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Cloud Point Extraction with Liquid Ion Exchange for the Separation and Determination of Magnesium (II) as anion

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Abstract

Liquid Ion Exchange joined with Cloud point extraction methodology was used for the separation of Magnesium (II) from aqueous and determine whereas 10 mL aqueous solution that contains 50 µg Mg^{2+} ion is complex with 1×10^{-3}M 8-Hydroxy quinoline (8-HQ) at a suitable basic medium it well give higher extraction efficiency at optimum conditions, needs heating the aqueous solution in suitable temperature degree for enough time to form a cloud point layer (CPL). Therefore, the optimum conditions that yielded the good CPL have a small aggregation volume which is appropriate for continuing the ion pair association Complex between Magnesium ion and 8-Hydroxy quinoline.

Introduction

The cloud point extraction method has been widely used in recent years due to the high susceptibility of this technique and its high efficiency in extracting many elements, as well as the diversity of surfaces that can be used well and meet the desire of the chemical analyzer to obtain a quantitative extraction from many different elements, and from these elements, Cr(VI) by using Triton X-100 as an active surfactant [1]. It was used to extract Fe (III) from different geological samples [2], Fe (III) ions after complexation reaction with organic reagent Zincon at pH [3]. It was also used to remove nanoplastics (NPs) pollution from environmental waters by using Triton X-45 as an active surfactant [4] which can be used to determine many different compounds such as alkaloids, medicine drugs and organophosphorus from complex matrices [5]. Besides, it was used, for the first time, to detect Cu (II) after the formation of a complex with complexing agent compound N-benzamido-N'-benzoylthiocarbamide[6]. Co (II) can be separated and extracted by applying this technique to separate and determine the metal ion from the real samples in the presence of Triton X- 100 as an active surfactant [7]. The extraction and determination of phosphate compounds at room temperature can be achieved by applying cloud point extraction procedures from water samples [8]. This procedure has used Triton X-114 to produce a suitable cloud point layer that was used to determine the Level of Nanoparticles Ag_2S and ZnS in Environmental Waters [9].

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Oxine anion is the main organic reagent used to form complex after being combined with Magnesium (II) and, afterward, being electrostatically bound with Rhodamine-B^+ to give an ion-pair association complex. Then, this complex was to be separated into a Cloud point layer (CPL) to determine Magnesium (II) spectrophotometrically at a wavelength of maximum absorbance that was 648nm, in addition, to using Triton X-100 for the sake of separation and determination.[10]. The onium method was the method this study depends on to use with cadmium (II) for the processes of separation and determination of different samples. The optimum conditions for application have illustrated the maximum absorbance for species at a wavelength of 260 nm. The Onium species needed 0.5M from HCl to form Onium species in the aqueous solution which contained 50µg/5mL of Cd^{2+} in presence of 2,4-dimethyl-3-pentanone as an organic reagent.[11]. Trace amounts of magnesium and silver were extracted and preconcentrated by used cloud point extraction (CPE) method in the first step and the determination was the second step by flame atomic absorption spectrometry (FAAS), at pH 10 with used to Triton X-114 as a non-ionic surfactant, a complexing agent used (MPBIM) to the determination of silver and magnesium in many urines, water samples, and other blood serum samples.[12]

For the measurement of a trace metal ion in various matrices, Triton X-100 was employed as the extractor in the CPE technique, which was used for separation and preconcentration. CPE is one of the “green chemistry” principles that include rushing in the time, low cost and higher preconcentration factors. The advantages process of micelle formation is divided when it is heated to a sufficient temperature as the original solution splits into two phases. [13]. Pre-concentration and extraction of Mg^{2+} ions are done by applying the CPE, this method used 2,4-dimethyl-pentane-3-one as extraction species. The creation of solvated species between the metal ion and organic solvent was done to realize the solvated species extraction to the cloud point layer and the maximum absorbance for the ethanolic solution that was 249 nm. This method needs to use different salts out to provide the extraction efficiency i.e., 0.5M from KNO_3 that gives higher extraction efficiency, and variable concentration of other nitrate salts that have been studied too.[14]

Sodium dodecyl sulphate (SDS) was a micelle medium for the determination of magnesium after complexation with 8-hydroxyquinoline. In this study, the maximum absorbance of the complex was 390 nm. critical parameters, like pH of the solution, affect the concentration of ligand and the accuracy and reproducibility of this method. The method has succeeded and given good accurate values for the determination of magnesium in some drugs and samples of mineral water.[15]. The method depends on the formation of ion-pair complexes that were used for the extraction and separation of two metals, ion Mg (II) and Ca (II), after being combined with EDTA (H_3Y-) to form a complex as (MgHY-) and (CaHY-). The anion transfer of the PCL, after using liquid ion exchanger Cinchonine (CK) and the ion-pair formation of magnesium complex was HCK+; MgHY-. [16]. The enhancement of the extraction efficiency for the Solvation technique has used Acetophenone as an organic agent to separate and determine Zn (II) ion. This technique needs salting-out like $NaNO_3$, for the enhancement of the extraction efficiency in presence of methanol when add to the aqueous solution.[17]

A developed extraction method with the micelle mediated for the formation of the ion association complex by using organic reagent brilliant green in acidic media to form an ion association complex with manganese oxyanion as well as many interferences effects of ions were studied.[18]. Crystal violet was used to form an ion-pair association after forming chloro anions in acidic media used for this purpose HCl; this technique is characterized by a sensitive extraction method and separation chloro anions of Zn (II) ion and Ni (II) ion creating a form of an ion-pair association complex.[19]

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Experiment

1. Chemicals and Reagents

Deionized water was utilized to make the solutions in this investigation. All materials Triton X-100 for analysis, Eriochrome Blake-T ACS reagent (indicator grade), Janus green B, $MgCl_2.6H_2O$, and others were purchased from Sigma-Aldrich and Merck. The $MgCl_2.6H_2O$ which was used to prepare other solutions was diluted with filtered water in a volumetric flask to make a standard solution of Mg (II).

2. Apparatus

A double beam UV-visible spectrophotometer a Biochrom (Biochrom Libra 560 made in Cambridge, UK) was employed for studying the absorption spectra of compounds produced, it is fitted with a 1cm glass cell path length.

3. General method

Preparing 10 mL aqueous solution that contains 50 µg Mg^{2+} ion, 1×10^{-3} M 8-Hydroxy quinoline, 0.05 M NaOH, 1×10^{-4} M Janus green B, 0.5 mL Triton X-100 (TX-100), and heating the solutions in an electrical water bath at 85 °C for 20 minutes (min.) to form CPL. Then, separation CPL from aqueous solution, and measuring the absorbance of ion-pair association complex of Mg2+ is achieved at λmax=365 nm vs. blank which is prepared at following the same way without Mg2+ ion, in order to determine Mg2+ ion residual in the aqueous solution. After extraction by following Eriochrome Blake-T spectrophotometric method,[20] then determine transfers Mg^{2+} ion to CPL as complex and calculate distribution ratio D by using a calibration curve as shown in Figure (1). All the absorbance values of the cloud point layer or the values which distribute ratios D were calculated, and are considered the result of reading three values for each sample that was checked.

Aluminizing – chromizing diffusion coatings are widely used for high-temperature oxidation and hot corrosion protection of turbine blades used in engine hot sections [2]. The pack cementation method is used for the position of protective coatings on the protection against oxidation, corrosion, and damage [4]. At high temperatures, Al and Cr in the coating are oxidized and form a thin Al2O3 and Cr2O3 scale, which works as the diffusion barrier and reduces the oxidizing speed of the base material. The coated elements are placed in closed or half-closed containers and covered with mixture powder, which consists of metals used for deposition (Al and Cr), the halide activating agent, and inactive filler. The coating is fabricated through the reduction of metal-halide vapors on the surface of the base material followed by diffusion in the solid-state between the introduced metal and the substance [5].

$$D=[Mg^{2+}]_{CPL}/[Mg^{2+}]_{aq} \qquad (1)$$

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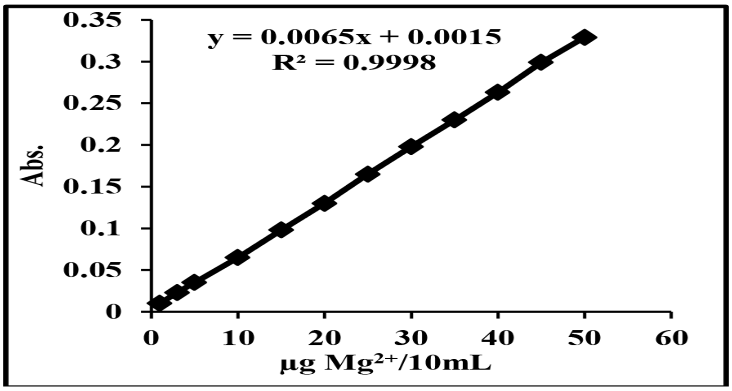

Figure 1Calibration Curve used for determining Mg2+ in aqueous solutions by Eriochrome black-T spectrophotometric method.

Results and Discussion

1. Spectroscopic study

Preparing to 10mL aqueous solution containing 50µgMg^{2+} ion, 1×10^{-3}M 8HQ, 0.3M NaOH, 1×10^{-4}M JGB and 0.5mL Triton X-100 needs heating these solutions in an electrostatic water bath for suitable temperature and time until the formation of CPL. After that, CPL was separated from the aqueous solution and dissolved in 5 mL ethanol, then spectrum for alcoholic Solutions is taken in UV-vis spectrophotometer as illustrated in figure (2).

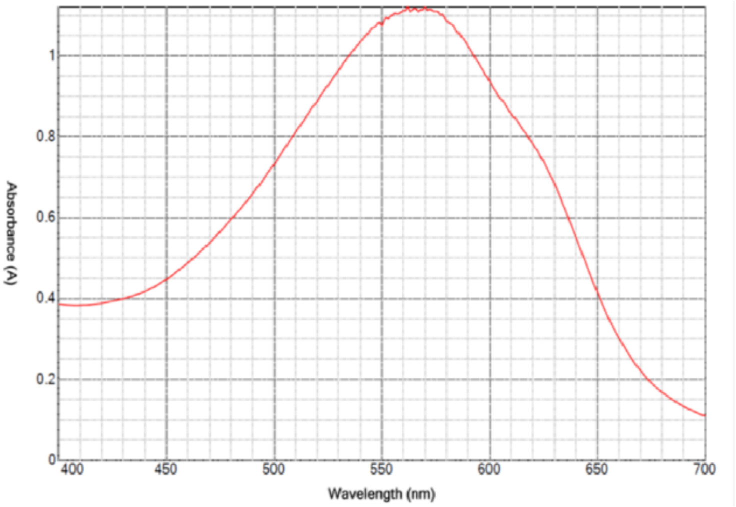

Figure 2 UV-Vis absorption spectrum for ion-pair association Complex

The spectrum clearly shows that the wavelength of maximum absorption for complex extracted was 565 nm.

2. Variation 8-hydroxy quinoline Concentrations

Creating aqueous solutions in 10 mL contains 50 µg of Mg^{2+} ion with different concentrations (Con.) of 8-HQ, 0.3 M NaOH, 1×10^{-4} M JGB and 0.5 mL TX-100, needs heating these solutions in an electrical water bath at 85 °C for 20 min., then, the experiment is completed as shown in the general method illustrated in figure (3).

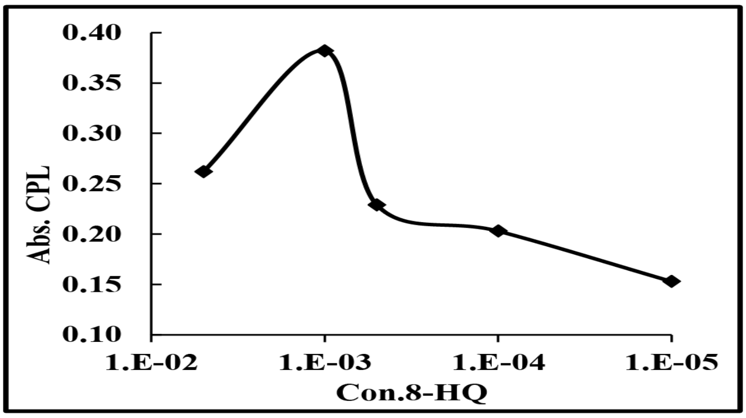

Figure 3 Effect of 8-HQ Con. on the development of the Mg2+ ion complex and its stability.

The Results show up 1×10^{-3}M, 8-HQ was the optimum concentration, in a way that gives higher extraction efficiency, and this concentration has contributed to reaching a higher rate of a thermodynamic relation to form an ion pair association Complex.

3. Variation NaOH Concentration

Preparing a series 10 mL aqueous solutions containing 50 µg Mg ion, 1×10^{-3} M 8-HQ, and different concentrations of NaOH, 1×10^{-4} M JGB and 0.5 mL TX-100, needs heating these solutions in an electrical water bath at 85 °C for 20 min., until the formation of CPL. Afterward, separate the CPL from the aqueous solution, then complete the labor according to the general method illustrated in figure (4) that shows the results.

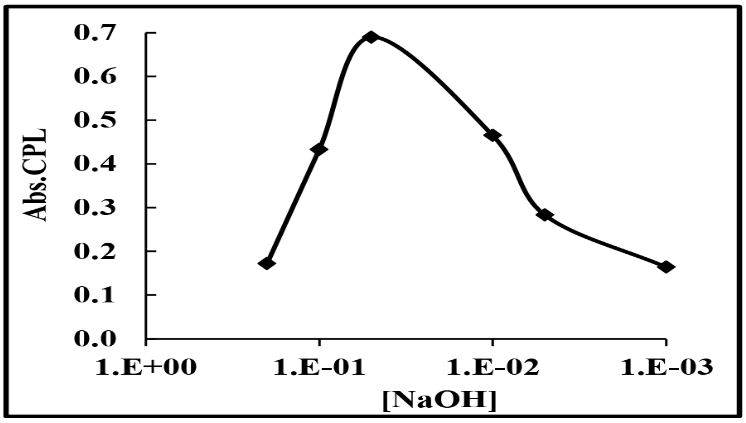

Figure 4 Effect of NaOH Con. on the development of the Mg2+ ion complex and its stability

The results demonstrate 0.05 M which was the optimum concentration of NaOH to give higher extraction efficiency and favorite rate of thermodynamic equilibrium that forms an ion pair association complex. Whereas at this NaOH, concentration produces maximum Con. of oxine (OX^-) to give a higher concentration of anion complex of Mg^{2+}, as well as a higher rate of liquid anion exchange to form a maximum concentration of ion-pair that associates complex extracted into cloud point layer.

4. Variation Mg Ion Concentration

Preparing many 10 mL aqueous solutions contain a rising quantity of Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH, 1×10^{-4} M JGB, and 0.5 mL Triton X-100. Then, these solutions are heated in an electrical water bath at 85°C for 20 min. until the

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formation of CPL. CPL is then separated from aqueous solutions and the experiment is completed according to the general method illustrated in figure (5) that shows the results.

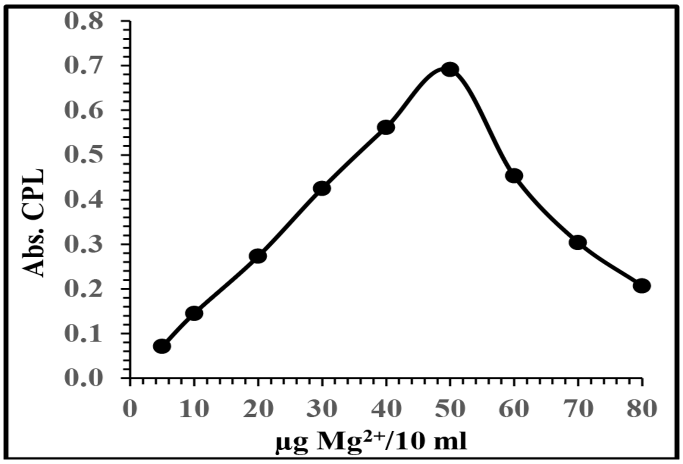

Figure 5 Effect of metal ion Con. on the development of the Mg2+ ion complex and its stability

The results show up an increasing metal ion concentration that gives a straight-line relationship with the increasing concentration of ion-pair association complex formations and D-Value. These results confirm that the metal in concentration is a Thermodynamic Data control thermodynamic equilibrium.

5. Variation JGB concentration

A series of 10 mL aqueous solutions that contain 50 µg Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH are prepared with different concentrations of JGB, and 0.5 mL, Triton X-100, needs heating these solutions in an electrical water bath at 85 °C for 20 min. until the formation of CPL. Then, the work is completed according to the general method illustrated in figure (6) which shows the results.

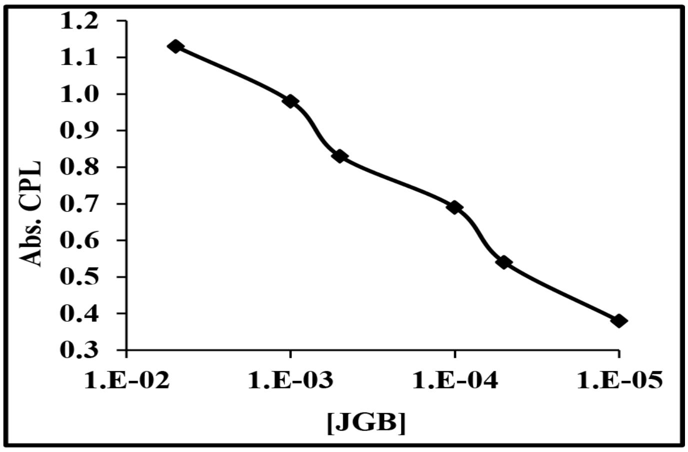

Figure 6 Effect of JGB Conc. on formation and stability for Mg2+ complex extracted

The Results show up an increasing extraction efficiency of Mg^{2+} ion with an increasing organic reagent JGB, which means an increasing JGB effect to increase the velocity of the thermodynamic equilibrium relation for the formation of an ion pair associations complex extracted to CPL. So that, these results confirm with the JGB concentration considered as thermodynamic Data.

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6. Variation Surfactant volume

Preparing many 10 mL aqueous solutions containing 50 µg Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH, 1×10^{-4} M JGB, with the existence of different volumes of Surfactant TX-100. These solutions are heated in the electrical water bath at 85°C for 20 min. until the formation of CPL. Then, this layer is separated and the work is completed according to the general method illustrated in figure (7) that shows the results.

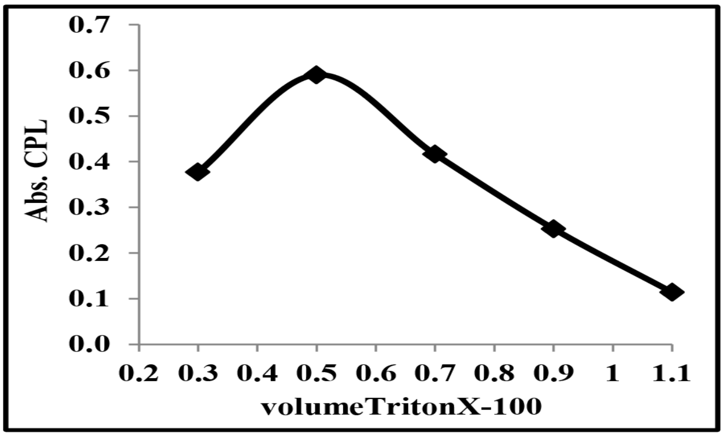

Figure 7 Effect of Triton X-100 volume on the Quality of CPL

The Results illustrated 0.5 mL of TX-100 which was the optimum volume of surfactant to form a high quality of CPL, which gives a higher extraction efficiency of ion pair association complex for Mg^{2+} ion with JGB and 8-HQ.

7. Variation Effect of Temperature

A series of 10 mL aqueous solutions contain 50 µg Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH, 1×10^{-4} M JGB, 0.5 ml Triton X-100. These solutions are heated in an electrical water bath at different temperatures for a suitable time until the formation of CPL. Then these layers are separated and the work is completed according to the general method illustrated in figure (8) that shows the results.

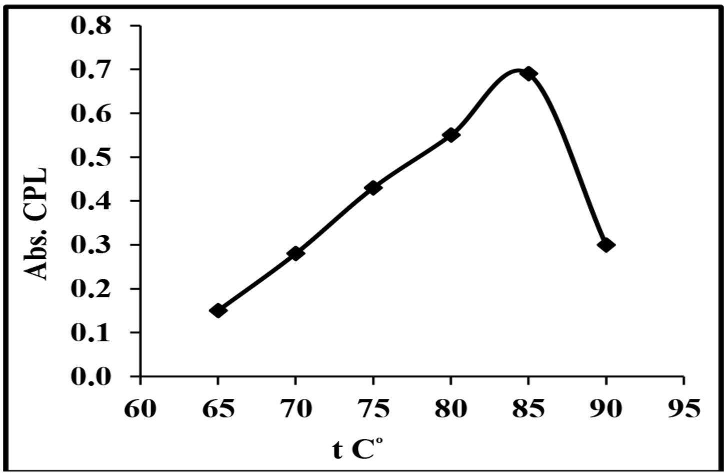

Figure 8 Effect of temperature on CPL development Quality

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The results show that 85°C was the optimum temperature that gives excellent quality for CPL and gives higher extraction efficiency. Afterward, one should calculate the extraction constant K_{ex} from D-values. Figure (9) shows the results.

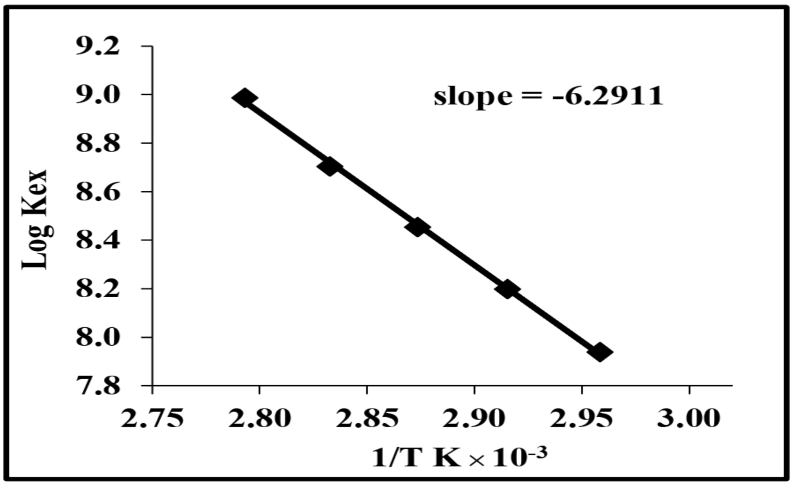

Figure 9 Effect of Temperature on extraction Constant of Mg2+ ion as ion pair association complex

Then from slope value of straight-line relations in Figure (9), and the Thermodynamic relation determined the thermodynamic Data of extraction Mg according to the compact method.

$$\Delta Hex = 0.1205 \ kJ.mol^{-1}$$

$$\Delta Gex = -61.36 \ kJ.mol^{-1}$$

$$\Delta Sex = 171.74 \ J.mol^{-1} \ k^{-1}$$

8. Effect of Heating Time

Preparing 10 mL aqueous solutions contain 50 µg Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH, 1×10^{-4} M JGB, 0.5 mL Triton X-100. This solution is heated in an electrostatic water bath at 85 °C for different times until the formation of CPL. Then, these CPL are separated and the following compact method is completed according to the general method illustrated in figure (10) that shows the results.

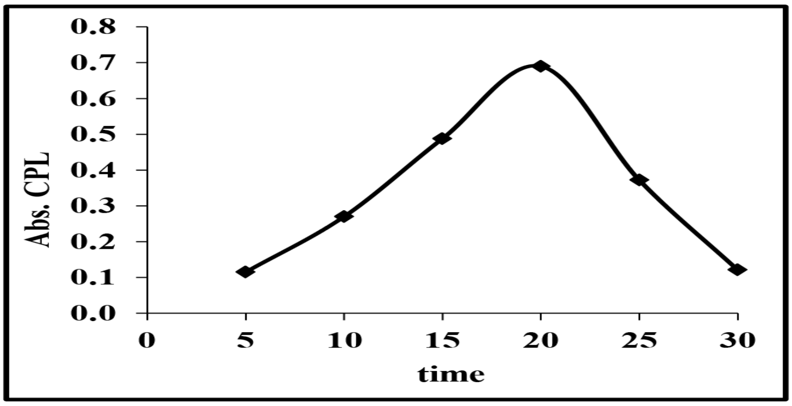

Figure 10 Effect of Temperature on extraction Constant of Mg2+ ion as ion pair association complex

The results appear within 20 minutes, which is the optimum time suitable for the formation of the best CPL. These results are in increased extraction efficiency because this CPL ability is capable of extracting a greater quantity of the Mg^{2+} ions after the formation of an ion pair association complex.

Stoichiometry

In order to see the composition of Mg^{2+} ion pair, an association complex was extracted to form CPL. The first method to be followed was the Slope analysis while the second one was the Slope ratio method, i.e., by the application of a compact method as detailed in the general method.

1. Slope analysis

The results of the Slope analysis method were as in Figure (11).

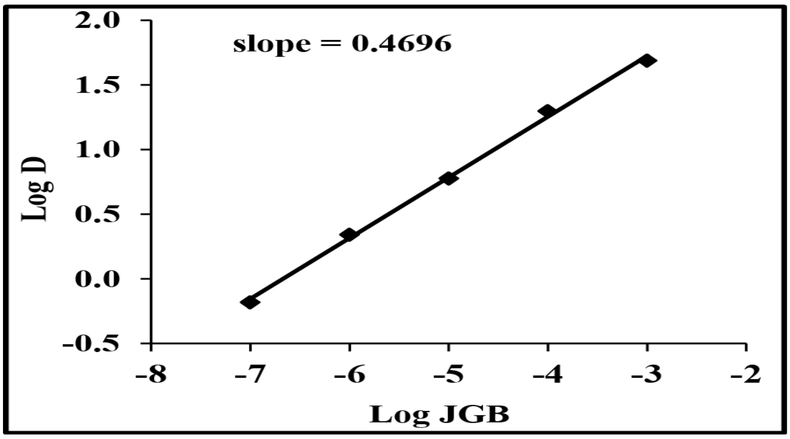

Figure 11 Slope analysis method.

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The results of the slope analysis confirm the structure of the ion pain association complex which was 1:1 JGB^{+}:$Mg(OX)_3^{-}$

2. Slope Ratio

The Slope ratio method was applied by a Compact method, and the results were illustrated in figure (12-13).

The results in the slope ratio method show that the Slope ratio value is equal to (566.01 /560.20 = 1.01), this confirms the structure of ion pain association complex was 1:1 JGB^{+}:$Mg(OX)_3^{-}$.

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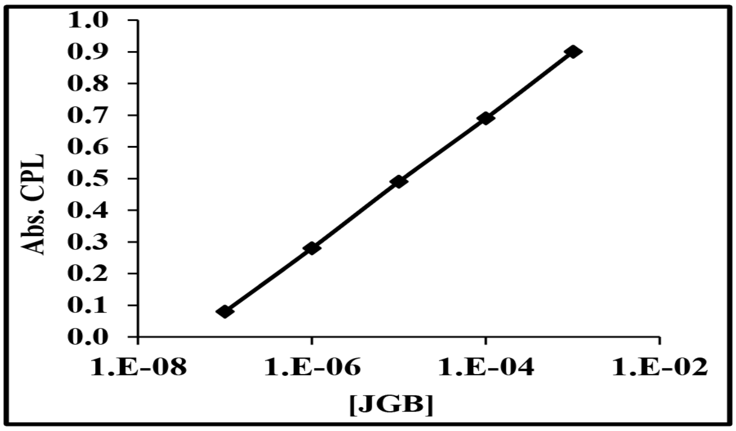

Figure 12 Effect of JGB Con. On development and stability of ion pair association Complex of Mg2+ ion.

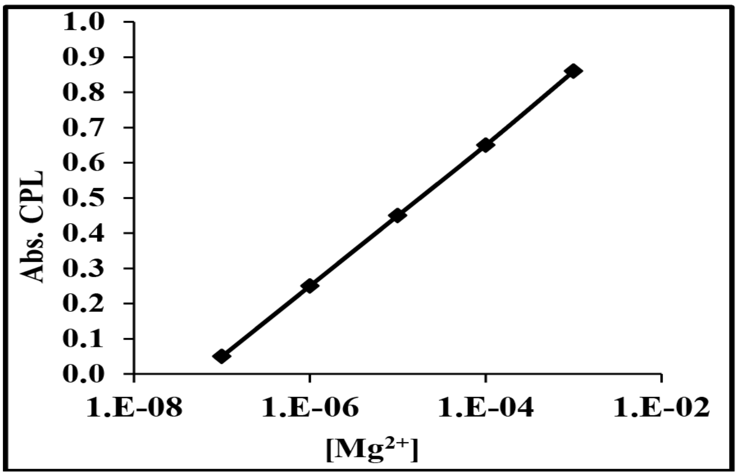

Figure 13 Effect of Mg2+ ion Con. on formation and stability of ion pair association Complex of Mg2+ ion.

Electrolyte effect

Through an application of the Compact method which is detailed in the general method that extracted Mg^{2+} ion at optimum conditions in existence different electrolyte salts in aqueous solution at 0.01 M Con., Table (1) shows the results.

Table (1): Effect of Electrolytes on Extraction Efficiency of Mg2+ ion

Electrolyte	Abs. CPL 565 nm	D
NaCl	0,883	33.21
KCl	0.812	29,95
NH_4Cl	0.754	23,07
$AlCl_3.6H_2O$	0.796	25.00

The results demonstrate the existence of electrolyte in aqueous solution side by side with Mg^{2+}ion effect to increase the extraction efficiency, that is the purpose of electrolyte salt effect is to increase the formation of an ion-pair association complex extracted to CPL, as well as the effect to increase dehydration to increase the quality of cloud point layer formations.

Interferences

Preparing 10mL aqueous solutions to contain 50 µg Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH, 1×10^{-4} M JGB, 0.5 mL Triton X-100, with existence 0.01M some metal ions, needs heating these solutions at 85°C for 20 minutes in an electrostatic water bath to form CPL. Then the work is completed according to the general method illustrated in the table (2) which shows the results.

Table (2): Effect of interferences on Extraction Efficiency of Mg^{2+} ion

interferences	Abs. CPL 565 nm	D
Zn^{2+}	0.510	11.26
Ca^{2+}	0.493	8.70
Ba^{2+}	0.362	5.57

The results show up the existence of foreign ion in aqueous solution side by side with Mg^{2+} ion which can develop ion pair-association complex with organic reagent JGB ad 8-HQ effect to decrease the extraction efficiency of Mg^{2+} ion due to the consumption of some 8HQ and JGB. Besides, their concentrations are declined less than the optimum concentration necessary for the extraction of Mg^{2+}ion with high efficiency.

Effect of Organic Reagent kind

Preparing three aqueous solutions 10mL in the volume containing 50 µg Mg^{2+} ion, 1×10^{-3} M 8-HQ, 0.05 M NaOH, 0.5 mL Triton X-100 and 1×10^{-3} M of different organic Reagent. Then, these solutions are heated in an electrostatic water bath at 85°C for 20 mint. until the formation of the CPL complex. the work is done according to the general method illustrated in table (3) which shows the results.

Table (3): Effect of Organic Reagent Kind.

Organic reagent	λmax	Abs. CPL	D
Safranin	535	0.520	14.12
Crystal Violet	513	0.601	16.52
Rhodamine 6G	539	0.492	10.02

The results show that there is a different extraction efficiency with different organic reagents because there is a different ability to form an ion-pair association complex with $Mg(OX)_3^-$, that functions as an ion exchange due to the structure of the organic reagent and its behavior in aqueous solution.

Conclusion

1. It is necessary to adjust the alkaline medium to form Oxine.
2. It is very important to specify 8-Hydroxy quinoline concentrations used in aqueous solution to form the magnesium anion complex $Mg(OX)_3^-$, thermodynamically.

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3. A suitable concentration of organic reagent must be used to get an excellent rate of formation direction of ion pair association complex in thermodynamic equilibrium.
4. By using LiCl as a strong electrolyte salt in an aqueous solution, enhancement should be given in the extraction efficiency of Mg^{2+}ion, because any increase will destroy hydration of Mg ion and lead to the dehydration of micelles became its smaller ionic Radius.
5. The heating time represents the kinetic site of the extraction method which has given sufficient time for the dehydration and formation of an ion pair complex, as well as the partition of an ion pair complex to the Cloud port layer.

Recommendation

1. It is possible to use this Compact method for the separation and determination of other metal ions in aqueous solutions.
2. It is possible to use EDTA as an ion instead of 8HQ to form an ion pair association complex.
3. For the separation and determination transition or lanthanide and Actinide of metal ions, high molecular amines or azo-derivative, Crown ether and Cryptands must be used in Acidic HCl medium.
4. In order to determine Mg^{2+} ion in aqueous solution samples, a suitable masking agent must be used for other metal ions existence in aqueous solutions.
5. When Crown ether and Cryptand are used to separate and determine a metal ion as an ion by liquid ion-exchange it is possible to apply this method from acidic, alkaline and Neutral media.

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