of Solvation species.

4- Structure of organic Reagent may be increase the hydrophobicity of solvation species and increase partition to cloud point layer to increase extraction efficiency.

5- Surfactant tweens not favorite for extraction Solvation species because Cloud point layer formed has small hydrophobic surface area.

6- Probably used this Compact method for determination Fe^{3+} ion in different samples.

Recommendations

1. It is possible to use this Compact method to separation, preconcentration and determination of Transition metal elements and lanthanides and Actinide.

2. Can be used drugs and vitamins which have Ketone and Ester structures in this Compact method as organic reagent to form solvation species.

3. When we are used this compact method the determination metal ion must be used masking agents to prevent happen any interference.

4- Can be Extraction and determinate a metal ion by Solvation method after dissolved the organic reagent in immiscible organic solvent and separate solvate species to organic solvent.

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