Simple Cloud Point Extraction-Spectrophotometric for the Determination for Cobalt (II) Using -3-Anilino-1-Phenylimino-Thiourea

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Abstract
Cloud-point extraction (CPE) procedure was applied to enrich cobalt (II) after complexing with 3-anilino-1-phenylimino-thiourea (R.) with subsequent analysis by uv–visible spectrometry with the presence of Triton X-114 as a suitable non-ionic surfactant. The experimental conditions that would affect the CPE process have been improved such as pH, concentration for (R) and volume Triton X-114(0.6ml), equilibration temperature 45∘C and duration and finally centrifugation (rate and time)4,000 rpm,20 min. Starting from the effect of pH at (4.9), pre-concentration of sample in a 5 mL solution consists of 2.5% (v/v) Triton X-114 with 1×10-3 mol.L−1 of (R.) permitted the detection (LOD) of (0.17) µg.mL-1 of cobalt (II) and limit of quantification (LOQ) (0.49) µg.mL -1. A linear calibration curve was obtained within the range (0.06 -3.0) µg. mL-1 with a precision that is expressed as the relative standard deviation (RSD, n =7,6.3 µg mL-1 Co (II))
Keywords: cobalt ion, Triton X-114, Cloud point extraction, 3-anilino-1-phenylimino-thiourea

1. Introduction

Cloud point extraction (CPE) is based on the phase behavior of non-ionic surfactants in aqueous solution, which exhibit phase separation after an increase in temperature or the addition of a salting out agent and use the centrifuge. Separation and preconcentration based on (CPE) are becoming an important and practical application of surfactant in analytical chemistry [1,2]. Cloud point extraction has been used for pre-Concentration of Co (II) after Complex with 3-anilino-1-phenylimino-thiourea, then spectrophotometric (U.V) for the determination for Co(II). The analyst is extracted to the phase rich in the Surfactant Tritonx-114[3].
The classical liquid–liquid extraction and separation methods are usually time consuming and labor extensive and require relatively large volumes of high purity solvents. Of additional concern is disposal of the solvent used, which creates a severe environmental problem. Cloud point extraction (CPE) is an attractive technique that reduces the consumption of and exposure to a solvent, disposal costs and extraction time [4–11]. Cloud point methodology has been used for the extraction and preconcentrations of metal ions after the formation of sparingly water-soluble complexes [12,13].
We used 3-anilino-1-phenylimino-thiourea as a complexing agent in cloud point extraction and applied it for selective separation and preconcentration of silver in acidic condition [4]. 3-anilino-1-phenylimino-thiourea is a strong ligand for metal ion extraction and is widely used in liquid–liquid [14] and solid phase extraction [15], because it can form stable complexes with many metal ions under proper conditions. The aim of this work was the development of a new cloud point extraction and preconcentration method for Co by the use of 3-anilino-1-phenylimino-thioureaas a complexing agent prior to spectrophotometric determination (U.V)

2. experimental

The reagent solution was prepared at 1 x 10-3 mol. L-1 in a 50 mL volumetric flask by dissolving a appropriate amount of (R) in 5ml NaOH (0.1M). Using distilled water, an appropriate amount of lead nitrate was dissolved to prepare 6 µg. mL-1 of the standard lead solution. Using distilled water, 2.5 mL of Triton X-114 was diluted in a 50 mL volumetric flask to obtain 5% (v/v) of Triton X-114.

2.1. Instrumentation

To determine the cobalt (II) content, been measured the absorption of the complex formed the pH of the solutions was measured with a pH meter, inolab, WTW, 720 (Germany). a spectrophotometer (Shimadzu UV-vis1650),(Tokyo, Japan) was used. To obtain suitable temperatures for aqueous samples, the water bath model (OPTIMA, Japan) was used, A medifuge centrifuge model was used to separate the aqueous phase from SRP

2.2 General CPE Procedure [16]

In a 5.0 mL volumetric flask, add 1.0 mL of 6 µg. mL-1 Co (II) standard solution and 1 mL (1 x 10-3) mol. L-1 reagent as a blank. complexing agent in the presence of 0.6 mL (2.5 percent (v/v)) Triton X-114 as a micellar medium and diluted the mixture with

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distilled water to the required volume. The sample solution is placed in a conical-shaped centrifuge tube and kept in a water bath at 45 C for 20 minutes, after which it is centrifuged at 4000 rpm for 20 minutes to obtain the desired result.

Bring the phase separation process to a successful conclusion. The aqueous phase is easily poured, and then 3.0 mL of ethanol absolute is added to the rich phase of the surfactant to treat the viscosity and finish the volume necessary. For analysis, in a cuvette the analytical signal is captured at max wavelength (550) nm to determine the cobalt (II) level by measuring the absorbance of the sample

3. Resuls and Discussion

3.1Absorption spectra

Using spectrophotometer (shimadzu uv-vis1650), the absorption spectrums of: [Co (II) – (R.)] complex by CPE, cobalt (II), and reagent (R) were determined as shown in (Figure- 1), the complex is shown the maximum absorption (λmax) at (550 nm)and can be considered evidence of its formation.

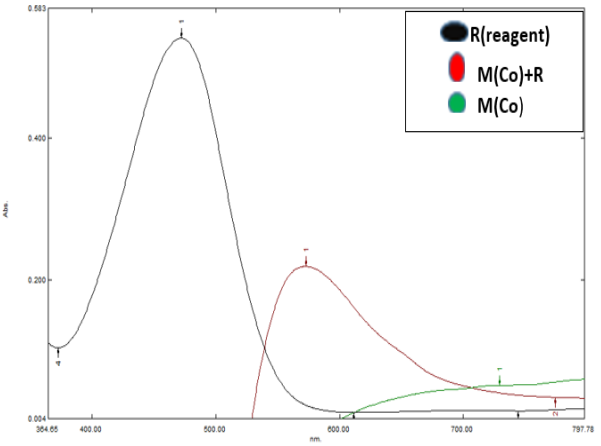
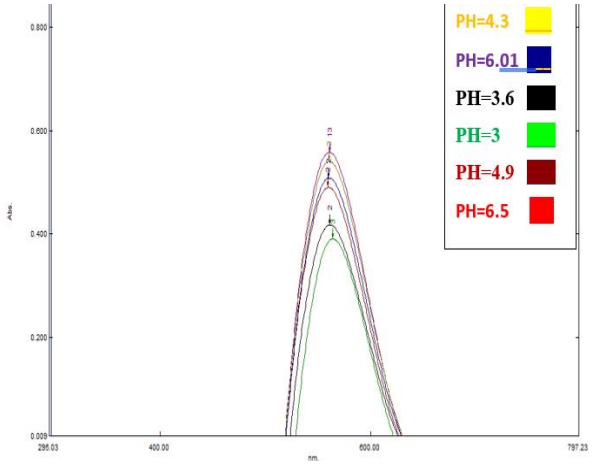
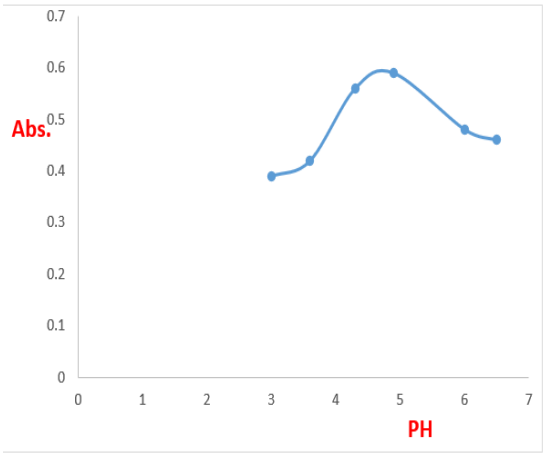


Figure (1): Absorption spectrums of: cobalt (II) ions, complexing agent (R), and [Co (II)-R] complex.

3.2 Effect of pH

Accordingly, Similar tests were performed at different pH ranges (3-6.5) which is adjusted with dilute solutions (0.1) mol. L^{-1} of HCl and NaOH. The results are in Figure. (2), where a pH of 4.9 can be considered an ideal point for cobalt (II) determination by CPE





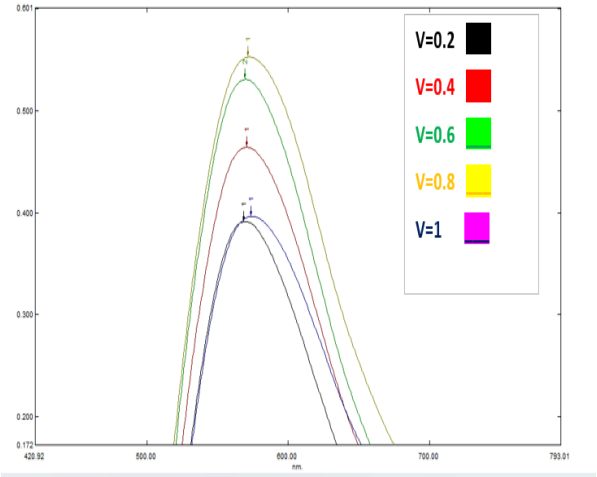


Figure (2): Absorption spectra of [Co (II) -R] complex u nder effect of pH

3.3 Effect of (R) Concentration

In this work, the effect of reagent concentration on the extraction process in CPE was tested by preparing similar solutions containing different reagent volumes (0.2-1) mL 1x 10^{-3} mol. L^{-1} with 6 µg. mL^{-1} of cobalt (II) standard solution in the presence of 0.6 mL 5% (v/v) of TritonX-114 at pH 4.9. (0.8) mL was chosen to be the optimal volume in this work

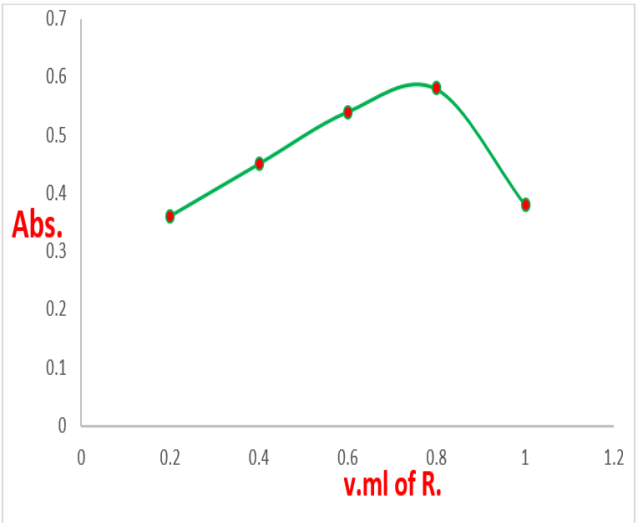


Figure (3): Absorption spectra of [Co (II) -R] complex under effect of concentration of reagent (R).

3.4 Effect of Triton X-114 volume

The influence of Triton X-114 percentage on the CPE was tested by adding different volumes of it in the range (0.2-1) mL at a concentration of (2.5% (v / v). It is observed from the data shown in Figure. (3-17) at (0.6) mL was chosen to be the optimal volume in this work

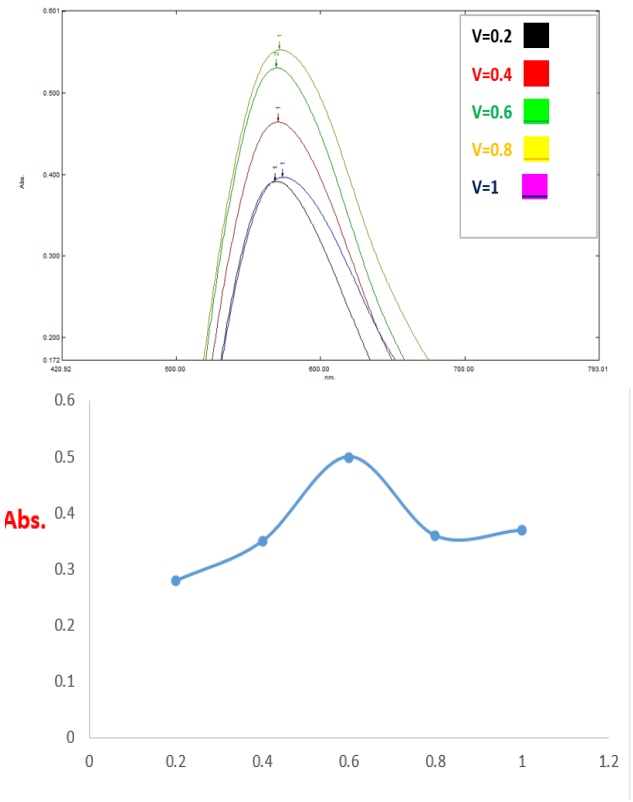
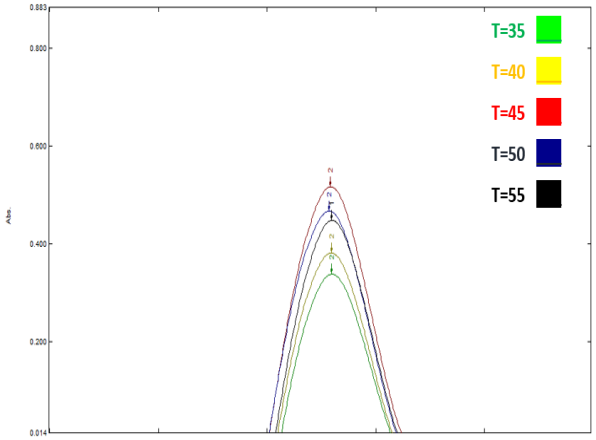


Figure (4): Absorption spectra of [Co (II) -R] complex under effect of concentration of Triton X-114.

3.5 Effect of Equilibrium Temperature and incubation time

the influence of the equilibrium temperature was tested from 35C to 55°C at 15 min. while maintaining the other parameters under optimal conditions. Figure (5) shows the results obtained from this test, where the best absorption was shown at 45 °C. On the other hand, it was observed that the incubation period of 20 minutes gave the best extraction of lead content during the experiment as shown in Figure. (6)



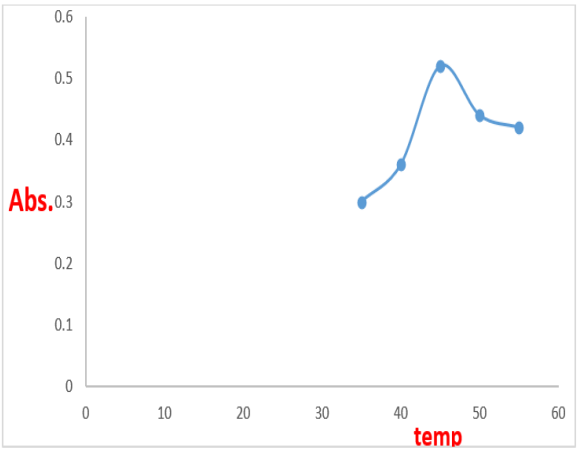
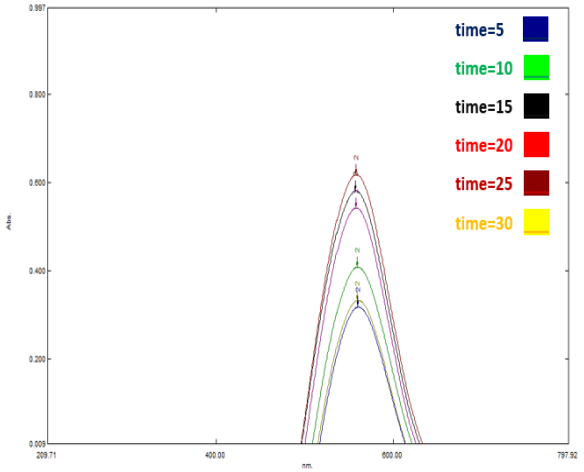


Figure (5): Absorption spectra of [Co (II) -R] complex undr effect of equilibration temperature



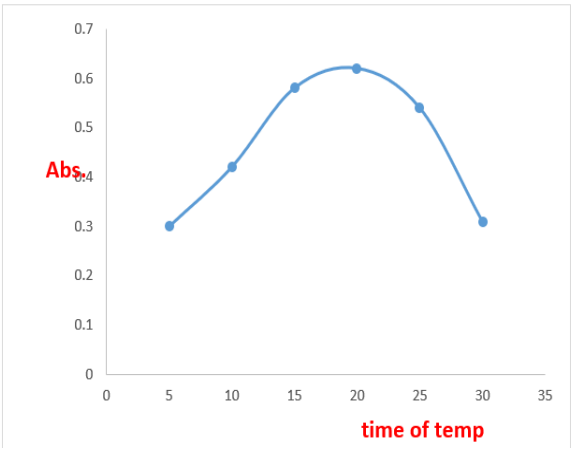


Figure (6): Absorption spectra of [Co (II) -R] complex under effect of Incubation time

3.6 Effect of Centrifugation Time and Rate

Time is one of the factors that are no less important than other experimental conditions in CPE, where the influence of time on the centrifugation process was evaluated for a number of samples in the range (5-30) min. (20) min was chosen as the ideal time. The absorption values for this test were shown in Figure (7), the effect of centrifugal rate on the extraction process was examined and from the experimental results in Figure. (8), 4000 rpm were chosen as an appropriate rate in improving the lead extraction efficiency of CPE.

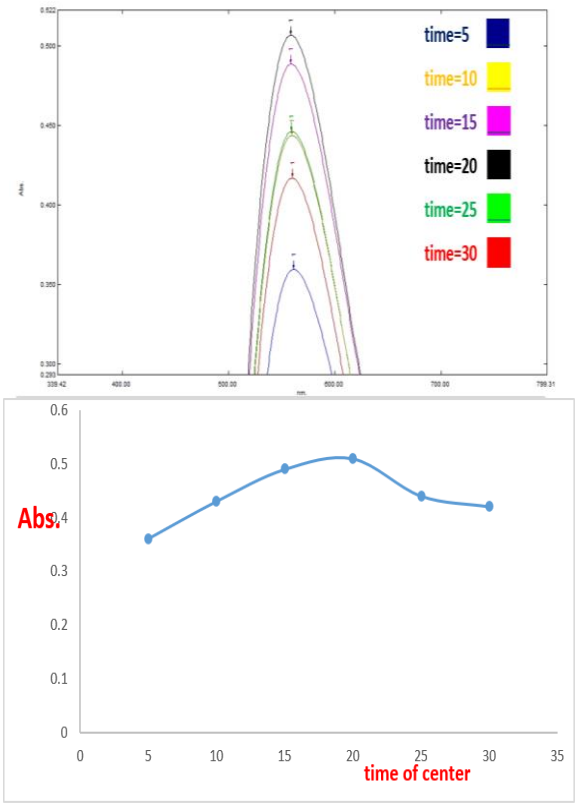


Figure (7), the effect of centrifugal time

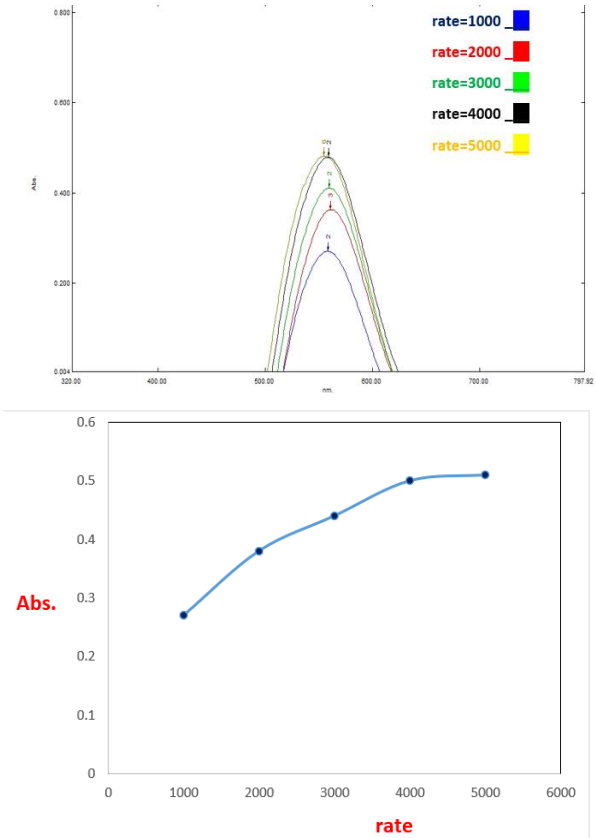


Figure (8): Absorption spectra of [Co (II) -R] complex under effect of centrifuge rate

3.7 The calibration curve and analytical performance of the CPE method

A linear calibration curve as shown in Figure (9) was obtained after applying all the optimal experimental conditions developed during the previous experiments.

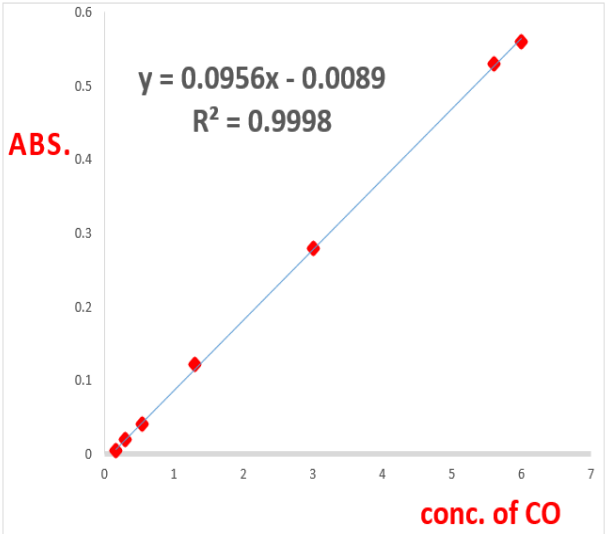


Figure (9): Calibration curve for [Co (II)-R] complex using CPE method

Table 1: Optimum conditions for CPE to determine Co (II).	
Conditions	Value
pH	4.9
Concentration of (R).	0.8 mL (1×10−3) (mol.L−1)
Concentration of surfactant	0.6 mL (2.5% (v/v)) of TritonX-114
Equilibrium temperature(∘C)	45
Equilibration time (min.)	20
Centrifugation rate (rpm)	4000
Centrifugation time (min.)	20

4. Conclusion

The CPE procedure was applied to determine the cobalt (II) content in aqueous solutions using (R.) as a ligand with the presence of Triton X-114 as a micellar medium. The improvement of the experimental conditions increased the sensitivity and reduced the detection limit. This technique can be used as a substitutional to conventional extraction methods because it is environmentally friendly through its low consumption of organic solvents, in addition to being efficient, fast and cheap

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