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Mohammed Nasser Hussain
Department of chemistry, college of science, University of Kerbala, Karbala, Iraq

Ahmed Fadhil Khudhair
Department of chemistry, college of science, University of Kerbala, Karbala, Iraq

Hussain Jawad Ahmed
Department of chemistry, college of science, University of Kerbala, Karbala, Iraq

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ORIGINAL STUDY

Evaluation of Iron Element by New CPE Method and (ICP-MS) in the Wastewater of the Lifting and Treatment Stations

Mohammed N. Hussain, Ahmed F. Khudhair*, Hussain J. Ahmed

Department of Chemistry, College of Science, University of Kerbala, Karbala, Iraq

Abstract

A new micell-mediated phase separation technique has been created to preconcentrate levels of iron before measuring it using a UV−Vis spectrophotometer. The method is based on the extraction of iron from the 1-Nitroso-2- naphthol reagent using Triton X-114 in a cloud point extraction procedure, where the method's ideal conditions were attained. 0.2 mL of Triton X-114, 2.887 mmol. L^{-1} of 1-Nitroso-2-naphthol reagent, pH = 8, 60 °C. The Iron detection limit for the method is 0.05 μg. mL^{-1}, and the RSD% is 4.728 μg. mL^{-1}. Linearity was maintained in the iron concentration range of 0.25−16 μg. mL^{-1}. Furthermore, the interference impact of certain cations was assessed. The proposed method was successfully applied to determine the Iron ion in the wastewater in ten different stations. Eight lifting stations and two treatment stations in the center and district of Al-Hurr in the Karbala city. In addition, the Iron element in the wastewater from the study sites was also directly evaluated using inductively-coupled plasma mass technology.

Keywords: Chemical pollutants, Cloud point extraction method, ICP-MS, Iron ion, Wastewater

1. 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, byproducts [2,3]. Environmental issues are caused by the over usage of these contaminants and their untreated discharge [4]. Other extremely dangerous substances, and poisonous additionally, metal ions have a tendency to bioaccumulate and might gradually leach into rivers, endangering aquatic life [5]. Heavy metals can exist naturally in the environment, ecosystem and soils [6]. Wastewater pollution is frequently caused by mining, recovery, and other economic activities that use several harmful metal ions [7]. It is well known that iron and copper have a substantial impact on environmental chemical and biological processes. Their presence is crucial to environmental cycles and not just essential [8]. Iron is one of the most vital substances in biological systems, the environment, business, and medicine. It is crucial for oxygen transport, immunity, growth regulation, and cell differentiation in the human body [9,10]. This ion can cause poisoning and even death in high doses, though. Because of this, it is crucial to quickly and accurately determine the presence of Fe (III) in biological and environmental samples [11,12]. There are several methods used to treat wastewater, including highly effective nanoparticles to remove contaminants [13−15]. Iron can be identified using ultraviolet−visible (UV−vis) spectrophotometric methods, which are crucial analytical instruments because they are rapid, easy to use, and have good precision and accuracy [16]. 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

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* Corresponding author.
E-mail address: ahmmed.mutar@uokerbala.edu.iq (A.F. Khudhair).

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selectivity. For this goal, various methods were developed, including solid phase extraction (SPE) [17,18]. Liquid−liquid extraction (LLE). [19], ion exchange [20]., and co-precipitation [21]. 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 foundation of CPE is the turbid behaviour of surfactant in aqueous solutions at temperatures approaching the cloud point [22,23]. CPE is an analytical technique that focuses based on the pre-concentration and separation of minute amounts of often hydrophobic organic molecules and elements, which has the ability to raise the detection limit and other analytical parameters [24]. The transfer of the analyte from one phase, typically a complex matrix phase, to another phase is a step in the separation technique [25−27]. A modified form of the LLE called cloud point extraction (CPE) employs micelles as the extraction solvent [28,29]. One of the preferred methods for preconcentrating and separating organic and inorganic metal ions is cloud point extraction [30,31]. Because it adheres to the tenets of green chemistry, it has drawn a lot of interest. CPE has a number of benefits over conventional extraction techniques, including: simplicity, avoiding the use of harmful chemical solvents in large quantities [32−35] The lifting and treatment plants in the city of Karbala were studied and some trace elements were estimated. The results showed an increase in the concentration of elements (lead, chromium, and cadmium) [36]. In this study, cations (Na^+, Ca^{2+}, K^+, Mg^{2+}) and anions (SO_4^{2-}, Cl^-, HCO^{3-}, NO^{3-}) were studied. Seven wells located in Karbala were selected, and studying the possibility of using well water for irrigation. It was found that this water cannot be used for irrigation purposes [37]. In a previous study, the quality of drinking water in the city of Baghdad was evaluated, comparing the quality of tap water and bottled water. TDS levels exceed palatable water levels (>600 ppm) in some areas. In addition, the sulphate concentration was relatively high and the hardness contents Cl^- and Pb^{2+} in both tap water and bottled water were within standard limits [38].

The study aimed to estimate the Iron ion in the wastewater of the eight pumping and two treatment stations using a new alternative UV−Vis spectrophotometic method after using the approach of cloud point extraction and direct estimation of Iron ion by inductively coupled plasma technique (ICP-MS) in wastewater.

2. Experimental section

2.1. Materials and methods

Deionized water was used during the testing, and all of the chemical reagents used were of the analytical reagent quality. The stock Fe (III) solution was created by dissolving 0.290 g of $FeCl_3$ (B.D.H) (Analar) in 100 mL of deionized water. 20% (v/v) Triton X-114 (AMRESCO) (98.0%), 2.887 mmol. L^{-1} 1-Nitroso-2-Naphthol Reagent (B.D.H) (98.0%), and 0.1 mol. L^{-1} of sodium hydroxide (B.D.H) (96.0%). Single Beam - Visible Spectrophotometer Model 721, (China), ICP-MS Agilent 7500 Made in USA.

2.2. CPE- UV−vis spectro. method

According to the typical procedures of the cloud point extraction method, 10 ml of 10 μg. mL^{-1} Fe (III) was added to 0.5 mL of (2.887 mmol. L^{-1}) (1-Nitroso-2-naphthol reagent placed in a centrifugal test tube (15 mL), resulting in the formation of a green complex with maximum absorption wavelength 707 nm. The pH was then adjusted to 8 using a (0.1 mol. L^{-1} NaOH) solution after which 0.2 mL of 20% (v/v) Triton X-114 was added to the solution. A thermostatic water bath at 60 °C and 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 4000 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 practical procedure was shown in Fig. 1 and the suggested mechanism for producing the Fe^{3+}-1-Nitroso-2-naphthol complex was shown in Fig. 2.

2.3. ICP-MS procedure

The most often used and recommended techniques are inductively coupled plasma-mass spectrometry (ICP-MS) and inductively coupled plasma-optoelectronic spectroscopy (ICP-OES) because of their well-known advantages of sensitivity, selectivity, and multi-element analytical capabilities [39−41]. In tests ranging from 1 ng. L^{-1} to high mg. L^{-1} ranges, ICP-MS shown to be more accurate than other methods at detecting components of biological or clinical origin at the lowest concentrations with good linearity, The exceptional sensitivity of [42].

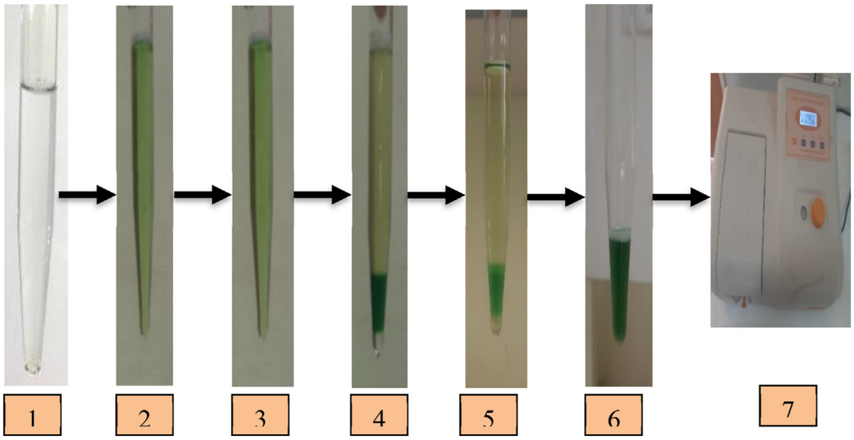

Fig. 1. Cloud point extraction process for Fe (III): (1) Fe (III) solution, (2) Mixing with 1-nitroso-2-naphthol reagent (3) Triton X-114 surfactant added during cloud solution creation. (4) phase separation following centrifugation, (5) cooling with an ice bath, (6) a rich phase of separation, (7) Use a Vis spectrophotometer to measure.

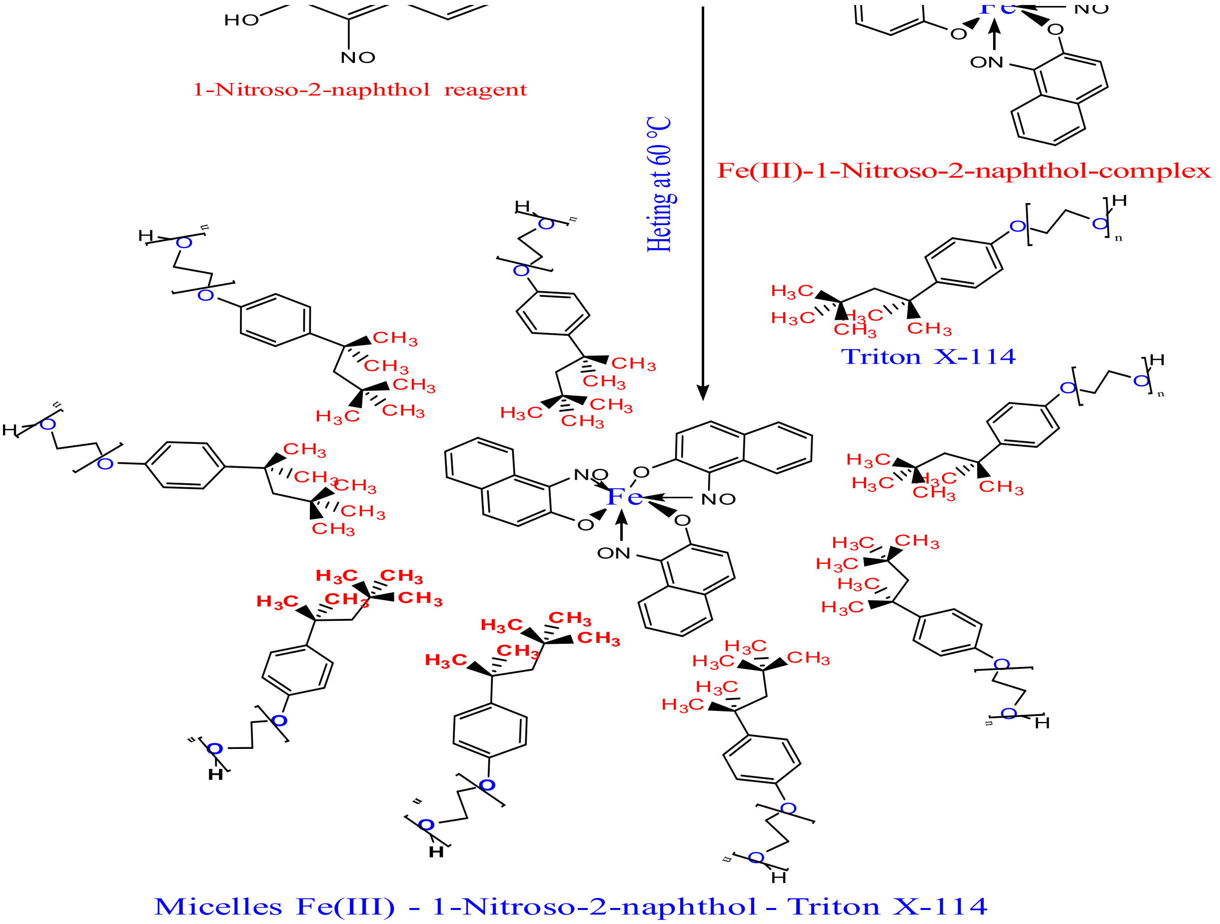

Fig. 2. Proposal mechanism of Fe (III) – 1-Nitroso-2-naphthol complex separation by CPE method.

The concentration of iron ion was measured for wastewater samples, where 1 mL was taken from the sample and a digestion process was carried out on it by adding nitric acid 1 mol. L^{-1} After the samples were entered and injected into the device, the concentration reading was taken directly.

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

2.4. Sampling

Wastewater samples were taken from Ten different stations. Eight lifting stations and two treatment stations in the centre and district of Al-Hurr in Karbala Governorate. Samples were taken from each station at a depth of 1–3 m. Wastewater samples were collected in pre-washed 1 L polyethylene containers. Wastewater samples were filtered using filter paper (Huathman 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. In Table 1 was shown the sampling stations information and Fig. 3 shows the sequence of lifting and treatment stations. The information system data for sewage systems was provided by the Directorate of Karbala Sewage (see Fig. 4). Information system data for sewage systems and the locations of lifting and treatment stations in the study area were provided by the Karbala Sewerage Directorate, unit Geographic Information Systems (GIS).

3. Results and discussion

3.1. Cloud point extraction method

Due to its simplicity and low cost, the UV–Vis Spectrophotometer approach was employed to calculate the amount of iron ions in wastewater [43]. Because wastewater contains little iron and a pre-concentration technique is needed to boost the method's sensitivity, coupling with the cloud point extraction method is necessary.

3.2. Absorption spectrum

UV–Vis spectrums of the Iron (III), and 1-Nitroso-2-naphthol reagent and Fe (III)- 1-Nitroso-2-naphthol -Triton X-114 complex were measured. The wavelength of Iron (II), 292 nm, was shown to have a considerable shift in the visible area., the complex's highest wavelength, 707 nm, was attained. The1-Nitroso-2-naphthol reagent's wavelength was 312 nm. Fig. 5 Shows the absorption spectrum of Iron ion and 1-Nitroso-2-naphthol and Fe (III)- 1-Nitroso-2-naphthol complex.

4. Optimization condition

4.1. Order of addition

The order in which the solutions of Fe (III) (10 μg. mL^{-1}, 10 mL) and 1-Nitroso-2-naphthol (2.887 mmol. L^{-1}, 0.5 mL) were added was one of the most crucial aspects that was evaluated. Triton X-114 20% (v/v) was added to the complex formation in the amount of 0.2 mL, with the order of additions determined by the optimum absorption value. (R + M + pH + T). As depicted in Table 2. Order of Addition effects on Fe (III), 1-Nitroso-2-naphthol complex absorbance.

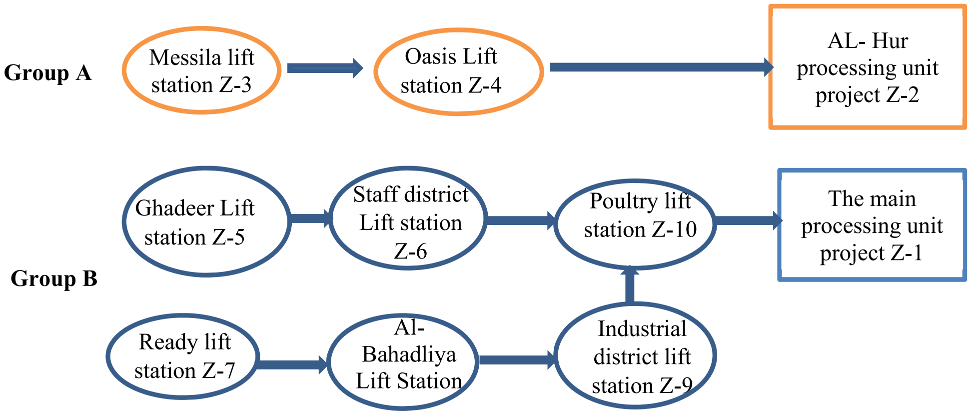

Fig. 3. The lifting and treatment stations A: Lifting and treatment stations for Al-Hurr District B: Lifting and treatment stations in the centre of Karbala city.

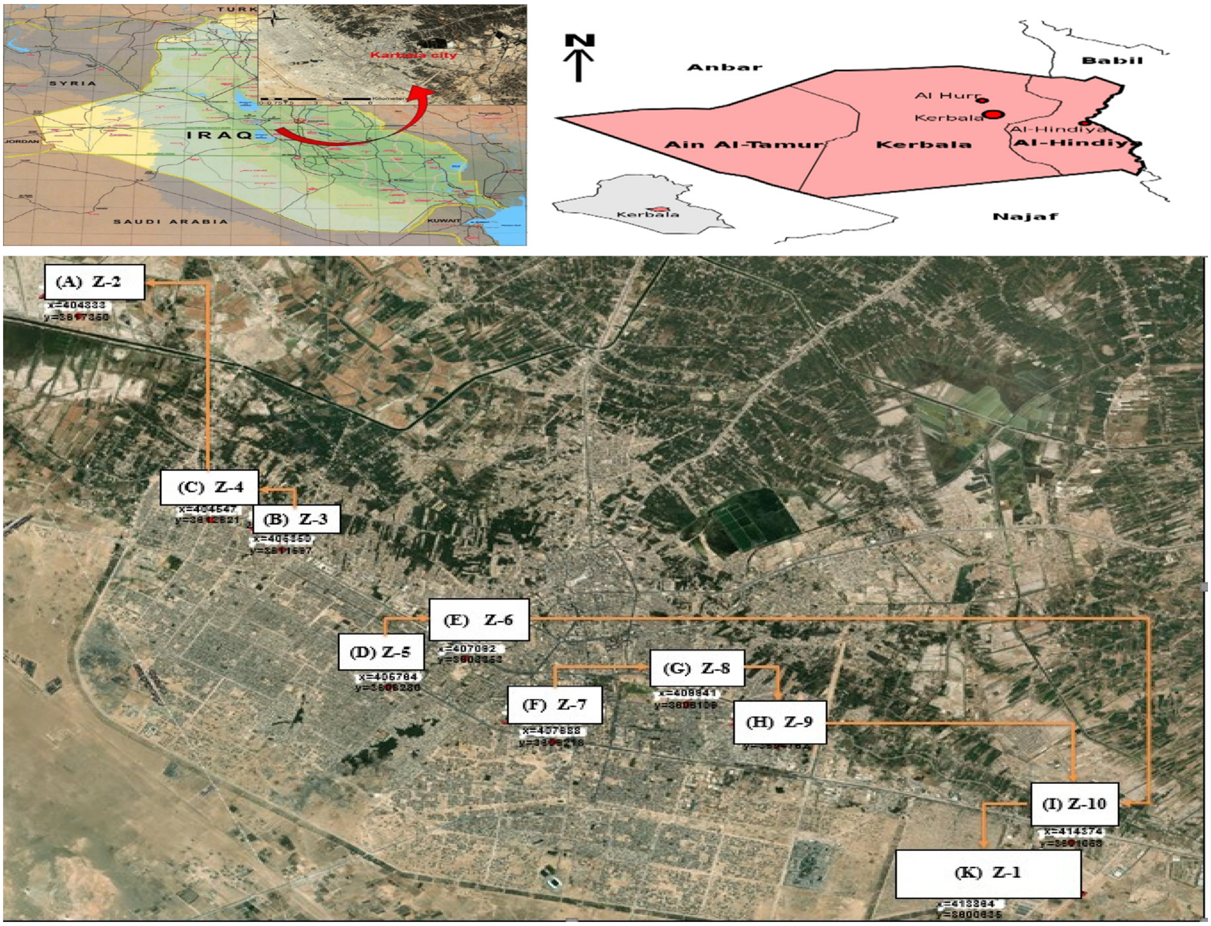

Fig. 4. Wastewater sampling site, (a): AL- Hur processing unit project Z-2 (b): Messila lift station Z-3 (c): Oasis Lift station Z-4 (d): Ghadeer Lift station Z-5 (e): Staff district Lift station Z-6 (f): Ready lift station Z-7 (g): Al-Bahadliya Lift Station Z-8 (h): Industrial district lift station Z-9 (i): Poultry lift station Z-10 (k): The main processing unit project Z-1.

4.2. Influence of pH

The creation of complex molecules and the chemical stability of solutions depend on the pH, which is one of among the essential elements influencing the success of extraction using the cloud point extraction method. The pH of the combination was adjusted between the ranges of 2 and 10 using 0.1 mol. L^{-1} HCl and 0.1 mol. L^{-1} NaOH solution. and the pH was then measured using a pH meter. The absorbance rose whenever the pH was equal to 8. However, at pH levels higher than 8, the complexation reaction is likely to fail because an iron hydroxide precipitate was formed. Shown in Fig. 6 The pH effect on Fe (III)-1-Nitroso-2-naphthol complex absorbance.

4.3. 1-Nitroso-2-naphthol concentration's impact

The impact of reagent concentration on absorbance value was investigated using a range of 1-Nitroso-2-naphthol solution concentrations (0.288, 0.577, 1.155, 2.130, 2.887, 4.331, and 5.775 mmol. L^{-1}) and volumes of (0.05,0.1,0.3,0.5,0.7, and 1 mL). While

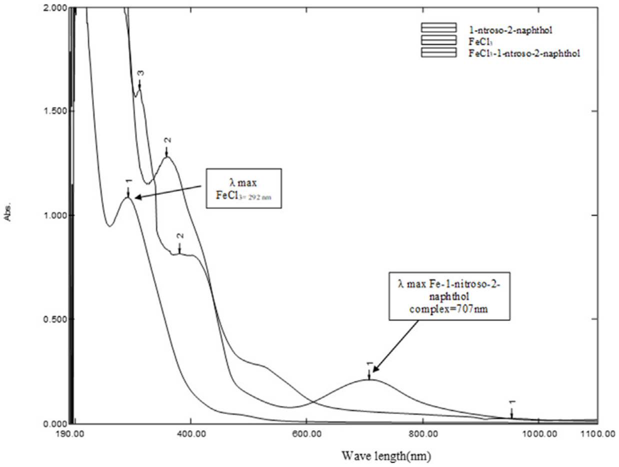

Fig. 5. UV−Vis spectrums of Fe (III),1-Nitroso-2-naphthol reagent and Fe (III)- 1-Nitroso-2-naphthol -Triton X114 complex.

Table 2. The influence of sequence addition on the absorbance of the Fe (III), 1-Nitroso-2-naphthol complex.

Order addition	Aaq	Aaq (Mean)	Abs rich phase	As (Mean)
M + R + pH + T	0.016,0.015, 0.017	0.016	0.185,0.180, 0.179	0.181
R + M + pH + T	0.026,0.020, 0.021	0.022	0.237,0.240, 0.233	0.237
M + pH + R + T	0.026,0.022, 0.021	0.023	0.123,0.128, 0.136	0.129
R + pH + M + T	0.024,0.026, 0.023	0.024	0.198,0.200, 0.210	0.203

Aaq/Absorption of an aqueous solutions. As/Absorbance of rich-phase, M:(Fe (III), R: 1-Nitroso-2-naphthol, T: Triton X-114).

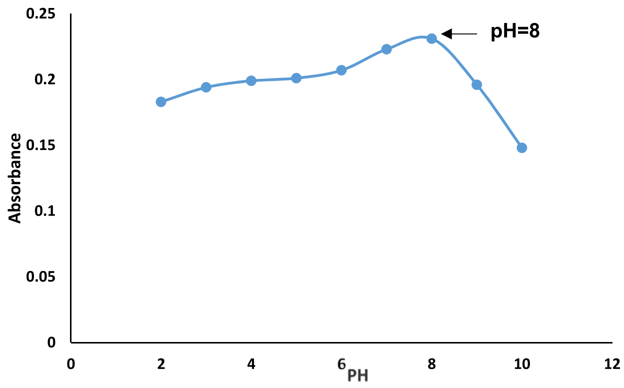

Fig. 6. The influence of pH on Fe (III)- 1-Nitroso-2-naphthol complex absorption at 707 nm. Conditions:10 μg. mL^{-1} Fe^{3+}, 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, 0.2 mL 20% (V/V) Triton X-114.

maintaining the other chemical and physical characteristics. The absorbance value increased as more reagent was applied. The reagent concentration that gave the best results was 0.5 mL (2.887 mmol. L^{-1}). When substantial amounts of reagent are added, the complex formed will precipitates. as seen in Fig. 7 (a, b, c and d). Effect of the concentration and volume of the 1-Nitroso-2-naphthol reagent on the absorbance of the Fe (III)- 1-Nitroso-2-naphthol complex.

4.4. Impact of surfactant concentration and kind

The effectiveness of cloud point extraction depends on the type of surfactant used. Triton X-100, Triton X-114, and Tween 80, among other surfactant types were utilized in this study at particular doses. The effects of surfactant on the absorbance of Fe (III)- 1-Nitroso-2-naphthol complex formation are shown in Fig. 8 (a). One non-ionic surfactant that is widely used in CPE is Triton X-114. This is due to its advantageous properties, which include being readily available in the marketplace, being extremely pure, having a low cloud point temperature, being non-toxic, being affordable, and having a high density of the surfactant-rich phase, which facilitates phase separation by centrifugation [44]. The effect of adding Triton X-114 non-ionic surfactant at doses ranging from 0.05 to 1 mL on Iron (III) extraction is shown. It was found that the complex's absorbance increased as Triton X-114 volume increased up to 0.2 mL, but that the complex's absorbance actually decreased as Triton X-114 surfactant volume increased past that point. The effect of surfactant type and concentration on the absorbance of the Fe (III)- 1-Nitroso-2-naphthol complex is shown in Fig. 8 (a) and (b).

4.5. 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. To further investigate the effects of temperature change on the surfactant-rich phase of Fe (III)- 1-Nitroso-2-naphthol complex separation, the temperature of the water bath was raised from (40–85) °C. All of the outcomes are shown in Table 3. It demonstrates how the absorbance signals changed as the temperature rose to 60 °C Fig. 9.

When using cloud point extraction techniques, the shortest incubation duration was favoured. In order to swiftly establish effective pre-concentration and phase separation. The cloud solution was tested while being heated in the water bath for five to 25 min in order to separate the Fe (III)- 1-Nitroso-2-naphthol complex. The outcomes shown in Fig. 10 (a) showed that 10 min was the optimal incubation time. and this time was used for further experiments to improve the conditions of the extraction technique. Fig. 10 (a) and (b). Demonstrate Incubation Time Effect on Absorbance of Fe (III)- 1-Nitroso-2-naphthol complex at pH (8), 10 mL of 10 μg. mL^{-1} Fe (III), 0.5 mL of 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, and 0.2 mL 20%(v/v) Triton X-114, 60 °C and Distribution ratio determined.

4.6. Salt out effect and interference study

In cloud point extraction, salt is added to the sample solution to improve phase separation and

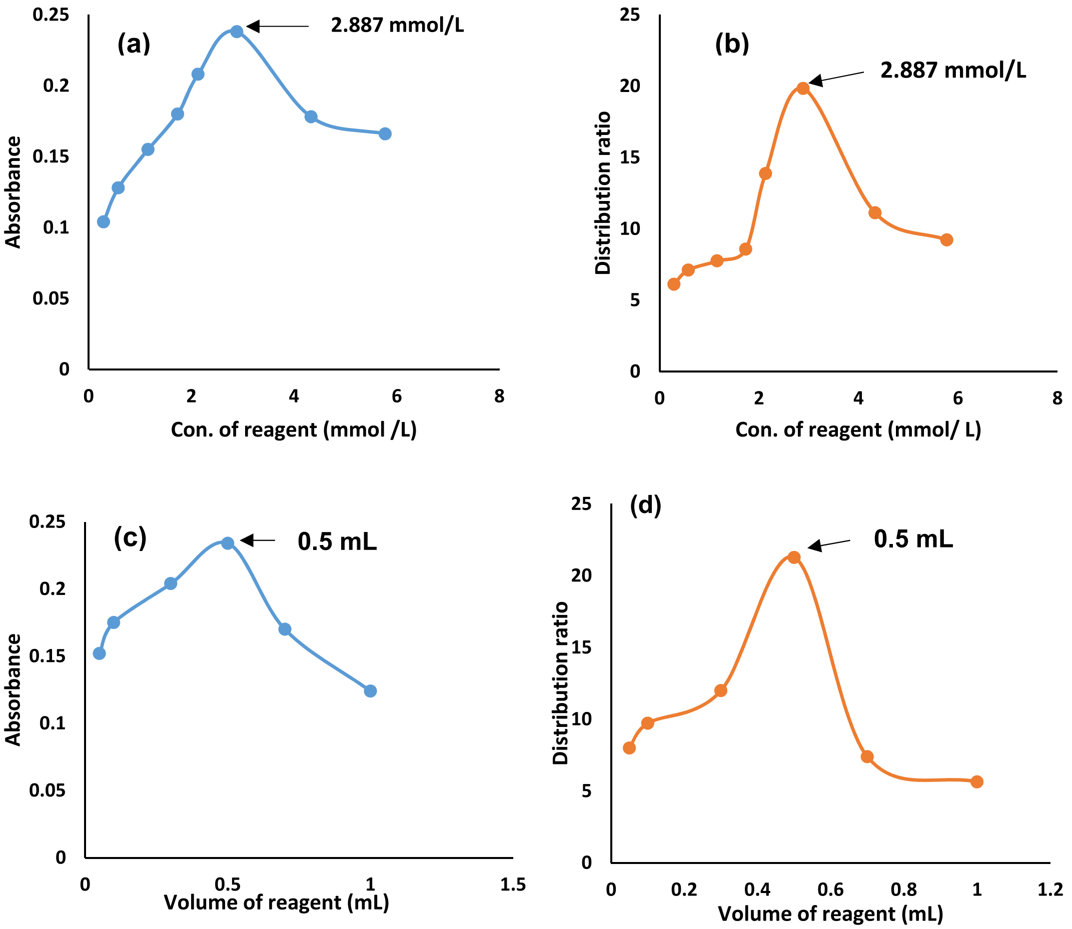

*Fig. 7. **(a)**. Influence of 1-Nitroso-2-naphthol concentration and, **(b)** Distribution ratio estimation. **(c)** Volume on Fe (III)- 1-Nitroso-2-naphthol complex absorbance at 707 nm. at: 10 μg. mL^{-1} Fe (III), 2.887 mmol. L^{-1} reagent, 0.2 mL 20% (V/V) Triton X-114, pH = 8 **(d)** Distribution ratio estimation.*

increase the mass transfer of the analyte from the aqueous phase to the surfactant-rich phase. By adding 0.5 mL of 15% w/v (KCl, KNO_3, Na_2SO_4, and $NaNO_3$) to the solutions of Iron (III), the impact of salts on CPE was examined. The obtained results showed that the presence of various salts caused the absorbance value to increase. Table 4 Display the impact of salts on the Fe (III)- 1-Nitroso-2-naphthol

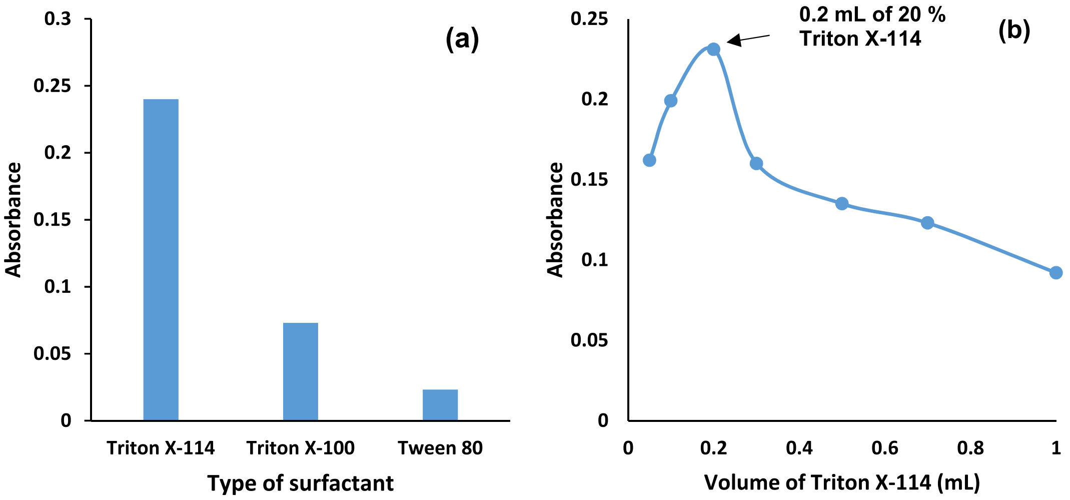

*Fig. 8. **(a)**: Impact of the types of surfactants and **(b)**: Concentration of surfactants on the formation of the Fe (III)- 1-Nitroso-2-naphthol complex. at 10 mL of 10 μg. mL^{-1} Fe (III), 0.5 mL of 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, and pH/8.*

Table 3. Impact of equilibrium temperature to the CPE technique of Fe (III) at pH (8), 10 mL of 10 μg. mL^{-1} Fe (III), 0.5 mL of 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, 0.2 mL of 20% (v/v) Triton X-114.

No	Temperature. (°C)	Abs. aq	Abs rich-phase	D	E%
1	40	0.026	0.114	4.385	81.430
2	45	0.025	0.138	5.520	84.663
3	50	0.022	0.165	7.500	88.235
4	55	0.020	0.186	9.300	90.291
5	60	0.012	0.229	19.083	95.021
6	65	0.018	0.216	12.000	92.308
7	70	0.021	0.201	9.571	90.540
8	75	0.026	0.183	7.077	87.619
9	80	0.035	0.175	5.000	83.333
10	85	0.031	0.170	5.484	84.577

Table 4. Salts impact on absorbance of Fe (III)- 1-Nitroso-2-naphthol complex, at pH (8), 10 mL of 10 μg. mL^{-1} Fe (III), 05 mL of 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, 0.2 mL of 20% (v/v) Triton X-114, 60 °C, Time 10 min.

Type of addition Salt	Abs. aq	Abs rich-phase	*Recovery%	*Ere%
Without salt	0.013	0.210	–	–
KNO_3	0.026	0.448	213.333	113.333
KCl	0.019	0.289	137.619	37.619
$NaNO_3$	0.019	0.334	159.048	59.048
Na_2SO_4	0.020	0.366	174.286	74.286

complex. Investigation of the effects of foreign ions on Fe (III) extraction was done. The analytical reagent (1-Nitroso-2-naphthol) may react with this cation. and might interact with the analytes and lessen the extraction's efficiency, to carry out this research. After adding 100 μg. mL^{-1} of interfering ions and 10 μg. mL^{-1} of Fe (III) to the solution and the procedure was carried out. The obtained results show that the efficacy of cloud point extraction is

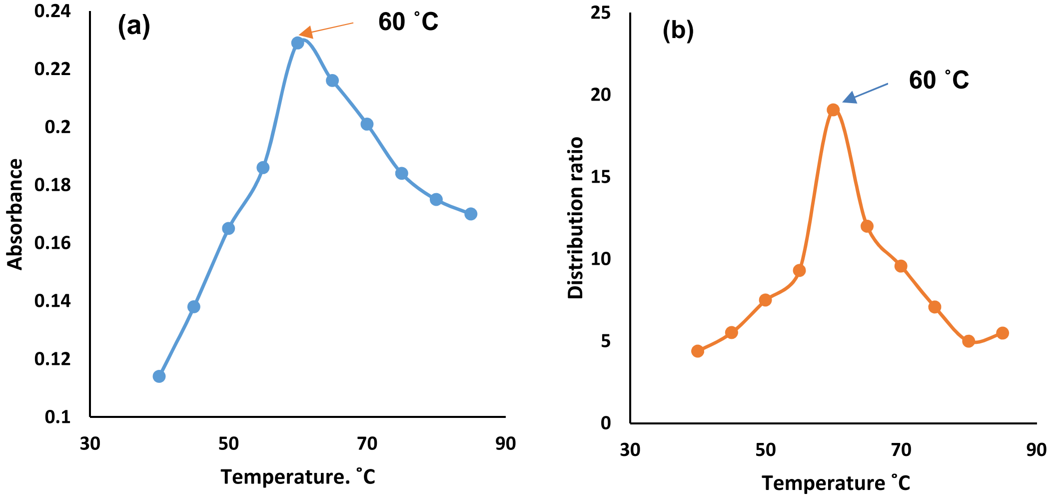

*Fig. 9. **(a)**. Influence of the equilibrium temperature to the CPE technique of Fe (III) at pH (8), 10 mL of 10 μg. mL^{-1} Fe (III), 0.5 mL of 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, 0.2 mL of 20% (v/v) Triton X-114, **(b)** Distribution ratio estimation.*

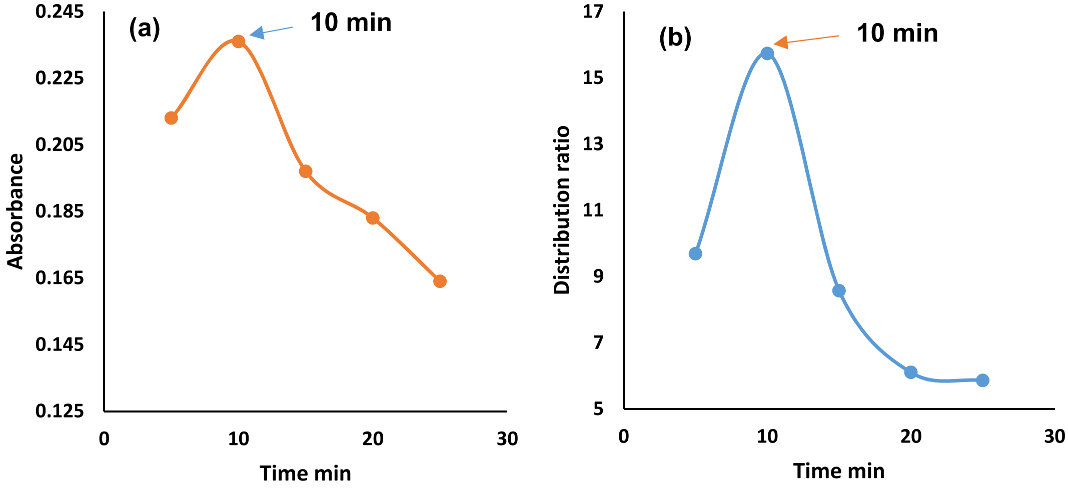

*Fig. 10. **(a)**. Influence of the Incubation Time to the CPE technique of Fe (III) at pH (8), 10 mL of 10 μg. mL^{-1} Fe (III), 0.5 mL of 2.887 mmol. L^{-1} 1-Nitroso-2-naphthol, 0.2 mL of 20% (v/v) Triton X-114, **(b)** Distribution ratio estimation.*

Table 5. Influence of different ions on the production of the Fe (III)- 1-Nitroso-2-naphthol complex as measured by UV–Vis-CPE spectrophotometer. The ratio of interferent concentration to Iron (III) ion was 10 μg. mL^{-1} (Interferent conc./metal conc. μg. mL^{-1} = 100/10 = 10 μg. mL^{-1}.

Interfering	Composition diver's ions compound	Abs rich-phase	Abs. aq	Fe (III)-reagent complex	
				Recovery%	*Ere%
Fe (III)	$FeCl_3$	0.208	0.011		
Pb (II)	Pb $(NO_3)_2$	0.275	0.019	132.212	32.212
Cu (II)	Cu $(NO_3)_2$ $3H_2O$	0.254	0.014	122.115	22.115
Co (II)	Co $(NO_3)_2$	0.236	0.018	113.462	13.462
Ni (II)	Ni $(NO_3)_2$ $6H_2O$	0.246	0.018	118.269	18.269
Cr(III)	Cr $(NO_3)_3$ $9H_2O$	0.224	0.014	107.692	7.693
Zn (II)	$ZnSO_4$	0.226	0.015	108.654	8.654
Cd (II)	Cd $(NO_3)_2$	0.216	0.019	103.846	3.846

Absorbance of the Iron (III) without the addition of interference 0.208 μg. mL^{-1}.

Table 6. The results of the Calibration curve of Fe (III) by UV–Vis- CPE.

Con Fe (III) μg. mL^{-1}	Abs y_i	Mean $\bar{y}$	$\sigma_{n-1\ (SD)}$	*RSD%	*C.I $\bar{y} + {}_{t0.05/2}\ \sigma_{n-1}/\sqrt{n}$
0.25	0.064, 0.066, 0.063	0.064	0.0015	2.344	0.0015 ± 0.0037
0.5	0.075, 0.073, 0.067	0.072	0.0042	5.833	0.072 ± 0.0104
1	0.083, 0.071, 0.089	0.081	0.0092	11.358	0.081 ± 0.0229
2	0.087, 0.095, 0.107	0.096	0.0101	10.521	0.096 ± 0.0251
4	0.125, 0.116, 0.129	0.123	0.0067	5.447	0.123 ± 0.0166
6	0.141, 0.149, 0.162	0.151	0.0106	7.020	0.151 ± 0.0263
8	0.173, 0.186, 0.169	0.176	0.0089	5.057	0.176 ± 0.0221
10	0.198, 0.215, 0.226	0.213	0.0141	6.620	0.2131 ± 0.0350
12	0.235, 0.241, 0.250	0.242	0.0075	3.099	0.242 ± 0.0186
14	0.275, 0.261, 0.270	0.269	0.0071	2.639	0.269 ± 0.0176
16	0.289, 0.313, 0.291	0.298	0.0133	4.463	0.298 ± 0.0330

C.I: confidence interval, SD: Standard deviation, RSD%: Relative Standard deviation and Critical value of $t_{0.05/2\ at\ (n\ =\ 3)} = 4.303$.

Table 7. Summary of calibration curve of Fe (III) by UV–Vis- CPE.

No	Range of Con. μg. mL^{-1}	Equation of Calibration curve	R^2	Detection limit (DL) μg. mL^{-1}
1	0.25–16	y = 0.0147x +0.064	0.9991	0.05

significantly adversely affected by the presence of a large number of species that are regularly detected in water samples. Where it was found that the absorbance increased in the presence of foreign ions. Table 5. The interference's impact on the extraction of the Fe (III)- 1-Nitroso-2-naphthol complex was assessed.

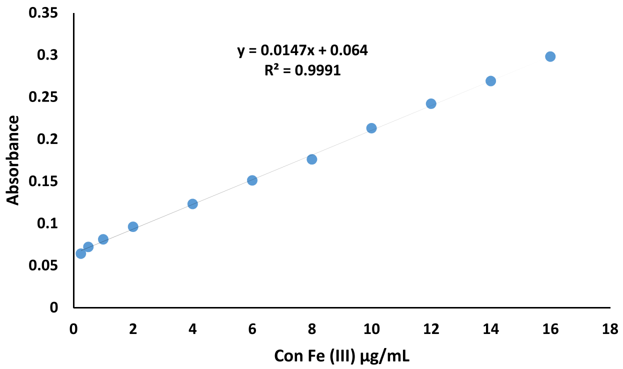

Fig. 11. Fe (III) – 1-Nitroso-2-naphthol complex in rich phase calibration curve by UV. Vis -CPE technique.

4.7. Calibration Curve of Fe (III) via UV–Vis- CPE

It is important to assess the linearity of the approach under ideal conditions created by the cloud point extraction approach for the Fe (III) ion. Table 6. Results for the Fe (III) calibration curve by UV–Vis-CPE are summarized here. Fe (III) solutions in various concentrations were produced. The range of Fe (III) concentrations that can be utilized to identify Fe (III) ion. Using the UV–Vis - CPE technique is described in Table 7 and Fig. 11.

Table 8. Fe (III)-1-Nitroso-2-naphthol complex repeatability under ideal conditions.

No. of repeating	Con. of metal μg. mL^{-1}	Abs	Mean ± SD	RSD %
6	4	0.124, 0.117, 0.110 0.113, 0.121,0.112	0.116 ± 0.005492	4.728
6	10	0.210, 0.215, 0.221 0.197, 0.218, 0.228	0.215 ± 0.01061	4.939

Table 9. The concentration of Iron ion 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. Fe (III) μg. mL^{-1} CPE	Con. Fe (III) μg. mL^{-1} ICP-MS
Z-1	0.105 ± 0.00640	0.109	0.0100
Z-2	0.108 ± 0.00656	0.118	0.0100
Z-3	0.103 ± 0.00808	0.104	0.1700
Z-4	0.104 ± 0.01044	0.107	0.0300
Z-5	0.113 ± 0.01457	0.131	0.0163
Z-6	0.109 ± 0.00513	0.120	0.1071
Z-7	0.111 ± 0.00907	0.126	0.0936
Z-8	0.101 ± 0.01069	0.098	0.0835
Z-9	0.117 ± 0.01040	0.142	0.1078
Z-10	0.118 ± 0.00608	0.145	0.0996

Pre-concentration factor for Fe (III) in CPE method N = 25 (preconcentration factor).

4.8. Repeatability

The repeatability test findings' Relative Standard deviation (RSD%) value can be calculated. The UV–Vis method's precision for determining Fe (III) ion was tested. The concentrations of Fe (III) were 4 and 10 μg. mL^{-1} for Fe (III). In Table 8 The Relative Standard deviation (RSD%) for Fe (III) are listed. This technique can result in a Relative Standard deviation percentage of less than 5%.

4.9. Application

The amount of Iron (III) ions in wastewater samples in the lifting and treatment stations was determined using the proposed method. The results obtained for the concentration of Iron using the new method were compared with the results of preliminary tests conducted to estimate the concentration of Iron ion in wastewater using inductively coupled plasma-mass (ICP-MS) technology. A comparison was made of the Iron ion concentration in the lifting stations and treatment stations with the acceptable Iron ion concentration value (0.3 μg. mL^{-1}) according to the World Health Organization

Table 10. Comparison of the CPE-UV-Vis method for estimating iron (III) ion concentration with a previously published CPE-UV-Vis method [45].

Type	CPE-UV-Vis New method	CPE-UV-Vis Published method
Sample	Wastewater	Urine
Colour	green	green
Reagent	1-Nitroso-2-naphthol	4, 4-biphenyldiamine Benzidine
λ max	707 nm	425 nm
LOD	0.05 μg. mL^{-1}	0.05 μg. mL^{-1}
RSD%	4.728	2.955
R^2	0.9991	0.9650
Linear regression equation	$y = 0.0147x + 0.064$	$y = 0.4694 X + 0.1269$
Con. of Fe (III) μg. mL^{-1} in Calibration Curve	0.25–160.00	0.25–3.00

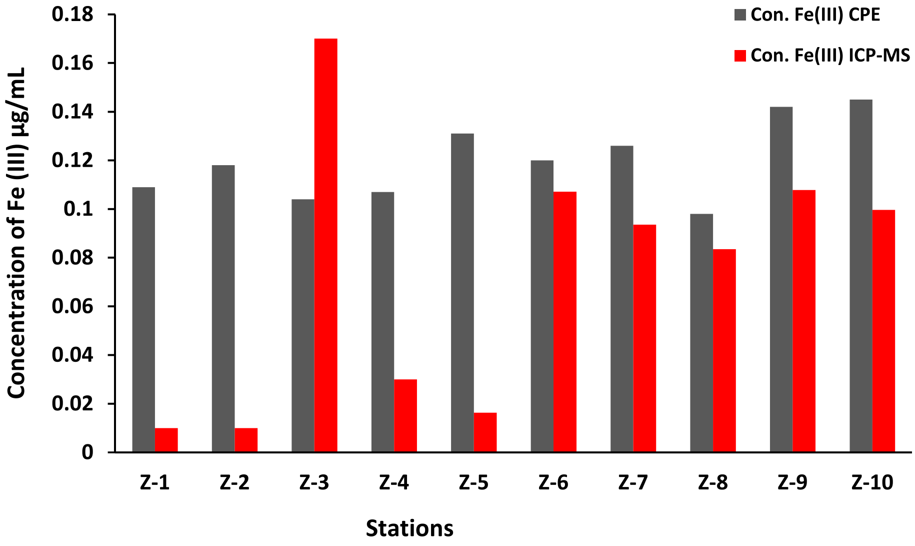

Fig. 12. Iron ion level estimation, series 1 CPE and series 2 ICP methods.

Table 11. Statically data t-test and F-test.

No	Mean ± SD CPE	Mean ± SD ICP-MS	One sample T-test CPE	One sample T-test ICP-MS	t-calculated for Pair T-test = $d/(Sd/\sqrt{n})$	F-test= $(SD1)^2/(SD2)^2$
1	0.120 ± 0.015986	0.072790 ± 0.053776	−35.607	−13.361	2.712	11.297

Value $t_{0.05}$ (n-1) n = 10(2.26), F0.05 (n-1) n = 10(2.98), P-value < 0.05 sig. Difference, p-value > 0.05 non sig. Difference. Acceptable Iron (III) concentration value in wastewater = 0.3 μg. mL^{-1} according to the World Health Organization and United State Environmental Protection Agency (U.S EPA).

and United State Environmental Protection Agency (U.S EPA), where it was found that the Iron concentration was within the acceptable limits for the all stations in Table 9 and Fig. 12. Table 9 demonstrates the amount of Iron present in the lifting and treatment stations in the city of Karbala using the CPE and ICP-MS techniques.

Table 10 was showed a comparison between the proposed new method using CPE-UV-Vis spectrophotometer to estimation the concentration of iron (III) ion in wastewater samples with a previous published method (CPE-UV-Vis) to estimate iron in urine samples. It was found that the proposed method has high sensitivity and can be used to estimation iron ion in other samples.

4.10. Statistical analysis of data

To find out if there are significant differences between the new method CPE and the standard method ICP-MS in order to determine Iron ion in wastewater samples. T test and F test were performed, with a confidence limit of 95% as shown in Table 11. The results obtained for the two methods indicated that there is a considerable difference between them. Where the p value of the T test and the F test was $p > 0.05$ is a significant difference, the null hypothesis was rejected and the CPE method can be used as an alternative technique to quantify Iron ions in the wastewater samples. While the ICP-MS method depends on identifying the Iron element in all formations (metal, ion and salts) at a high confidence limit of 95%.

5. Conclusion

Using Triton X-114 as a surfactant and 1-Nitroso-2-naphthol as a reagent, the cloud point extraction technique was effectively utilized to determine the presence of Iron in water and wastewater samples using a UV–Vis spectrophotometer. The suggested method has the following benefits: simplicity, speed, and low cost of analysis. The technique presented here is quick and highly reproducible. The use of a cheap surfactant prevented the use of harmful organic solvent extraction, which would have caused problems with waste management. When compared to other methods, the suggested cloud point extraction method is preferable since it has a lower RSD and lower detection Limits.

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