Evaluation of Lead Ion in the Wastewater of the Lifting and Treatment Stations Using ICP-MS and CPE Methods

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Abstract: *To pre-concentrate trace amounts of lead before determining it by UV-vis spectrophotometer, a new method for micelle-mediated phase separation has been created. The process depends on the extraction of lead from iodine media using Triton X-114 in the cloud point extraction method without the need for any chelating agents, where the optimal conditions for the method were achieved, which temperature 50 °C, pH 4, and 30 mmol L^{-1} concentration of KI, 0.3 mL of 2% (v/v) Triton X-114, and time of 10 min in the water bath. Linearity was followed between 1 and 16 µg/mL of lead concentration. The method's lead detection limit is 0.1 µg/mL and %RSD 3.633. Additionally, the interference impact of certain cations was evaluated. The proposed technique was successfully applied to determine the lead ion in the wastewater in ten different stations in the center and district of Al-Hur in Karbala City. The lead ion of the wastewater of the stations under study was also determined directly using inductively coupled plasma-mass spectrometry (ICP-MS) technology comparing its results with the new method and performing the statistical analysis of both methods. The p-value was less than 0.05, showing significant differences between both methods.*

Keywords: *chemical pollutants; cloud point extraction method; ICP-MS; lead ion; wastewater*

■ INTRODUCTION

Wastewater which is discharged from homes, businesses, and industrial facilities, is a complicated mixture of water-carrying waste [1]. Municipal and industrial water outlets may contain pollutants, medicinal products, and also other industrial waste, heavy metals, oils, pesticide residues, sludge, and byproducts [2]. The excessive use of these pollutants and their untreated discharge contribute to environmental problems [3]. Other highly hazardous substances, such as toxic metal ions, tend to bioaccumulate and may gradually leak into rivers, endangering aquatic life [4]. Heavy metals present in higher concentrations have a number of harmful consequences for the health of humans and other creatures [5]. Heavy metals can exist naturally in the environment, ecosystem and soils [6]. Wastewater pollution is often caused by the use of various harmful metal ions in mining, recovery and other industrial processes [7]. Lead (Pb), chromium, arsenic, and other dangerous heavy metals can be found in industrial wastewater [8].

Pb is a dangerous heavy metal that is known to be highly toxic, even at very low concentrations, and to have a tendency to accumulate in important human organs, which can have significant negative health effects [9]. Pb is known as one of the extremely harmful species that poses a risk to human health as well as the natural environment. It can have detrimental effects on the heart, kidneys, nervous system, and induce venereal toxicity [10]. World Health Organization (WHO) has listed Pb as one of the hazardous elements and it has issued a stern warning [11].

In analytical chemistry, preconcentration and separation techniques are crucial phases that constitute a significant challenge for researchers looking to improve the methodology's sensitivity and selectivity. For this goal, various methods were developed, including solid phase extraction (SPE) [12], liquid-

liquid extraction (LLE) [13], ion exchange [14], and co-precipitation [15]. A well-known method for removing hydrophobic species from a variety of matrices without the use of organic solvents is cloud point extraction (CPE). The basis of CPE is the turbid behavior of surfactants in aqueous solutions at temperatures approaching cloud point [16]. One of the preferred methods for preconcentrating and separating organic and inorganic metal ions is CPE [17-18]. Because it adheres to the principles of green chemistry, it has attracted a lot of attention. CPE has a number of advantages over traditional extraction techniques, including simplicity and avoiding the use of harmful chemical solvents in large quantities [19-20]. The CPE is influenced by several variables, including temperature, ionic strength, pH, concentrations of the extracting agent and surfactant, and extraction time. Throughout any extraction procedure, it is necessary to optimize how these parameters affect the analyte extraction [21]. Pb(II) ion was removed and detected in the aqueous solution by a new functional composite on high-order mesoporous silica. The limit of detection was 0.51 µg/L [22]. A unique method of immobilizing ligand onto a silica monolith was used to create a highly porous composite material (PCM) that would be used for solid-liquid separation-based Pb(II) ion detection and adsorption from wastewater. The limit of detection was 0.33 µg/L [23]. A novel ligand-based conjugate material (CMA) was prepared for Pb(II) ion monitoring and removal from aqueous solution. The limit of detection was 0.42 µg/L in this method [24]. Pb(II) ion detection and elimination was achieved by directly immobilizing 2-methyl-8-quinolinol onto porous silica to create the mesoporous composite material (MoCA). The detection limit was 0.21 µg/L [25]. A microporous organic composite material was prepared to understand the detection and removal of Pb(II). The detection limit was 0.44 µg/L [26].

The study aimed to estimate the Pb(II) ion in wastewater in ten different stations. Eight lifting stations and two treatment stations were used by using a new alternative UV-vis spectrophotometric method after using the approach of CPE and direct estimation of Pb(II) ion by ICP-MS in wastewater.

■ EXPERIMENTAL SECTION

Materials

All chemical reagents of analytical reagent grade were used, also, deionized water was utilized while taking the tests. Stock Pb(II) solution was made by dissolving 0.159 g of $Pb(NO_3)_2$ (99% purity B.D.H) in 100 mL of deionized water. Triton X-114 at a concentration of 2% (v/v) was prepared from the Triton X-114 standard solution at a concentration of 20% (v/v). Iodide solution KI, and pH buffer reagent (potassium hydrogen phthalate potassium acid) with 0.05 mol L^{-1} concentration.

Instrumentation

All instrument and equipment used in research work was calibrated. Single Beam - Visible Spectrophotometer Model 721, China with a band path 0.1 cm and ICP-MS Agilent 7500 Made in USA instruments were used to detect the metal ion concentration in wastewater.

Procedure

CPE-UV-vis spectroscopy method

According to the typical procedures of the CPE method, 10 mL of 10 µg/mL Pb(II) was added to 1 mL of 30 mmol/L KI and placed in a centrifugal test tube of 15 mL. A yellow color solution of PbI_2 was formed from a dilute solution of l Pb(II) ion and reagent, with a maximum absorption wavelength 424 nm. The pH was then adjusted to 4 using a buffer solution and then 0.3 mL of Triton X-114 in a concentration of 2% (v/v) was added to the solution. A thermostatic water bath set at 60 °C for 10 min was used to heat the sample after it had been shaken, the aqueous phase was separated from the surfactant-rich phase more quickly using heat and the separation was accomplished by centrifugation for 5 min at 3,800 rpm. In order to increase the viscosity of the surfactant-rich phase, the mixture was chilled in an ice bath (0–5 °C), then, the aqueous phase was easily decanted by flipping the tube. Absolute ethanol was used to dilute the micellar extract for this process to 0.5 mL. The solution's absorbance was measured at a maximum wavelength of 424 nm. The proposed mechanism for producing the Pb(II)-KI-Triton X-114 and the CPE for Pb(II) separation, respectively, are presented in Fig. 1.

Sampling

Wastewater samples were taken from ten different stations, which eight lifting stations and two treatment stations in the center and district Al-Hur in Karbala city. Samples were taken from each station at a depth of 1–3 m (a graduated cylinder and a special piece of iron were used for sampling). Wastewater samples were collected in pre-washed 1 L polyethylene containers. Wastewater samples were filtered using filter paper (Whatman 42, 44). Vials and examination equipment were washed with deionized water before use. All samples were kept at low temperature between receipt and analysis to ensure that the samples were not damaged during the laboratory examination. For the samples that were measured using ICP-MS, the digestion process was carried out for the samples before they were examined using HNO_3 of 1.00 mol/L. Fig. 2 shows the sequence of lifting and treatment stations and Table 1 displays the name, symbol, and type of stations. The information system data for sewage systems was provided by the Directorate of Karbala Sewage. Fig. 3 shows the sampling of wastewater procedure.

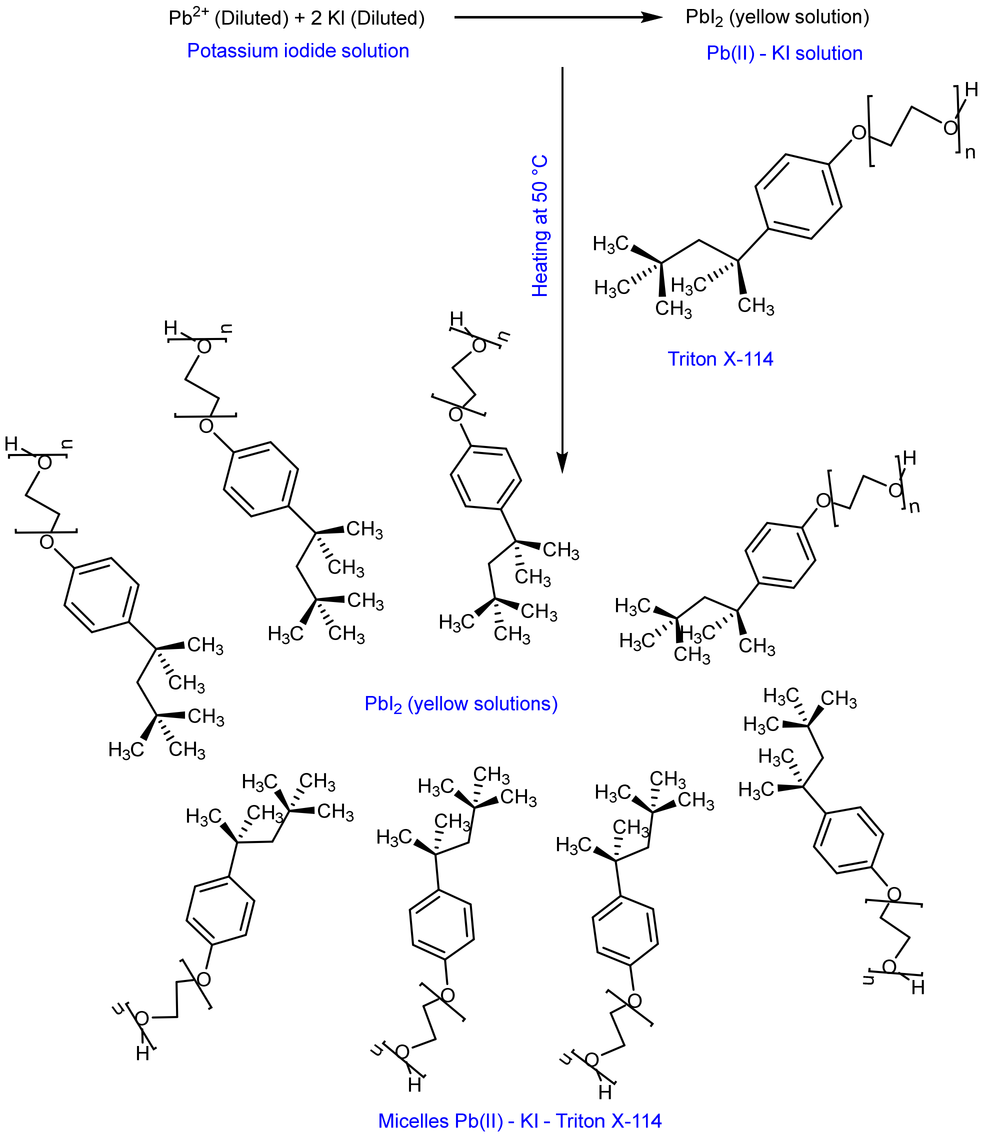

Fig 1. Pb(II)-KI solution separation by the CPE method has been suggested as a methodology

Table 1. Type of the lifting and treatment stations for sewage in this study

Station symbol	Station name	Station type
Z-1	The main processing unit project	Wastewater treatment plant
Z-2	Al-Hur processing unit project	Wastewater treatment plant
Z-3	Messila lift station	Sewage lifting station
Z-4	Oasis station	Sewage lifting station
Z-5	Ghadeer station	Sewage lifting station
Z-6	Staff district station	Sewage lifting station
Z-7	ready lift station	Sewage lifting station
Z-8	Al-Bahadliya station	Sewage lifting station
Z-9	Industrial district lift station	Sewage lifting station
Z-10	Poultry lift station	Sewage lifting station

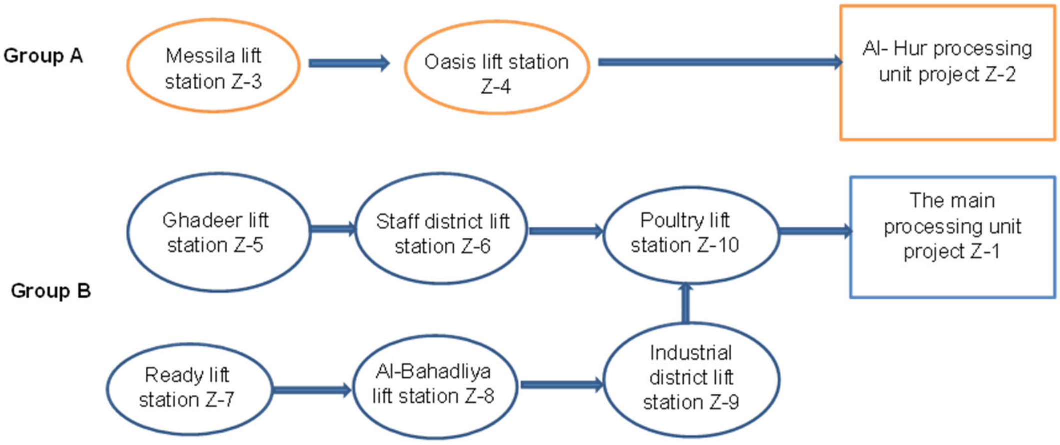

Fig 2. The lifting and treatment stations (A) lifting and treatment stations for Al-Hur District and (B) lifting and treatment stations in the center of Karbala Governorate

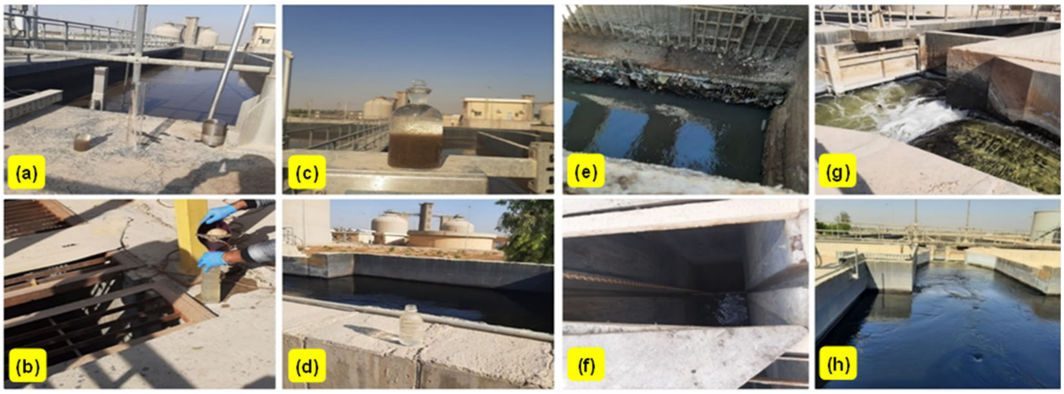

Fig 3. Instruments used and sample collection locations in the lifting and treatment plants, (a) modelling tools, (b) sampling method, (c) sample shape before processing, (d) shape of the sample after processing, (e, f) lifting stations site, and (g, h) treatment stations site

ICP-MS procedure

The most often used and recommended techniques are ICP-MS and inductively coupled plasma-optoelectronic spectroscopy (ICP-OES) because of their well-known advantages of sensitivity, selectivity, and multi-element analytical capabilities [27-29]. ICP-MS technology currently allows for the simultaneous identification of several trace elements [30]. In tests ranging from 1 ng/L to high mg/L ranges, ICP-MS was shown to be more accurate than other methods at detecting components of biological or clinical origin at the lowest concentrations with good linearity and exceptional sensitivity [31].

■ RESULTS AND DISCUSSION

CPE Method

Spectrophotometer UV-vis technique was used to estimate the Pb(II) ion in wastewater because it is an easy and cheap method [32]. Coupling with the CPE method because the Pb(II) concentration in wastewater is very low and requires a pre-concentration process to increase the sensitivity of the method.

Absorption Spectrum

UV-vis spectrums of the Pb(II), KI reagent, and Pb(II)-KI-Triton X-114 were measured. The wavelength of Pb(II) at 301 nm was shown to have a considerable shift in the visible area. When the acid was added, the highest wavelength of the Pb(II)-KI-Triton X-114, at 424 nm was achieved. The KI reagent wavelength was 341 nm.

Optimization Condition

The solution pH, KI concentration, amount of Triton X-100, temperature, and heating time were all optimized to obtain the maximum separation efficiency. According to Eq. (1), recovery of extraction (Recovery%) was used to measure the procedure efficiency [33];

$$\text{Recovery\%} = \left(\frac{C_m \times V_m}{C_i \times V_i} \right) \times 100 \qquad (1)$$

where C_m is the Pb(II) quantity in the micelle phase of volume V_m. Then, C_i and V_i are the initial concentration and volume, respectively.

Order of Addition

The most important factor examined was the sequence of additions [34]. The Pb(II) (10 µg/mL, 10 mL) and KI solution (30 mmol/L, 1 mL) were added. The complex formation was supplemented with 0.3 mL of 2% (v/v) Triton X-114, and the best recovery value was chosen at the sequence of additions, (M + R + pH + T). Fig. 4 displays the impact of order of addition on Pb(II)-KI-Triton X-114 formation.

Influence of pH

The pH measure is among the most essential significant variables impacting the effectiveness of extraction in accordance with the CPE method, because pH plays a critical role in the formation of complexes and the chemical stability of alternatives [35]. The pH may also have an impact on the metal complex's stability constant, which has a major impact on how well CPE separates metal ions [36]. Using 0.1 mol/L HCl and 0.1 mol/L NaOH solution, the pH of the mixture was changed between the ranges of 2 and 10, and the pH was then measured using a pH meter. As shown in Fig. 5(a), the recovery was increased whenever the pH level was equivalent to 4. However, the complexation reaction is likely not complete at pH levels greater than 4, where a precipitate of lead hydroxide was created. By utilizing 0.1 mol/L solutions of HCl, H_2SO_4, and CH_3COOH as well as KHP buffer solution with a pH of 4, the effects of various types of acids were examined. Buffer solution was used to ensure that no precipitates of chlorides or sulphates are formed with the Pb(II) ion solution [37], as shown in Fig. 5(b).

KI Concentration's Impact

The metal ion in CPE should form a stable complex that is separable from the surfactant layer [38]. With a variety of KI solution concentrations (5, 10, 15, 20, 25, 30, 35, and 40 mmol/L) and a volume of 1 mL of KI solution, the effect of iodide concentration on the recovery value

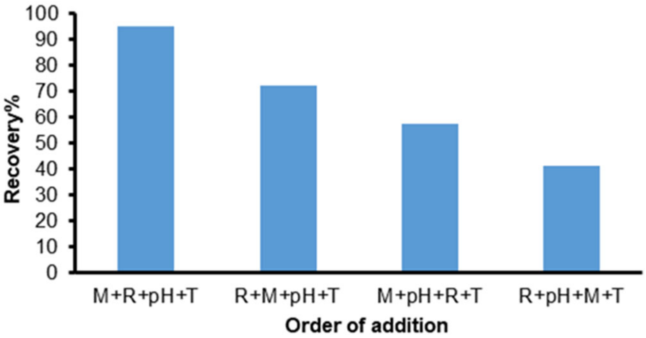

Fig 4. Sequence of addition on Pb(II)-KI-Triton X-114 formation

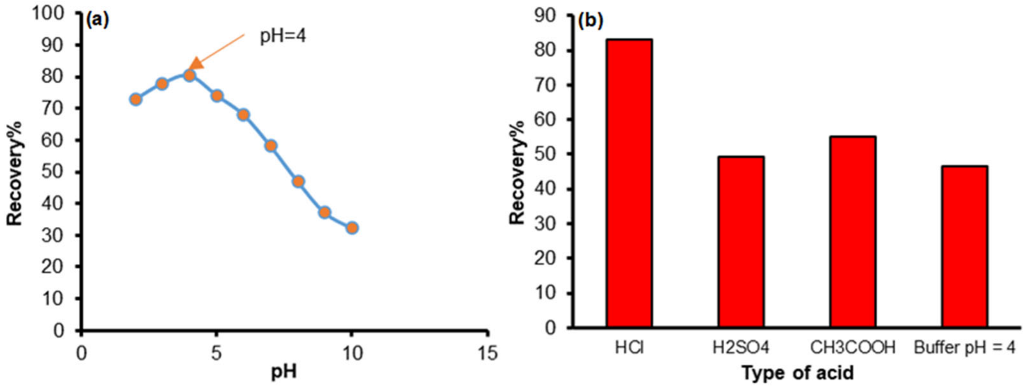

Fig 5. (a) The impact of pH on the Pb(II)-KI-Triton X-114 formation at 424 nm, 10 µg/mL Pb(II), 30 mmol/L KI, and 0.3 mL 2% (v/v) Triton X-114, pH = 4 are the required conditions and (b) type of acid

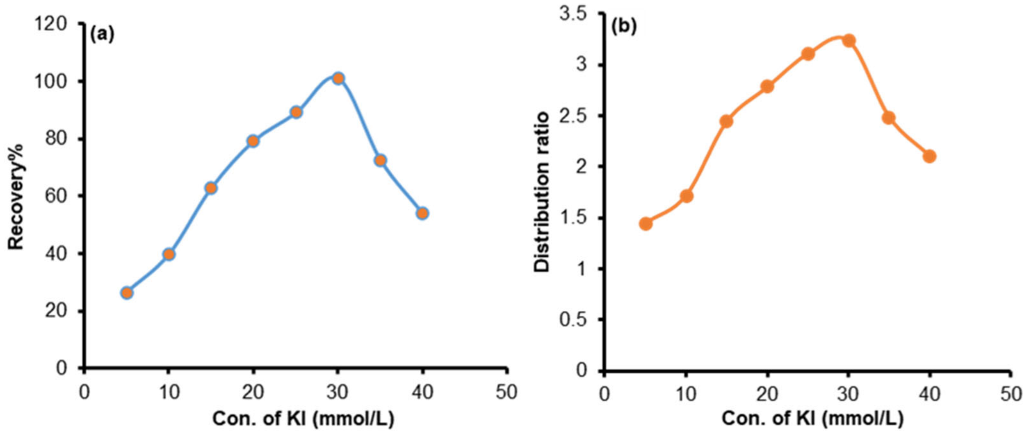

Fig 6. (a) Influence of KI concentration on the Pb(II)-KI-Triton X-114 formation at 424 nm, at 10 µg/mL Pb(II), 30 mmol/L KI, 0.3 mL 2% (v/v) Triton X-114, pH 4 and (b) distribution ratio

was examined. While maintaining the other chemical and physical characteristics. Fig. 6(a) illustrates that as more KI reagent was added the recovery value increased. The reagent quantity that produced the best results was 1 mL (30 mmol/L) of KI reagent [39]. The solution precipitates when KI reagent is added in significant amounts. Fig. 6(b) shows an estimation of the distribution ratio.

Impact of Surfactant Concentration and Type

In CPE, the surfactants are important reagents. They are required for the formation of micelles, which take the substance to be separated. These molecules have a charged or polar non-ionic head and are amphipathic, with a non-polar chain. The most common forms of CPE are the non-ionic varieties. They are water-soluble and their ionization is very weak [40-41]. The type of surfactant employed determines how effective CPE is. In this investigation, specific concentrations of various surfactant types, including Triton X-100, Triton X-114, and Tween 80, were used. The result in Fig. 7(a) demonstrates how surfactant affects the recovery of Pb(II)-KI-Triton X-114 formation. The non-ionic surfactant Triton X-114 is frequently used in CPE. This is because of its benefits characteristics include being commercially available, being highly pure, having a low cloud point temperature, being non-toxic, being inexpensive, and having a high density of the surfactant-rich phase, that makes phase separation by centrifugation easier [42]. The impact of adding Triton X-114 non-ionic surfactant in concentrations ranging from 0.05 to 1 mL on Pb(II) extraction is depicted in Fig. 7(b). The result demonstrates that when the amount of Triton X-114 2% (v/v) is 0.3 mL, the recovery of Pb(II) reaches its maximum limit. At volumes less than 0.3 mL,

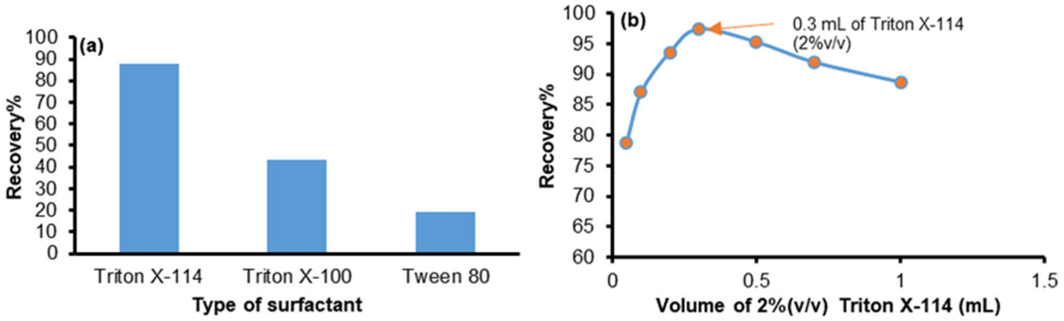

Fig 7. (a) Impact of the types of surfactants and (b) concentration of surfactants on the formation of the Pb(II)-KI-Triton X-114 at 10 mL of 10 µg/mL Pb(II), 1 mL of 30 mmol/L KI, pH = 4, 0.3 mL of Triton X-114 2% (v/v)

the separation was insufficient, owing to incomplete assemblies that did not completely collect the hydrophobic complexes [43]. Therefore, the volume of 0.3 mL of Triton X-114 2% (v/v) was selected as optimum.

Equilibrium Temperature Effect and Incubation Time Effect

For micelle-mediated extraction to be more effective, the solution needs to be warmed above the temperature of the surfactant cloud. The mixture thus divides into two layers. The organic layer, which is primarily made up of surfactant aggregation with hydrophobic groups, and the aqueous layer, which contains free ions and other hydrophilic molecules [40]. To further investigate the effects of temperature, change on the surfactant-rich phase of Pb(II)-KI-Triton X-114 separation, the temperature of the water bath was raised from 40 to 85 °C. At higher temperatures, phase separation becomes more difficult. As a result, the optimal temperature of 50 °C was chosen for future studies. Thermodynamic functions of ΔH_{ex}, ΔG_{ex}, and ΔS_{ex} have also been calculated to be 0.0231, −18.833, and 58.378 J/mol, respectively. A positive value of entropy indicates the stability of Pb(II)-KI-Triton X-114. All of the outcomes are shown in Table 2 and Fig. 8. These results demonstrate how the absorbance signals changed as the temperature rose to 50 °C. In addition, the distribution ratio, extraction percentage, and stability constant were computed.

The shortest incubation time was preferred when using cloud point extraction methods so that good pre-concentration and phase separation can be established quickly [44]. In order to separate the Pb(II)-KI-Triton X-114, the cloud solution was tested for 5 to 25 min while being heated in the water bath. The time for 10 min was found to be the ideal incubation time, and this time was used for subsequent experiments to enhance the extraction methods conditions. Fig. 9 demonstrates the incubation time effect on the recovery

Table 2. The CPE method for Pb(II) is affected by equilibrium temperature at pH (4), 10 mL of 10 µg/mL Pb(II), 1 mL of 30 mmol/L KI, 0.3 mL of 2% (v/v) Triton X-114

Temp K	Abs. aq	Abs. rich-phase	D	Log D	E%	1/T (K)	K_{ex}	log K_{ex}
313	0.034	0.055	1.618	0.209	61.798	0.00319	865.052	2.937
318	0.036	0.059	1.639	0.266	64.835	0.00314	976.563	2.990
323	0.030	0.071	2.367	0.374	70.297	0.00310	1111.111	3.046
328	0.036	0.064	1.778	0.250	64.000	0.00305	771.605	2.887
333	0.037	0.065	1.757	0.245	63.725	0.00300	730.460	2.864
338	0.035	0.063	1.800	0.255	64.286	0.00296	816.327	2.912
343	0.036	0.066	1.833	0.263	64.706	0.00292	771.605	2.887
348	0.037	0.067	1.810	0.258	64.423	0.00287	730.460	2.864
353	0.034	0.065	1.912	0.281	65.657	0.00283	865.052	2.937
358	0.036	0.063	1.750	0.243	63.636	0.00279	771.605	2.887

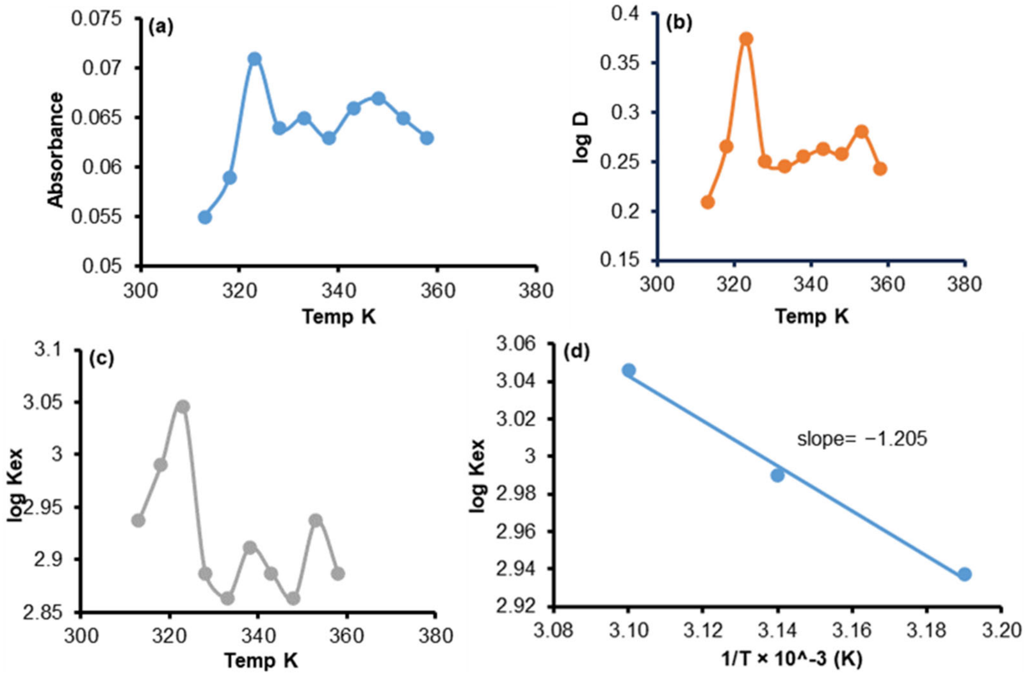

Fig 8. Influence of the equilibrium temperature on the Pb(II)-KI-Triton X-114-CPE technique, pH (4), 10 mL of 10 µg/mL Pb(II), 1 mL of 30 mmol/L KI, and 0.3 mL of 2% (v/v) Triton X-114, where (a) absorbance of rich phase, (b) log D, (c) log K_{ex}, and (d) inverse temperature

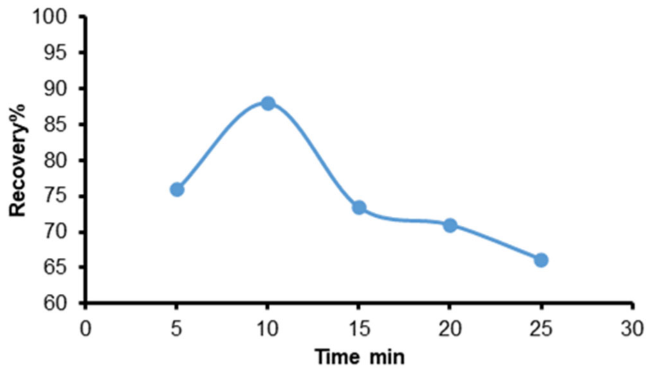

Fig 9. Incubation duration on the Pb(II)-KI-Triton X-114-CPE technique, pH 4, 10 mL of 10 µg/mL Pb(II), 1 mL of 30 mmol/L KI, 0.3 mL of 2% (v/v) Triton X-114, temp. 50 °C, and time 10 min

of Pb(II)-KI-Triton X-114 formation at pH 4, 10 mL of 10 µg/mL Pb(II), 1 mL of 30 mmol/L KI, and 0.3 mL 2% (v/v) Triton X-114, at 50 °C.

Salt Out Effect and Interference Study

According to the salting-out effect, salt addition may reduce the solubility of analytes in aqueous solution and increase their partitioning into adsorbent or organic phases in traditional microextraction. However, when the ionic strength of the medium increases, so do the viscosity and density of the aqueous solution, resulting in a decrease in mass transfer efficiency [45]. By adding 0.5 mL of 15% (w/v) of KCl, KNO_3, Na_2SO_4, and $NaNO_3$ to the solutions of Pb(II), the impact of salts on CPE was examined. The obtained results showed that the presence of various salts caused the absorbance value to reduce. Table 3 displays the impact of salts on the Pb(II)-KI-Triton X-114.

The effects of foreign ions on Pb(II) extraction were investigated. The analytical reagent (KI) may react with this cation and might interact with the analyses and lessen the extraction's efficiency, to carry out this research. After adding 100 µg/mL of interfering ions and 10 µg/mL of Pb(II) to the solution, the procedure was carried out. The obtained results show that the efficacy of CPE is negatively affected by the presence of a large number of species that are regularly detected in water samples. It was found that absorbance increases in the presence of some foreign ions and decreases in other types. Table 4 shows the impact of interference on the extraction of the Pb(II)-KI-Triton X-114.

Table 3. Salts impact on absorbance of PbI_2 solution/pH/4, 10 mL of 10 µg/mL Pb(II), 1 mL of 30 mmol/L KI, 0.3 mL of 2% (v/v) Triton X-114, 50 °C, Time 10 min

Type of addition salt	Con. of addition salt (mol/L)	Abs. aq	Abs. rich-phase	Recovery%	Ere%
Without salt	0.453	0.040	0.070	100%	0.000
KNO_3	1.484	0.037	0.052	74.286	25.714
KCl	2.011	0.048	0.059	84.286	15.714
$NaNO_3$	1.765	0.020	0.036	51.429	48.571
Na_2SO_4	1.056	0.043	0.055	78.571	21.429

Table 4. Influence of different ions on the production of the PbI_2 solution as measured by UV-vis-CPE spectrophotometer. The ratio of salt concentration to Pb(II) ion was 10 µg/mL (Interferent conc./metal conc. µg/mL =100/10= 10 µg/mL)

Interfering	Composition diver's ions compound	Abs rich-phase	Abs. Aq	PbI_2 solution	
				Recovery%	Ere%
Pb(II)	$Pb(NO_3)_2$	0.068	0.046	100	0.00
Cu(II)	$Cu(NO_3)_2·3H_2O$	0.050	0.040	73.529	26.471
Fe(III)	$FeCl_3$	0.075	0.041	110.294	|−10.294|
Co(II)	$Co(NO_3)_2$	0.047	0.037	69.118	30.882
Ni(II)	$Ni(NO_3)_2·6H_2O$	0.055	0.041	80.882	19.118
Cr(III)	$Cr(NO_3)_3·9H_2O$	0.074	0.048	108.824	|−8.824|
Zn(II)	$ZnSO_4$	0.049	0.038	72.059	27.941
Cd(II)	$Cd(NO_3)_2$	0.056	0.037	82.353	17.647

Pb(II) absorbance is 0.085 µg mL^{-1} in the absence of an interfering material

Calibration Curve of Pb(II) via UV-vis-CPE

In order to evaluate the method's linearity under the optimal conditions created by the CPE method for the Pb(II) ion. Table 6 summarizes the Pb(II) calibration curve results by UV-vis-CPE. Pb(II) solutions in various concentrations were produced. The range of Pb(II) concentrations that can be utilized to identify Pb(II) ion using the UV-vis-CPE technique is described in Tables 5 and 6.

Repeatability

Unfortunately, repeatability is commonly interpreted as repeated measurements of the same sample solution, which should be referred to as system precision (or injection precision) [46]. The repeatability test findings relative standard deviation (%RSD) value can be calculated. The UV-vis method's precision for determining Pb(II) ion was tested. The concentrations of Pb(II) were 4 and 10 µg/mL for Pb(II)). In Table 7, the

Table 5. Summary of result for calibration curve of Pb(II) by UV-vis-CPE

Con. Pb(II) (µg mL^{-1})	Abs y_i, n = 3	Mean $\bar{y}$	σ_{n-1}	%RSD	*C.L, $\bar{y}$ + $_{t0.05/2}\ \sigma_{n-1}/\sqrt{n}$
1	0.033, 0.039, 0.032	0.035	0.0038	10.8171	0.035 $\pm$ 0.0094
2	0.042, 0.040, 0.040	0.041	0.0012	2.8171	0.041 $\pm$ 0.0029
4	0.049, 0.050, 0.050	0.050	0.0006	1.1540	0.050 $\pm$ 0.00114
6	0.056, 0.058, 0.060	0.058	0.0020	3.4483	0.058 $\pm$ 0.0050
8	0.065, 0.067, 0.068	0.067	0.0015	2.2806	0.067 $\pm$ 0.0038
10	0.072, 0.079, 0.076	0.076	0.0035	4.6184	0.0761 $\pm$ 0.0087
12	0.085, 0.087, 0.086	0.086	0.0010	1.1628	0.086 $\pm$ 0.0025
14	0.094, 0.096, 0.095	0.095	0.0010	1.0526	0.095 $\pm$ 0.0025
16	0.108, 0.100, 0.106	0.105	0.0042	3.9648	0.105 $\pm$ 0.00103

Table 6. Summary of calibration curve of Pb(II) by UV-vis-CPE

Range of con. (μg/mL)	Equation of calibration curve	r^2	Detection limit (DL) (μg/mL)
1–16	$y = 0.0046x + 0.0309$	0.9993	0.1

Table 7. Pb(II)-KI-Triton X-114 repeatability under ideal conditions

No. of repeating	Con. of metal (μg/mL)	Abs	Mean $\pm$ SD	%RSD
6	4	0.051, 0.049, 0.053, 0.050, 0.054, 0.052	0.051 ± 0.0018	3.633
6	10	0.068, 0.074, 0.069, 0.071, 0.070, 0.073	0.070 ± 0.0023	3.271

Table 8. The concentration of Pb(II) in the lifting and treatment stations in the Karbala city using the CPE and ICP-MS methods

Symbol of station	Abs Mean ± SD, n = 3 /CPE	Con. Pb(II), (μg/mL) CPE	Con. Pb(II), (μg/mL) ICP-MS
Z-1	0.043 ± 0.00493	0.105	<0.001
Z-2	0.044 ± 0.00200	0.114	<.0.001
Z-3	0.046 ± 0.00305	0.131	<0.001
Z-4	0.051 ± 0.00251	0.175	<0.001
Z-5	0.048 ± 0.00200	0.149	0.013
Z-6	0.046 ± 0.00251	0.131	<0.001
Z-7	0.047 ± 0.00404	0.140	0.009
Z-8	0.045 ± 0.00360	0.123	0.133
Z-9	0.044 ± 0.00208	0.114	0.155
Z-10	0.047 ± 0.00529	0.140	0.127

The value of the concentration of the Pn(II) in ICP-MS analysis (< 0.01) was approximately equal to zero. Pre-concentration factor for Pb(II) in CPE method N=25 (preconcentration factor)

%RSD for Pb(II) are listed. This technique can result in a relative standard deviation percentage of less than 5%.

Application

The amount of Pb(II) ions in wastewater samples from the lifting and treatment plants was determined using the proposed method. The results obtained for the concentration of Pb(II) using the new method were compared with the results of preliminary tests conducted to estimate the concentration of Pb(II) ion in wastewater using ICP-MS technology. A comparison was made of the Pb(II) ion concentration in the pre-treatment lifting stations and treatment plants with the acceptable Pb(II) ion concentration value 0.01 μg mL^{-1} according to the WHO (2011), where it was found that the Pb(II) concentration was within the acceptable limits except for the Z-8, Z-9, and Z-10 stations outside the acceptable limits. Table 8 and Fig. 10 demonstrate the amount of Pb(II) present in the lifting and treatment stations in the city of Karbala using the CPE and ICP-MS techniques.

Statistical Analysis of Data

To find out if there are significant differences between the new method CPE and the standard method ICP-MS to determine Pb(II) ion in wastewater samples. T-test and F-test were performed, with a confidence limit of 95% as shown in Table 9. The results obtained for the two methods indicated that there is a slight difference between them. where the p value of the T-test and the F-test was $p > 0.05$ is a significant difference. Where the ICP-MS method depends on identifying the Pb(II) element in all formations (metal, ion, and salts) while the CPE method identifies Pb(II) in the form of the ion only. The reason for the increase in concentration values is due to the pre-concentration method with UV-vis spectrophotometer determines the concentration of elements to low concentrations that may reach μg/mL, while the ICP-MS technique determines the concentration of elements to very low concentrations that may reach ng/mL.

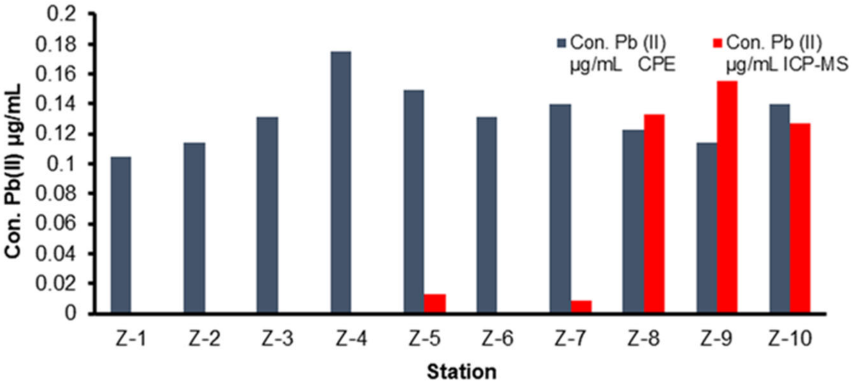

Fig 10. Pb(II) ion level estimation in the lifting and treatment stations

Table 9. Statically data T-test and F-test

Mean ± SD CPE	Mean ± SD ICP-MS	One sample T-test CPE	One sample T-test ICP-MS	T-calculated for pair T-test= d/(Sd/√n)	F-test= $(SD1)^2/(SD2)^2$
0.1322 ± 0.020346	0.043700 ± 0.065825	18.993	1.618977	3.82212	10.467

Value $T_{0.05}$ (n-1) n=10(2.26), $F_{0.05}$ (n-1) n=10(2.98), p-value < 0.05 sig. difference, p-value > 0.05 non sig. difference. Acceptable Pb(II) concentration value in wastewater = 0.01 µg mL^{-1} according to the WHO

CONCLUSION

A new technique has been established to estimate the amount of some chemical pollutants in wastewater that depends on micellar-mediated phase separation to pre-concentrate the level of Pb(II) in wastewater and measure it using a UV-vis spectrophotometer. The method is based on the extraction of Pb(II) from KI as a reagent using Triton X-114 as a surfactant. The new method is cheap, easy, and environmentally friendly. It is also very sensitive by reaching the lowest detection limit. The detection limit for Pb(II) with the proposed method was 0.1 µg/mL, and the %RSD was 3.633, linearity was maintained in the range of Pb(II) concentration from 1.0 to 16 µg/mL, respectively.

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CONFLICT OF INTEREST

The authors do not have conflict of interest.

AUTHOR CONTRIBUTIONS

Mohammed Nasser Hussain conducted the experiment and did the calculations. Mohammed Nasser Hussain, Ahmed Fadhil Khudhair and Hussain Jawad Ahmed drafted and edited the document. The final draft of this manuscript was approved by all authors.

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