ORIGINAL ARTICLE

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

ALI AHSSAN HAMZ NAJM[1], AHMED S. ABBAS[2]
[1-2]Babylon University, College of Science, Department of Chemistry
Correspondence to: Ali Ahssan Hamz Najm, Email: ali.najam.scihigh126@student.uobabylon.edu.iq

ABSTRACT

After complexing with 3-anilino-1-phenylimino-thiourea (R.), the cloud-point extraction (CPE) process was used to enrich lead (II), which was then analyzed using uv–visible spectrometry using Triton X-114 as a suitable non-ionic surfactant. pH, concentration for (R) and volume Triton X-114(0.6ml), equilibration temperature 50C and duration, and lastly centrifugation (rate and time)4,000 rpm,20 min have all been enhanced as experimental variables that impact the CPE process. Pre-concentration of sample in a 5 mL solution contains 2.5 percent (v/v) starting with the influence of pH at (4.2). The detection (LOD) of (0.018) µg.mL^{-1} of lead (+2) and the limit of quantification (LOQ) of (0.08) µg.mL^{-1} were achieved using Triton X-114 with 1×10^{-3} mol.L^{-1} of (R.). A linear calibration curve with a precision of relative standard deviation (R.S.D, n =7,0.3 µg. mL^{-1} Pb (II)) were obtained within the range (0.06 -3.0) µg.mL^{-1}.

Keywords: Pb^{+2}; Triton(X—114) ,Cloud point extraction(C.P.E); 3-anilino-1-phenylimino-thiourea;

INTRODUCTION

(CPE) is dependent also on phase behavior of non-ionic surfactants in aqueous solution, that show phase separation with a temperature rise or even the presence of the a salting out reagent, including use of of the a centrifuge. Surfactant separating and pre - concentration depending on (CPE) becomes a popular and useful application in chemical analysis [1,2]. During complexing by 3-anilino-1-phenylimino-thiourea, cloud point extraction was utilized for pre-concentrate Pb(II), followed by spectrophotometric (U.V) measurement for pb (II). The analyst is removed to a Surfactant Tritonx—114[3] rich phase.

the traditional liquid–liquid extraction and separating procedures are time-consuming and labor-intensive, and they necessitate relatively large amounts solvents. The disposal of a solvents used, which produces a serious environmental problems, is a source of worry. Cloud point extraction (CPE) is a popular method for reducing solvents usage and exposure, as well as disposal costs and extraction time [4–11]. Metal ions have been extracted and preconcentrated using cloud point methods following the production of sparingly water soluble complexes[12,13].

We initially employed{ 3-anilino-1-phenylimino-thiourea }as a metal complex in {CPE} and then used it for selected silver separate and preconcentration in acidic conditions [4]. As it can form stable complexes with various metal ions under the right circumstances, 3-anilino-1-phenylimino-thiourea is a powerful ligands of metal ion extracting and also is frequently employed in liquid–liquid [14] and solid phase extraction [15]. The goal of this study was to create a new cloud point extraction and pre - concentration approach of pb^{+2} using 3-anilino-1-phenylimino-thiourea as a complexing agent prior to(uv) spectrophotometer

Experimental: In a 50 milliliter volumetric flask, a reagents solutions were produced using dissolving a suitable quantity for using 5 milliliter NaOH (0.1M) at 1×10^{-3} mol.L^{-1}. The standard lead solution was prepared by dissolving a sufficient quantity of lead nitrate with D.W to obtain 3µg .mL^{-1}. 2.5 millileter of Triton X-114 was dissolved inside a 50 milliliter with D.W to reach 2.5 percent {v/v}.

Instrumantation: The absorbance of a complexes produced was measured to estimate the pb^{+2} concentration. A pH meter, inolab, WTW, 7 20 , was used to measure the ph of the solutions {Germany}. The spectrophotometer utilized was indeed a Shimadzu UV-visible1650 {Tokyo, Japan} spectrophotometer. The water bath models (OPTIMA, Japan) were utilized to establish acceptable temperatures of aqueous samples. Its aqueous phase were separated form SRP using a medifuge centrifugation type.

General {CPE }Procedure[16]: Add 1.0 millileter of 3µg .mL^{-1} Pb $^{+2}$ standard solution and {1 millileter }(1 x 10^{-3}) mol.L-1 reagents as a blank to a 5.0 millileter volumetric flask . {0.4 millileter }(2.5 cents (v/v.)) Triton X—114 has been used as a micellar medium in the presence of the complexing agent, as well as the combination

was diluted to the appropriate amount using {D.W}. As attain the desired results, the sample solution is put in a conical-shaped centrifuged tube then maintained in a water bath around 50 ° c for {15 min} before being centrifuged around 4000 rp m for (20min).

Ensure that the phase separation procedure was completed successfully. This aqueous phase is readily poured, when{ 3.0 milliliter} of ethanol absolutely is added to the surfactant's rich phase for treat viscosity as well as complete the volume required. The analytical signal is caught inside a cuvette with maximum wave lengths (560 nm)to ascertain the pb^{+2} content by calculating a sample's absorption.

DISCUSSIONS AND CONCLUSION

Spectrum of absorbance: Spectrum of absorbancAs absorbance spectrum of the [Pb^{+2} –- (R.)] combination with CPE, pb^{+2} and reagents (R.) was obtained to use a spectrophotometer (Shimadzu uv—visibile1650,), as seen in {Fig.- 1}, the combination has the greatest absorbance(maximum) around {560 nm} and may be regarded proof from its creation.
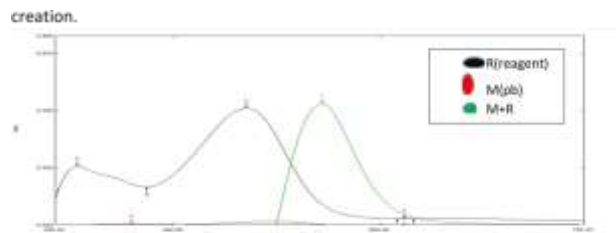
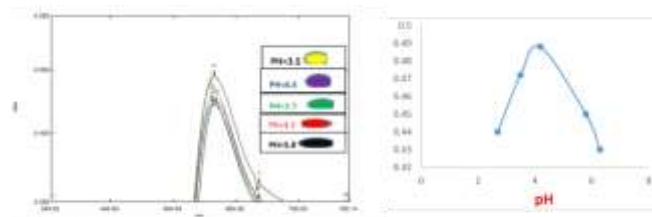{Figure- 1}Pb $^{+2}$; {R .} & Complexes [Pb^{+2}–R] Uv - visible absorption

fig. (2). Pb^{+3} extract efficiency as well as the ph . 3 µgmL-1 Pb $^{+2}$; 1 =t0^{-3} mol l^{-1}(R) . ; 0.6millileter Triton X — 114, 2.5 percent {v/v}; Equilibration temp 50°C, incubating duration 15 min).

The pH Effect: Its {CPE} process is critically reliant on pH since it has a direct influence on the formation and stability of a complexing metals [17]. As a result, similar experiments are carried out at various pH values (3.5-5.8) using weak solutions

(0.1) mol.L-1 of HCl and NaOH to modify the pH. Fig. (2) shows the results, which show that a pH around (4.2) is an optimal pH for{ CPE} lead (2+) detection.

Effects of Volume of 3-anilino-1-phenylimino-thiourea (R.) on Pb+2 Extraction: (5 milliliter) volumetric flasks containing 3 µg mL-1 pb+2 standard solution and (0.8 milliliter) (1 x 10-3) mol. L-1 reagent in the presence of 0.6 milliliter (2.5 cent (v/v)) Triton X-114 at pH 4.2, temp 50 °C, with heating duration 15 min After finishing the appropriate volume with D.W and using the normal technique for(CPE), its absorbance of the samples at (560 nm.).
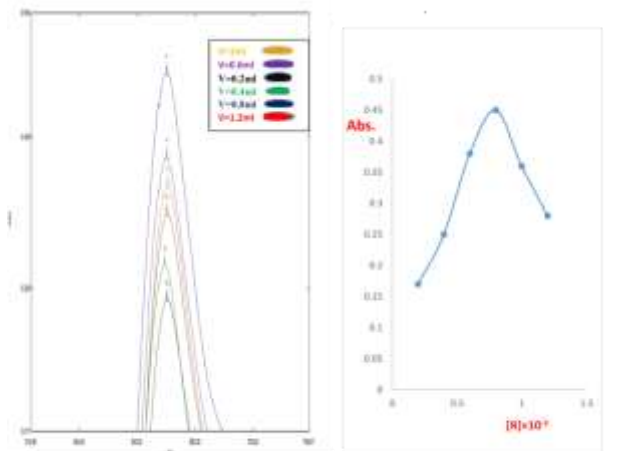

Fig. (3). A influence of reagents volume of Pb+2 extraction effciency was tested . pH 4.2, 3 µg mL-1 Pb +2, 2.5 percent Triton X-114 (v/v), Equilibration temp 50 °C, with incubating time (15 min).

The volume of Triton X—1´14 has an affect: By adding varied volumes with Triton X-114 in the ranges of (0.2-1) mL at a concentrations of (2.5 cent (v / v), the effect of Triton X-114 on (CPE)were investigated. The extraction sensitivity steadily increases with increasing the quantity of surfactant until it reaches (0.6) milliliters, then the analysis signal gradually diminish with the ongoing increase in volume of Triton X-114, as shown in{ Figure.(4)}. To clarify this, during first case, there is insufficient volume in the rich phase of the surfactant to capture the ;greatest amount of $[Pb^{+2} – (R.)]$ complicated,., In the seconds case, however, The existence of an excessive quantity of Triton X-114 causes SRP viscosity to rise, resulting in reduced sensitivity and a reduction in analysis signal [17]. Depending on the preceding, the best volume inside this study was 2.5 percent (v/v) 'Triton X-1´14 at (0.6)millileter.
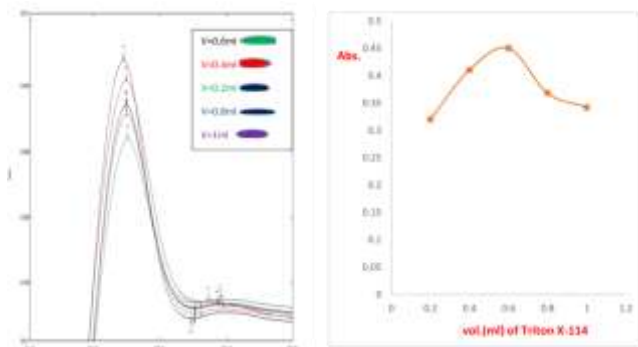
Figure(4). The influence with Triton X—114 concentrations of Pb+2 extraction efficiency were tested , pH 4.2, 3 µg.mL-1 Pb+2, (R.), 1× 10-9 mol. 1 L-1, Temp of equilibrium is 50°C, and the incubating period (15 min)

The Effect of Equilibrium Time and Temperature: "Since of their influence on the rich phase of the surfactant, equilibrium temp as well as duration are also among the experiments influencing the CPE method for its role through enhancing extraction efficiency, as both can induce a small decrease inside the water activity and therefore contribute to the a phase with little volume. A few literature has suggested that dehydration is induced by the rupture of hydrogen bonds due to a rise in temp [18]. As a result, its effect of a average temperature was investigated by varying this from 30°C to 60°C for 15 minutes whilst keeping other factors constant. Figure (5) depicts the results of this test, with the better absorption occurring at 55 ° C. On either side, this was discovered that a 15-minute incubation resulted in the best lead content extraction as during experiment, as seen in Fig. (6)"
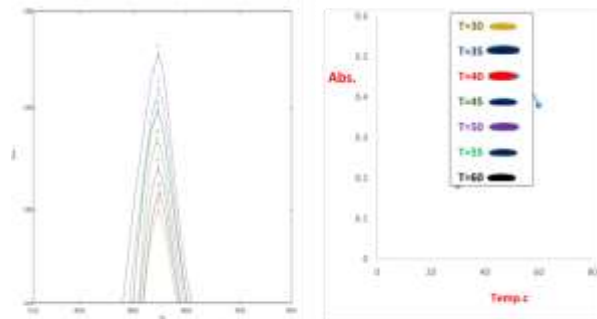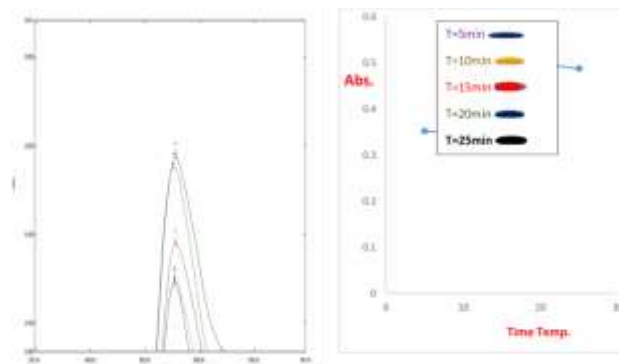

"Fig. (5):the influence the equilibration temp of Pb+2 extraction efficiency were tested .   pH 4.2, 3µg . mL-1 Pb+2: (R.),   1×10-3 mol.L-1; 0.6 millileter, Triton X — 114, 2.5 percent (v/v) after 15 minutes incubating}."

"(Figure 6) Results of tests on the influence of equilibration duration on

(Pb+2)extraction efficiency . pH 4.2; 3µg .mL-1 Pb(II); (R.), 1 ×10-3 mol.L–1; 0.6 millileter Triton X – 114, 2.5 percent "(v/v); 50 °C equilibria temp."

Duration & rate of centrifugation have an effect: As {CPE}, the impact the time on the centrifuged process was investigated for a sample data in the range (5-25) min, and it was found to be no less essential than some other experimental parameters. The absorbance results for with this testing are presented in Fig. (7), and it has been discovered that increasing the centrifuge duration enhanced extraction efficiency, therefore 20 minutes was selected as the optimal time to finish the centrifugation separation process. On the other side, keeping a time factor equal (20) minutes in mind, The influence of centrifuge rate on the extracting process was studied, and based on the experimental data in Fig. (8), 4,000 rpm was determined being an acceptable rate for enhancing {CPE} lead extraction efficiency.

"(Figure 6) Results of tests on the influence of equilibration duration on

"{Fig. 7}: Result of tests on the impact of centrifugation period on Pb^{+2} extraction efficiency . pH 4.2; 3 μgmL^{-1} Pb (II), (R.); 1 ×10^{-3} mol. L^{-1}, 0.6millileter Triton X–-114 ;2.5 percent (v/v;), 50"°C equilibration temp, and incubation time; (15 min)."
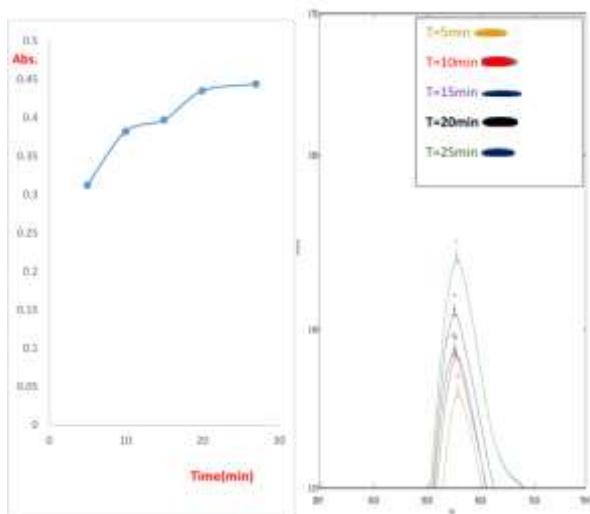

Figure 7:

Effect of salt out: According to the research, salting can enhance the extraction proces in a more effective extraction [19,20]. Following adding(1)milliliter of 0.1 mol.L^{-1} NaCl, MgSO4.7H2O, NaNO3, and NH4NO3 in identical solutions, ;the influence of salting here on extraction of lead (+2) was studied. After completing the requisite processes, the findings in Figures (9) revealed that salt employed in this experiment won't result in either rise inside the analysis signals, indicating thus salting did not play a role inside this study, as described in table (2).
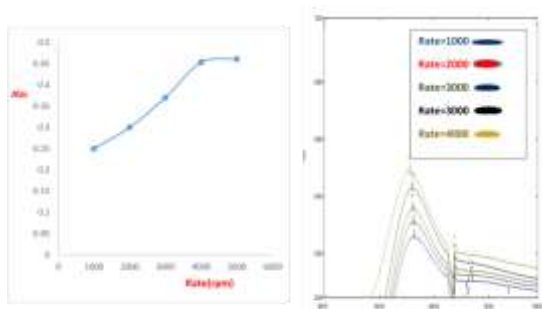

Fig . 8 shows the results of tests here on influence of centrifugation rate on Pb^{+2} extraction efficiency . pH 4.2, 3 μg.mL.$^{-1}$ Pb^{+2}, (R.), 1 ×10^{-3} mol.L^{-1} Equilibration temp 50°C with equilibrium period 15 min; L^{-1},0.6 millileter Triton X –-114,2.5 percent (v/v); Equilibration temp 50°C with equilibrium period 15 min;."

Table of contents 1: The influence of salts on the absorption of the [Pb(II) (R.)] combination

Salt out	Abs
NaNO$_3$	0.409
MgSO$_4$.7H$_2$O	0.411
NaCl	0.483
NH$_4$NO$_3$	0.488

Determination of the Stoichiometric Complexes: The stoichiometry between Pb (II) &(R.) were determined using Job's approach (R). Its absorbance parameters were recorded at 560nm after adding various volumes of standards pb (+2) solution and reagents solutions at same concentrations level of 1 x 10^{-3} mol.L^{-1}. A UV-visible spectrophotometer is used. The stoichiometric ratio between Pb (ii) with (R.) was 1:2, as shown in Fig. 10.
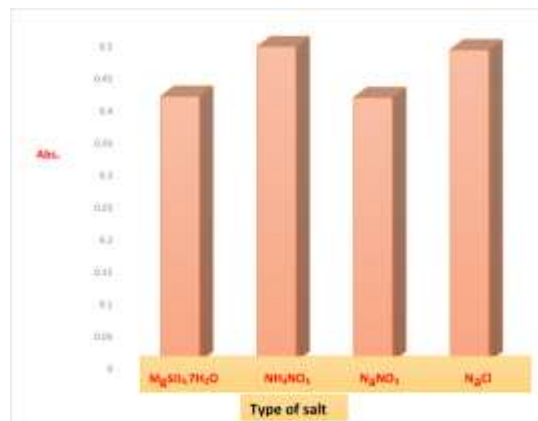

Fig. (9). Result from tests on the influence of salt removal on Pb^{+2} extraction efficiency . pH 4.2; 3 μg.mL^{-1} Pb^{+2}; quantity of (R.), 1 ×10^{-3} mol L^{-1},0.6 milliliter Triton X–-114 (2.5 percent v/v); equilibration temp 50°C; incubation period 15 minutes
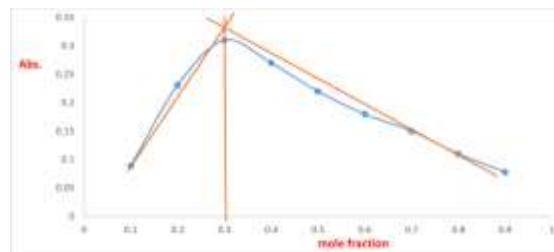

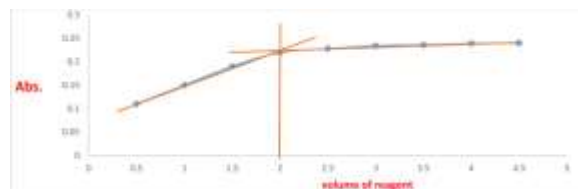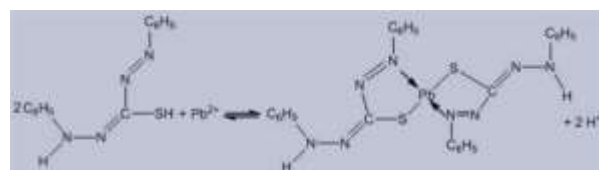Fig 10: Continuous variables"

Fig 11: molar ratios

"A most structure of the combination produced [21] reveals the stoichiometry between Pb (ii) with (R) in {Fig.12.}

"(Fig . 12) shows; At PH 4.2, the chemical structure of the reagents and the combination produced between pb (ii) and (R.)."

The (CPE) method's calibration curve with analysis resulits' The CPE method's calibration curve including its analysis results: By following all the ideal test conditions created during in the preceding trials, a linear calibration curve such shown in Figure (12) was achieved. Calibration data and other analysis factors are included in Tables (3). The calibration curve's standard deviation (RSD) were calculated by seven multiple measures of the standard lead(+2) solution .
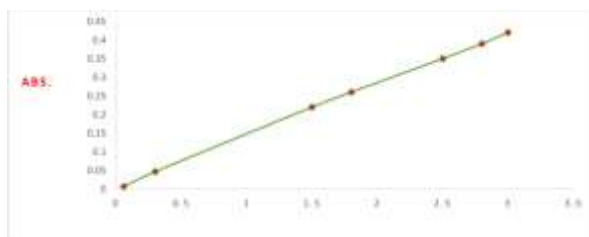
Fig 12: Calibration curve of (Pb(II) – (R.))"

Table 2: In this paper, the principles of the CPE technique are discussed.

Parameters	
Calibration curve	0.1406x + 0.0064
"Correlation coefficient, R2"	0.9997
Linear calibration range µg.mL^{-1}	0.06-3.0
Relative standard deviation	1.9%
Limit of detection µg.mL^{-1}	0.018
Limit of quantification µg.mL^{-1}	0.06
M.L	1.2

Table(3) : Optimum experimental conditions to determine lead(II) by CPE

Conditions	Value
pH	4.2
Conc. of Triton X- 114	2.5 % (v/v)
Centrifugation time (min.)	20
Centrifugation rate (rpm)	4000
Temp. (°C)	50
Conc. of complexing agent	1×10^{-3} (mol.L^{-1})
Incubation time (min.)	15

aqueous solutions. The sensitive and detecting limits were both improved when the experimental circumstances were improved. This approach can be used to replace traditional extraction procedures since it is ecologically beneficial due to the low usage of organic solvents, including being effective, quick, and inexpensive.

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CONCLUSION

Using (R.) as a ligands and Trit'on X-114 as a micellar medium, the CPE technique was used to evaluate the pb (II) concentrat'ion in