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Spectrophotometric determination of 4-ethylphenol using cloud point extraction in different water samples

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Abstract

This study describes the development of an easy, inexpensive, accurate and fast spectrophotometric technique for the determination of 4-ethylphenol. The primary method involves converting 3-nitroaniline to the diazonium salt and then reacting with 4-ethylphenol in an alkaline medium. It is yellow in color and has a maximum absorption at 426 nm. It obeys Beer's law in a linear range of 5-12 μg mL^{-1} with a correlation coefficient of 0.9994 and a molar absorptivity 6.0024×10^3 L. mol^{-1}. cm^{-1}. Cloud point extraction was used to quantify trace amounts of phenol using TritonX-114 as a surfactants, and subsequently, a measurement procedure was performed using a UV spectrophotometer. The value of the correlation coefficient was 0.9998, the molar absorbance was 1.04676×10^4 L. mol^{-1}. $cm^{-1}$and the linear range was 2-11 μg mL^{-1}. The limits of detection and quantification were determined to be 0.42103 and 0.140345 μg mL^{-1}, respectively. The proposed method was successfully used for the determination of phenol in different environmental samples.

Keywords: CPE, Determination, Environmental water samples, 4-ethylphenol, Spectrophotometry.

Introduction

Phenolic compounds are in the byproducts of diverse sectors such as petroleum refining, wood production, resin manufacturing, medicines, cooking processes, paint, pulp, plastics, paper, and petrochemicals[1-4]. Improper disposal of these substances poses significant health hazards and jeopardizes the well-being of humans, animals, and the ecosystem [1,5]. The US Environmental Protection Agency (EPA) and Canada's National Pollutant Release Inventory (NPRI) designated phenolic compounds as priority pollutants.[6,7]As outlined by the EPA, the allowable threshold for phenol in surface water is under 0.001 Mg mL^{-1}, while the potentially harmful concentration range is situated between 9 and 25 Mg mL^{-1} [8]. Many liquid and gas chromatographic techniques, as well as electrochemical techniques, have been developed for the selective and sensitive analysis of phenol in aqueous media from various types of samples[9,10]. Even though their analytical performance is excellent (low detection limit, high selectivity and accuracy, etc.), they are unsuitable for phenol analysis in industrial laboratories due to the high cost of analyses, multi-step and laborious sampling procedures, long work times, expensive equipment, and so on [11]. According to a review of the literature, numerous methods for quantifying phenols have been documented. Potentiometric titration is one of them[12]. Potentiometric visible, spectrophotometric, spectrofluorimetric, quenched continuous fluorescence, LC-ESIMS, HPLC, and flow-injection chemiluminescent techniques [13-15.] One method for determining phenols and other compounds is the diazotization coupling reaction. Diazonium salts play a crucial role as fundamental ingredients in the production of aromatic substances, pharmaceuticals, colorants, and various other organic compounds[16,17]. Due to their electrolytic characteristics, these salts are valuable in analytical procedures as they can interact with other electron-rich compounds such as the phenolic compounds. and amino-acids[18]. Furthermore, they contain colored compounds that can be used to estimate very tiny amounts of organic elements, light absorption, or polygraphy by measuring current propagation[17].

In optimizing conditions, cloud point extraction has been demonstrated where these techniques are able to extract and pre-concentrate a wide range of organic compounds from the aqueous phase. The cloud point extraction techniques result in fast extraction, high preconcentration factor, and the removal of the need to use toxic and environmentally unfriendly organic solvents that make it advantageous compared to other techniques, CPE involves the creation of two homogenous phases, namely a water-deficient phase and an organic-enriched phase [13]. CPE relies on the phase tendencies of nonionic surfactants when present in aqueous solutions[18,19]. Demonstrating phase segregation upon temperature elevation or introduction of a salt agent[20].The separation and concentration of surfactants through CPE are increasingly recognized as significant and practical applications within the realm of analytical chemistry.[21-23] This technique is simple, highly sensitive, non-expensive, and is environmentally conscious, as it utilizes a minimal sample size for analysis.[24,25]

The aim of this study is to create a simple, inexpensive, fast, precise, accurate, and environmentally friendly process for determining phenols using cloud point extraction.

Materials and Methods

Instrumentation and Apparatus

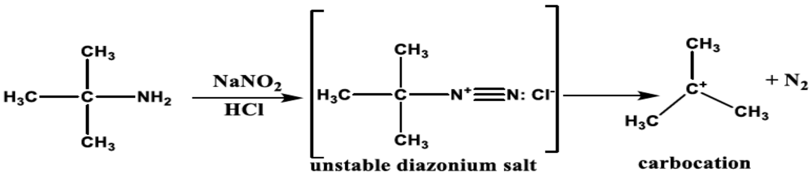
Sensitive electric balance, Sartorius, Germany, Bench centrifuge, type (MSE) manufacture/England, a thermostatic water bath /Germany and UV-Visible Spectrophotometer Shimadzu Model(160A) Japan with a response time of 0.1s was employed for spectrophotometric analysis. A quartz cell with a 3 mL internal volume and 1cm route length was employed to test absorbance.

Standard Solutions Preparation

This study's reagents and ingredients were all of excellent quality, 3-nitro aniline was purchased from company (BDH). To dissolve and prepare solutions, distilled water was used. A stock solution 1000 µg mL^{-1} of 4-ethylphenol and 3-nitro aniline were prepared by dissolving 0.1 g in pure water and completing to the mark in a100 mL volumetric flask with distilled water. A sodium nitrite solution 1% was prepared by dissolving 1.0 g of $NaNO_2$ in distilled water and completing to the mark in a100 mL volumetric flask , 1% urea solution was made by dissolving 1.0 g of urea in 100 mL of distilled water. 1M of Phosphoric acid, H_2SO_4, HCl, and CH_3COOH and 1M of sodium hydroxide, sodium bicarbonate, potassium hydroxide, and barium hydroxide were prepared by dissolving an appropriate amount in distilled water, 10% V/V of each [TritonX-100, TritonX-114, and Tween 20] was prepared By dissolving 10 mL in distilled water and completing to the mark in 100 mL volumetric flask .

Azo Coupling Procedure in General:

The azo coupling product that has been prepared is introduced into a 10 mL volumetric flask while placed in an ice bath, followed by adding 1.0 mL of 3-nitro aniline, 1.0 ml of HCl, 1.0 mL of sodium nitrite ,1.0 mL of urea, 1.0 mL of 4-ethyl phenol ,and 1.0 mL of sodium hydroxide. The absorption is then determined using UV-VIS, The spectra of maximum wavelength absorption are analyzed .Examples of coupling reactions are shown in Scheme 1[26].



Scheme 1. azo coupling reactions

Procedure of Cloud Point Extraction

The cloud point test consists of a number of procedures: in a series of 10ml volumetric flask, the ideal volumes obtained previously are added. [1.3 mL nitro aniline, 1.0 mL HCl, 1.3 mL $NaNO_2$, 1.0 mL urea, 1.5 mL $NaHCO_3$, 1.0 mL 4-ethyl phenol, and 1.0 mL 10% (v/v) Triton X114]. Then the volume was completed to the mark with distilled water. The volumetric contents were moved to a centrifuge test tube, and for 20 minutes, the combination was placed in a bath of water at 60^0, before being centrifuged at 4000 rpm for a period of twenty minutes. Then after 1 minute in a bath with ice to increase viscosity, the micelles separated easily, the separated sediments were dissolved in 3ml of ethanol, and the absorbance was recorded with a UV-VIS spectrophotometer.

Recommended Procedure for Environmental Water Samples

The samples of waste water was collected from different places : Rustamiya, Tap water , under Al doura bridge, Diyala bridge, and kept in plastic bottles then it's filtered with filter papers ,first to prepare (10, 20 µg mL^{-1}) of 4-ethyl phenol ,in 100 mL volumetric flask take (1,2 ml) from(1000 µg mL^{-1}) 4-ethy phenol and complete the volume with distilled water, then in a series of 10 mL volumetric flask the optimal volumes were adding [1.3 mL nitro aniline, 1.0 mL HCl, 1.3 mL $NaNO_2$, 1.0 mL urea,(10,20) µg mL^{-1} of 4-ethyl phenol, 1.5 mL $NaHCO_3$], and complete the volume with distilled water, the absorption was measured with spectrophotometer, then the same procedure follow but with waste water and the absorbance was measured. After that the absorbance was measured with cloud point technique by add all optimal volumes in series of 10 mL volumetric flask [1.3 mL nitro aniline, 1.0 mL HCl, 1.3 mL $NaNO_2$, 1.0 mL urea, (10,20 µg ml^{-1}) of 4-ethyl phenol, 1.5 mL $NaHCO_3$,10% [v/v] tritonx114] and complete the volume with waste water.[22]

Results and Discussion

Absorption Spectra

The basic research demonstrates the diazotization process of 3-nitro aniline and coupling with 4-ethyl phenol to form a yellow color mixture at λ max 426nm. The spectrum of absorption of yellow color versus a blank is shown in Fig. 1.

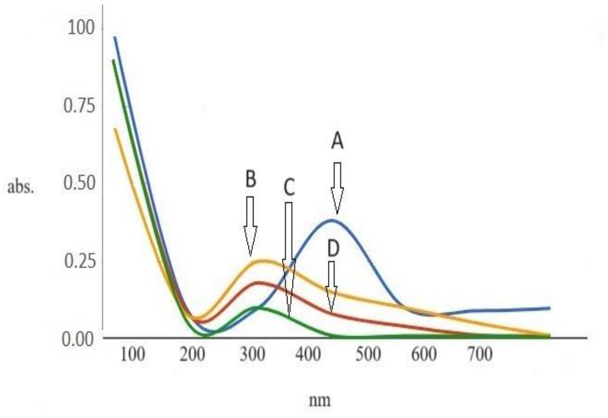


Figure 1. (A)Absorption of the Resulting Dye's Spectra of 4-etyl phenol versus blank, (B) 4-ethyl phenol versus the ethanol,(D) the reagent 3-nitro aniline versus ethanol, (C) the blank versus distilled water.

Analyzing the Optimization Diazonium Salt Reaction

The degree of absorption of colored azo products was influenced by a variety of factors such as acid type and volume, volume of sodium nitrite, volume of reagent, and sodium hydroxide volume. The effect of several acids (HCl, H_2SO_4, H_3PO_4, and CH_3COOH) (1M), the production of diazonium salt was investigated, and the outcomes are shown in Table.1, Fig.2, the best acid is HCl and the best volume of (1M) hydrochloric acid was 1.0 mL which ranged from 0.2 to 2.0 mL as shown in Table 2 , Fig. 3, The optimum volume of acid is followed in the next analysis.

Table1. The impact of acid type

Type of acid	HCl	H_2SO_4	CH_3COOH	H_3PO_4
Abs.λmax 426 nm	0.902	0.372	0.001	0.082

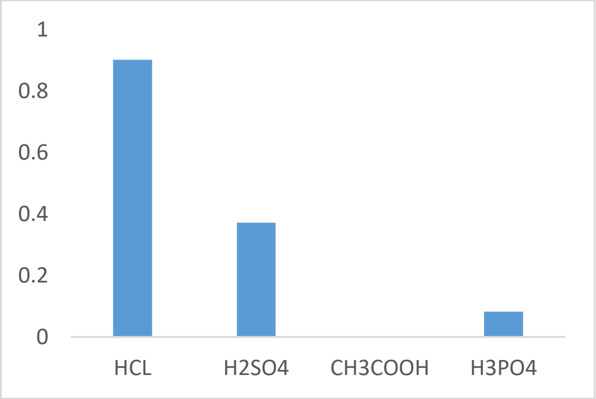


Figure 2. Effect of different type of acids on color intensity.

Table 2. Optimum Volume of 1M HCl

Volume of 1M HCl/mL	Absorbance at λmax =426
0.2	0.781
0.4	0.802
0.6	0.847
0.8	0.856
1	0.919
1.3	0.555
1.5	0.492
1.7	0.398
2	0.283

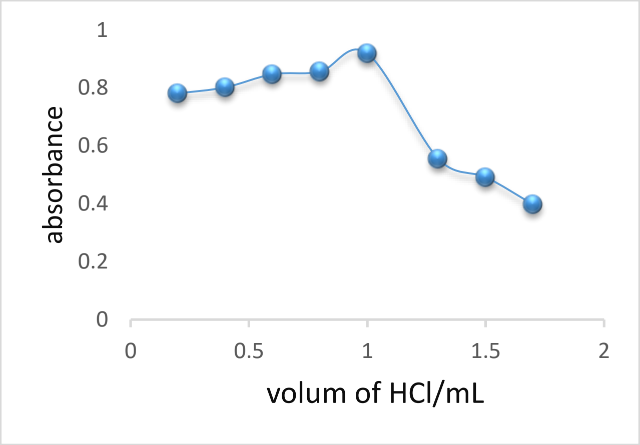


Figure 3. The acid volume impact

The effect of different bases on the process of synthesis of the azo compound (1M) of KOH, NaOH, Br(OH)2, and NaHCO3 was investigated. The outcomes demonstrate that sodium bicarbonate was the best base, as shown in Table 3, Fig. 4. the best absorbance was obtained by adding (1.5 mL) of sodium bicarbonate to different volumes of 1M NaHCO3, which ranged from 0.2 to 2.0 mL, as shown in Table 4, Fig. 5. The optimum volume of base is followed in the next analysis.

Table 3. the impact of bases type

Type of base	NaOH	$NaHCO_3$	KOH	$Br(OH)_2$
Abs λmax 426nm	0.999	1.13	0.140	0.078

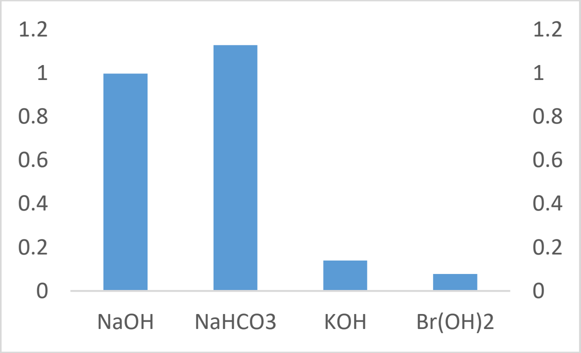


Figure 4. Effect of base volume

Table 4. Optimum Volume of 1M $NaHCO_3$

Volume of 1M $NaHCO_3$/mL	Absorbance at λmax =426
0.2	0.36
0.4	0.43
0.6	0.56
0.8	0.64
1	0.78
1.3	1.02
1.5	1.18
1.7	0.99
2	0.81

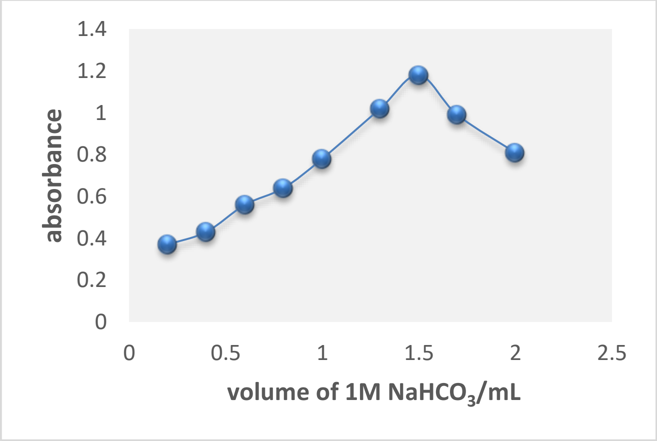


Figure 5. Effect of $NaHCO_3$ volume

The effect of sodium-nitrite concentration was studied through varying the volumes of $NaNO_2$ solution used in the diazotization method from 0.2-2.0 mL and discovering that 1.3 mL presented the best absorption, as shown in Table 5, Fig. 6. The optimum volume of $NaNO_2$ is followed in the next analysis.

Table 5. Optimum Volume of 1% $NaNO_2$

Volume of 1%$NaNO_2$ mL	Absorbance at λmax =426
0.2	0.31
0.4	0.33
0.6	0.36
0.8	0.43
1	1.07
1.3	1.20
1.5	1.112
1.7	1.10
2	1.08

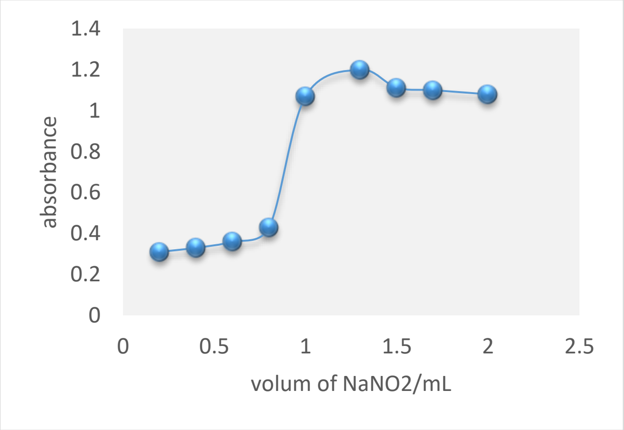


Figure 6. Effect of 1% sodium nitrite

A variety of (1%) urea volumes ranging from 0.2-2.0 mL were used to empty out the excess of nitrite; as shown in Table 6, Fig. 7, one milliliter of urea was enough to remove the remaining amount. The optimum volume of urea is followed in the next analysis.

Table 6. Optimum Volume of 1%urea

Volume of 1% urea	Absorbance of 1% urea
0.2	1.4
0.4	1.5
0.6	1.57
0.8	1.58
1	1.59
1.3	1.53
1.5	1.52
1.7	1.51
2	1.48

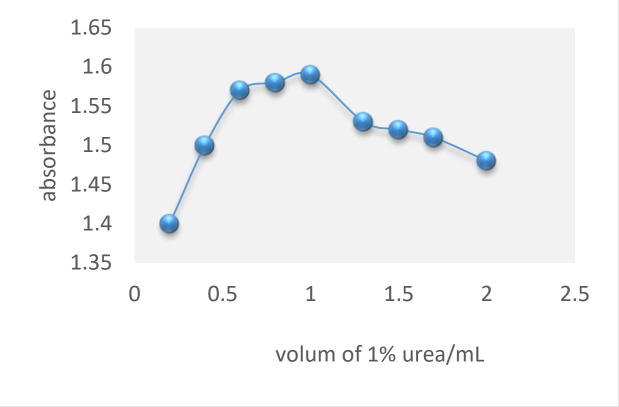


Figure 7. Effect volume of urea (1%)

According to Table 7, Fig. 8, 1.3 mL of 3-nitro aniline Reagent used to produce a high absorbance at λmax 426nm.

Table 7. Optimum Volume of (1000 μg ml^{-1}) reagent.

Volume of (1000 μg ml^{-1})3-nitro aniline	Absorbance of 3-nitro aniline
0.2	0.73
0.4	0.81
0.6	1.34
0.8	1.39
1	1.55
1.3	1.62
1.5	1.60
1.7	1.52
2	1.48

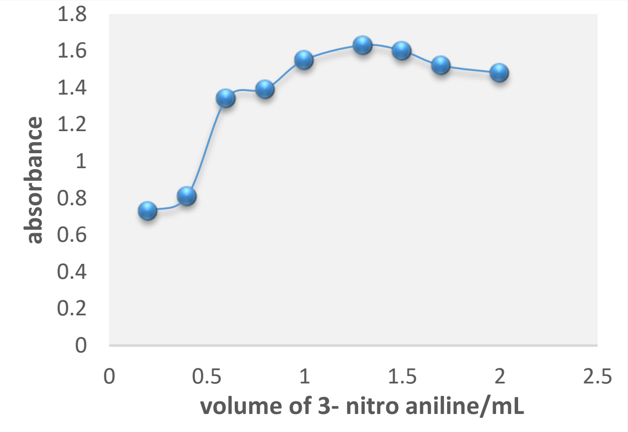


Figure 8. Results of the Optimal Volume of Reagent (3-nitro aniline) (1000 μg ml^{-1})

Stoichiometric Determination of Product

To determine the stoichiometry of the 4-ethylphenol ratio, The method of mole ratio was used by add a series of different volumes are prepared (0.1-3.5) mL from ($3*10^{-4}$) 3-nitro aniline ,with 1.0 mL of ($3*10^{-4}$) 4-ethyl phenol with the optimal conditions of [1.0 mL HCl, 1.3 mL $NaNO_2$, 1.0 mL urea , 1.5 mL $NaHCO_3$],and the continuous variation method (job's method) was use by add series of different volumes are prepared(0.1-0.9) from each reagent and phenol with the optimal conditions [1.0 mL HCl, 1.3 mL $NaNO_2$, 1.0 mL urea , 1.5 mL $NaHCO_3$]. The results showed that the ratio of 4-ethyl phenol to reagent is 1:1 as shown in Figs. 9 and 10.

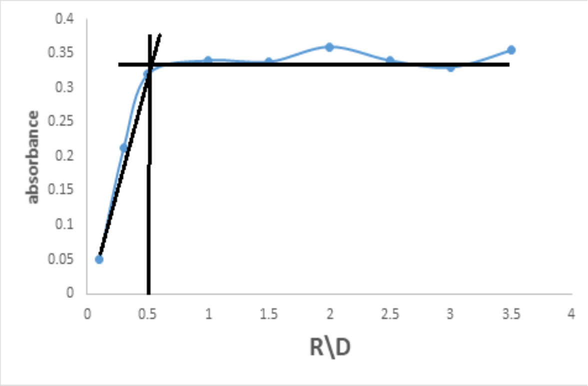


Figure 9. mole ratio of 4- ethyl phenol

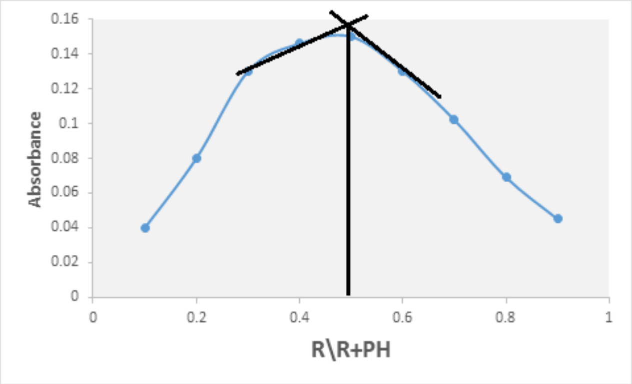


Figure 10. The continuous variation

The Calibration Curve for a 4-ethyl phenol Complex

A small amount of 10 mL solution with an increasing concentration of 4- ethyl phenol is prepared by adding [1.3 mL 3-nitro aniline, 1.0 mL HCl, 1.3 mL $NaNO_2$,1.0 mL urea, (5–12) μg ml^{-1} 4 ethyl phenol, 1.5 mL $NaHCO_3$] to several sets of 10 mL volumetric flasks set in an ice bath, Fig. 11. The phenol is then spectrophotometrically measured at its maximum wavelength, Table.8 shows the precision and accuracy of the proposed method for the determination of standard of complex.

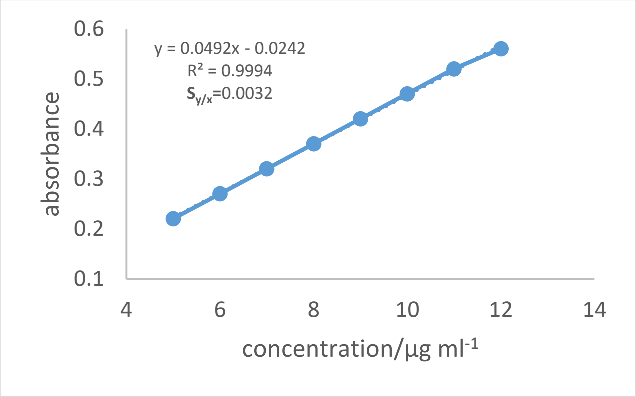


Figure 11. calibration curve of 4-ethyl phenol

Table 8. Precision and accuracy of the proposed method for the determination of standard of complex (n=4)

Amount Taken (μg mL^{-1})	Amount found (μg mL^{-1}) (mean±SD)	C.L. at 95%	Recovery%	$E_{rel.}$%	RSD %
5	4.96±0.0180	4.96±0.0287	99.20	0.8	7.358
7	6.9±0.0178	6.9±0.0284	98.57	1.42	5.290
10	10.044±0.0229	10.044±0.0365	100.44	0.44	4.823
12	11.87±0.0268	11.87± 0.0427	98.91	1.083	4.836

$$Confidence\,Limit = \overline{X} \pm t\left(\frac{S}{(n)^{1/2}}\right)_{27,28}$$

S =Standard deviation

n = The number of samples (number degrees of freedom)

X¯= Rate of measurements (concentration or absorbance)

t = Test- t at (n-1) from degrees of freedom at Confidence Limit 99%

RSD% =(S/ X¯) X100 [29]

S= Standard deviation

X¯= The arithmetic mean of the measurements.

The relative error percent Erel%

Erel% = [(Found- Taken) / Taken] X 100

Found = Analytical result of the concentration

Taken = The real result of the concentration

The recovery percent Rec%

Rec% = Erel% + 100 [30]

Or / Rec% = (Found / Taken) X 100

Surfactant Type Effect

The type of surfactant is very important in the cloud point extraction procedure because each surface has spectral characteristics that are determined by the practical basis of micelles. The method of cloud point was used by add a series of different volumes are prepared (0.2-2) mL in 10 mL volumetric flask solutions contains optimal volumes [1.3 mL of 3-nitro anilin, 1.0 mL of HCl, 1.3 mL of $NaNO_2$, and 1.0 mL of urea. 1.0 mL 4-ethyl Phenol 1.5 mL $NaHCO_3$, and 10% (v/v) Triton X-114] and complete the volume with distilled water, then are mixed, heated at 60 °C for 20 min to form cloud point, then centrifuged at 4000 rpm for 20 min, separating the surfactant-rich phase, and dissolved it in 3 mL ethanol and measure at λmax 426nm. The best volume of surfactant is 1.0 mL as shown in Fig. 12.

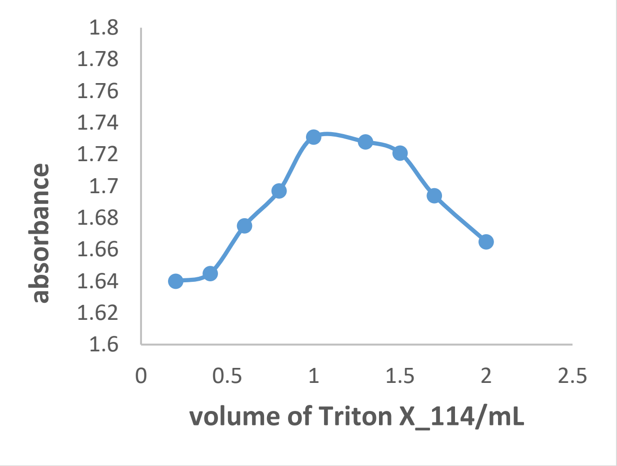


Figure 12. Volume of Triton X-114 for 4-ethyl phenol

The two circumstances to improve effective extraction and pre-concentration of phenol. To complete the cloud point extraction, equilibrium temperature and time incubation were deemed necessary steps. The temperature ranged from 30 to 65 °C, and the incubation time was between 5-35 minutes. In the following steps, temperature of 60 °C for equilibration and 20-minute time limit were selected and centrifuged for 20 minutes at 4000 rpm before cooling for 1 minute, resulting in a high recovery of phenol in a short period of time. The aqueous solution was decanted after the extraction procedure (CPE) was completed., and to reduce the viscosity of the surfactant-rich phase and facilitate its transfer into a spectrophotometric cell, EtOH was added. In the following experiment, 3 mL of ethanol was used.

Data Analytics

Using the cloud point extraction (CPE) technique's optimized parameters to evaluate phenol, a linear curve for calibration was constructed by plotting phenol concentration (2–11 µg mL^{-1}) versus absorbance, as illustrated in Fig. 13. Table 9. lists the analytical parameters with and without a cloud point. As shown in Table.9, the technique of extracting at the cloud point, which has high enrichment and pre-concentration factors, is a great novelty for extracting small amounts of phenol, Table.10 shows the precision and accuracy of the proposed method for the determination of standard of complex by cloud point.

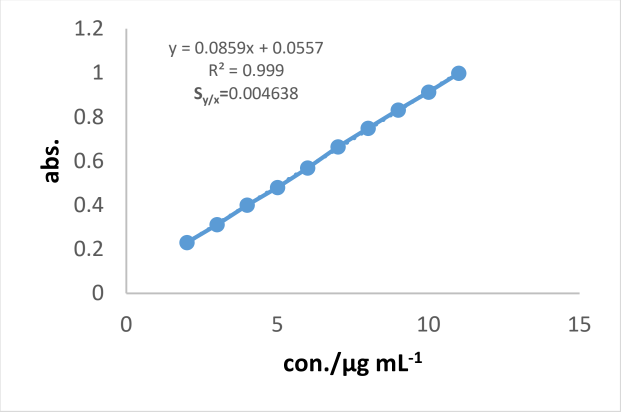


Figure13. Calibration curve of CPE of 4-ethylphenol

Table 9. Analytical parameter of cloud point extraction method

Type of parameter	Before CPE	After CPE
color	yellow	Orang
Wave length λ_{max} (nm)	426	426
Concentration rang (µg mL^{-1})	5-12	2-11
Regression equation(y)	y = 0.0492x - 0.0242	y = 0.0858x + 0.0565
Correlation coefficient(r)	0.9994	0.9997
Correlation coefficient (r^2)	0.9998	0.9988
Limit of Detection (µg mL^{-1})	0.051819	0.42103
Limit of Quantitation (µg mL^{-1})	0.1728	0.140345
Sandell's sensitivity (µg cm^{-2})	0.0203	0.0117
Slope (m)	0.0492	0.0858
Intercept (C)	0.0242	0.0565
Standard error for regression line ($S_{y/x}$)	0.0032	0.004638
Sa	0.0043	0.003629
Sb	0.00049	0.0005107
C.L for slope (b±tSb) at 95 %	0.0492 ± 0.001191	0.0858 ± 0.0012
C.L for intercept (a±tSa) at 95 %	0.0242 ± 0.0106	0.0565 ± 0.00243
C.L for 5 µg mL^{1} at 95%	4.96 ± 0.0287	4.939 ± 0.0076
C.L for 7 µg mL^{1} at 95%	6.9± 0.0284	7.0815 ± 0.0059
C.L for 10 µg mL^{-1} at 95%	10.044± 0.0365	9.969± 0.0021
C.L for 11 µg mL^{-1} at 95%		10.935± 0.0014
C.L for 12 µg mL^{-1} at 95%	11.87± 0.0427	
Molar absorptivity(L. mol-1. cm-1)	6.0024x 10^3	1.04676 x 10^4
Composition of product	1:1	
Preconcentration factor(Enrichment Factor)%	174.4%	174.4%

Table 10. Precision and accuracy of the proposed method for the determination of standard of complex (n=4) by cloud point

Amount Taken (µg mL^{-1})	Amount found (µg mL^{-1}) (±SD)	C.L. at 95%	Recovery% (mean±SD)	$E_{rel.}$%	RSD %
5	4.939 ±0.00476	4.939± 0.00758	98.79±0.004763	1.21	0.995
7	7.0815±0.0037	7.0815 ± 0.0059	101.17±0.0037	1.165	0.559
10	9.969±0.00129	9.969± 0.0021	99.786±0.00129	0.315	0.143
11	10.935±0.00083	10.935± 0.0014	99.408±0.00083	0.593	0.084

Application of Real Samples

The developed method is applied to determine 4-ethyl phenol in aqueous environment, a different concentration is taken (10,20 µg mL^{-1}) from 4-ethyl phenol and proposed method is applied and then the concentrations have been extracted from regression line equation of calibration curve. Table 11 shows the results before and after the cloud point extraction technique.

Table 11. Application of the proposed CPE for the evaluation of 4-ethyl phenol.

			Before CPE				
water source	Amount added (µg mL^{-1})	Amount found (µg mL^{-1}).	Recovery%	Average Recovery%	Erel%	Average Erel%	RSD%
Tap water	10	10.045	100.5	100.3	0.45	0.34	4.33
	20	20.045	100.2		0.23		2.04
Rustamiya	10	9.34	93.48	93.48	-6.5	-6.5	1.70
	20	19.74	96.8		-3.2		0.61
under Al doura bridge	10	10.043	100.43	100.43	0.44	0.324	0.441
	20	20.043	100.2		0.22		0.542
Diyala bridge	10	10.02	100.3	100.16	0.212	0.159	1.01
	20	20.02	100.1		0.106		0.31
			After CPE				
West water source	Amount added (µg mL^{-1})	Amount found (µg mL^{-1}).	Recovery%	Average Recovery%	Erel%	Average Erel%	RSD%
Tap water	10	10.18	101.8	101.4	1.7	1.3	1.34
	20	20.18	100.9		0.8		0.38
Rustamiya	10	9.65	96.5	97.4	-3.57	-2.68	2.79
	20	19.65	98.22		-1.78		0.213
under Al doura bridge	10	10.025	100.25	100.19	0.25	0.187	1.55
	20	20.025	100.13		0.13		0.52
Diyala bridge	10	9.97	99.74	99.81	-0.25	-0.187	0.825
	20	19.87	99.88		-0.125		0.689

Conclusion

A simple, fast, and spectrophotometric technique for estimating small amounts of 4-ethyl phenol with 3-nitro aniline has been developed. The first technique, which involved converting phenol to azo pigment, was spectrophotometrically measured. The second method used cloud point extraction to determine and pre-concentrate phenol. The proposed method was found to be extremely effective and recoverable, and it was applied to some sources of water from different regions. It was discovered by comparing it to other methods in the literature that it is the best method for simplifying the application, is regarded as ecological friendly due to the fact that it contains no organic components that are dangerous to the ecosystem, and that it has a wide linear range.

Acknowledgment

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.

- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.
- No animal studies are present in the manuscript.
- No human studies are present in the manuscript.
- No potentially identified images or data are present in the manuscript.

Authors' Contribution Statement

A.M.I conducted and carried out the experiments, data acquisition, analysis and interpretation, and participated in drafting the manuscript. S.A.D & A.J.M helped in interpretation, analysis, conceptualization and interpretation of manuscripts. All authors read and reviewed the final manuscript.

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References

1. Said KAM, Ismail AF, Karim ZA, Abdullah MS, Hafeez A. A review of technologies for the phenolic compounds recovery and phenol removal from wastewater. Process Saf Environ Prot. 2021; 151: 257-89. https://doi.org/10.1016/j.psep.2021.05.015.
2. Eryılmaz C, Genç A. Review of treatment technologies for the removal of phenol from wastewaters. J. Water Chem Technol. 2021; 43(2): 145-54. https://doi.org/10.3103/S1063455X21020065
3. Anku WW, Mamo MA, Govender PP. Phenolic compounds-natural sources, importance and applications. 2017; 419-43. https://dx.doi.org/10.5772/66927
4. Motamedi M, Yerushalmi L, Haghighat F, Chen Z. Recent developments in photocatalysis of industrial effluents: A review and example of phenolic compounds degradation. Chemosphere. 2022; 296: 133688. https://doi.org/10.1016/j.chemosphere.2022.133688
5. El-Naggar NA, Moawad MN, Ahmed EF. Toxic phenolic compounds in the Egyptian coastal waters of Alexandria: spatial distribution, source identification, and ecological risk assessment. water sci. 2022; 36(1): 32-40. https://doi.org/10.1080/23570008.2022.2031724.
6. Zamri MSFA, Sapawe N. Performance studies of electrobiosynthesis of titanium dioxide nanoparticles (TiO2) for phenol degradation. Mater Today Proc. 2018; 5(10): 21797-801. https://doi.org/10.1016/j.matpr.2018.07.034
7. Prebihalo S, Brockman A, Cochran J, Dorman FL. Determination of emerging contaminants in wastewater utilizing comprehensive two-dimensional gas-chromatography coupled with time-of-flight mass spectrometry. J Chromatogr A. 2015; 1419: 109-15. https://doi.org/10.1016/j.chroma.2015.09.080
8. Kamel RM, Shahat A, Anwar ZM, El-Kady HA, Kilany EM. Efficient dual sensor of alternate nanomaterials for sensitive and rapid monitoring of ultra-trace phenols in sea water. J Mol Liq. 2020; 297: 111798. https://doi.org/10.1016/j.molliq.2019.111798
9. Tsuruta Y, Kitai S, Watanabe S, Inoue H. 2-Methoxy-4-(2-phthalimidinyl) phenylsulfonyl chloride as a fluorescent labeling reagent for determination of phenols in high-performance liquid chromatography and application for determination of urinary phenol and p-cresol. Anal Biochem. 2000; 280(1): 36-41. https://doi.org/10.1006/abio.2000.4492
10. Lehtonen M. Gas-liquid chromatographic determination of volatile phenols in matured distilled alcoholic beverages. J AOAC Int .1983; 66(1): 62-70. https://doi.org/10.1093/jaoac/66.1.62
11. Beitollahi H, Tajik S, Biparva P. Electrochemical determination of sulfite and phenol using a carbon paste electrode modified with ionic liquids and graphene nanosheets: application to determination of sulfite and phenol in real samples Measur.2014; 56: 170-7. https://doi.org/10.1016/j.measurement.2014.06.011
12. Kadhim EA, Dhahir SA, Sando MS. New Diaz Coupling Reaction, Cloud Point Extraction Spectrophotometric Determination of Sulphadimidine Soudium in Pure form and Pharmacetical Preparation with Salicylic Acid as the Coupling Reaction. Indian J Forensic Med Toxicol. 2020; 14(2): 868-74. https://doi.org/10.37506/ijfmt.v14i2.2989
13. Hassan SS, Shaheed IM, Mohammed NJ, Dhahir SA. A New Visible Spectrophotometric Approach for Mutual Determination of Allopurinol drug in Pharmaceuticals after Cloud Point Extraction. IOP Conf. Ser : Earth Environ Sci 2021; 722(1): 012033. https://doi.org/10.1088/1755-1315/722/1/012033

14. Abed SS. Spectrophotometric and reverse flow injection method determination of nitrazepam in pharmaceuticals using O-coumaric acid as a new chromogenic reagent. Baghdad Sci J. 2020; 17(1 (Suppl.)): 0265 https://doi.org/10.21123/bsj.2020.17.1(Suppl.).0265
15. ALmashhadani IMJ, Abed SS. Kinetic-spectrophotometric Method for the Determination of Naringenin in Pure and Supplements Formulations. Baghdad Sci J. 2019; 16(3): 595-602. https://doi.org/10.21123/bsj.2019.16.3.0595 .
16. jassam Alaallah N, Dhahir SA, Ali HH. Determination of Sulfacetamide Sodium in Pure and Their Pharmaceutical Formulations by Using Cloud Point Extraction Method. Baghdad Sci J. 2021; 18(3): 0575-582. https://doi.org/10.21123/bsj.2021.18.3.0575
17. Mo F, Qiu D, Zhang L, Wang J. Recent development of aryl diazonium chemistry for the derivatization of aromatic compounds. Chem Rev. 2021; 121(10): 5741-829. https://doi.org/10.1021/acs.chemrev.0c01030
18. López-Mayan J, Barciela-Alonso MC, Domínguez-González MR, Pena-Vazquez E, Bermejo-Barrera P. Cloud point extraction and ICP-MS for titanium speciation in water samples. Microchem J. 2020; 152: 104264. https://doi.org/10.1016/j.microc.2019.104264
19. Al-Yousefi DA, Ali IR. Spectrophotometric determination of transition elements by cloud point extraction with use laboratory by thiazol azo reagent and applied in environmental samples. AIP Conf Proc. 2022; 2386(1) https://doi.org/10.1063/5.0067206
20. Dhahir SA. Determination of mercury and manganese by using new reagent azo after cloud point extraction for some environmental sample in Iraq. Am J Environ Sci. 2015; 11(5): 392-401. https://doi.org/10.3844/ajessp.2015.392.401
21. Ali MS, Ali IR . Preparation and characterization of new reagent derivative of thiazol azo for spectral evaluation of some metal elements in different samples using cloud point technique. AIP Conf Proc.2022; 2386(1) https://doi.org/10.1063/5.0067208
22. Azooz EA, Shabaa GJ, Al-Muhanna EHB, Al-Mulla EAJ, Mortada WI. Displacement cloud point extraction procedure for preconcentration of iron (III) in water and fruit samples prior to spectrophotometric determination. Bull Chem Soc Ethiop. 2023; 37(1): 1-10. https://dx.doi.org/10.4314/bcse.v37i1.1
23. Dhahir SA, Bakir SR. Cloud point extraction spectrophotometric determination of copper, chromium and cobalt by salen as reagent in wastewater of Iraq. Asian J Chem. 2014; 26(16): 5305. https://doi.org/10.14233/ajchem.2014.17754
24. Mortada WI. Recent developments and applications of cloud point extraction: A critical review. Microchem J. 2020; 157: 105055. https://doi.org/10.1016/j.microc.2020.105055
25. Arya S, Kaimal AM, Chib M, Sonawane SK, Show PL. Novel, energy efficient and green cloud point extraction: technology and applications in food processing. J Food Sci Technol. 2019; 56: 524-34. https://doi.org/10.1007/s13197-018-3546-7
26. Kojro G, Wroczyński P. Cloud point extraction in the determination of drugs in biological matrices. J Chromatogr Sci. 2020; 58(2): 151-62. https://doi.org/10.1093/chromsci/bmz064
27. Schotten C, Leprevost SK, Yong LM, Hughes CE, Harris KD, Browne DL. Comparison of the thermal stabilities of diazonium salts and their corresponding triazenes. Org Process Res Dev. 2020; 24(10): 2336-41. https://doi.org/10.1021/acs.oprd.0c00162
28. Yamamoto Y, Kumamaru T, Hayashi Y. New method for the determination of pentachlorophenol by atomic absorption spectrophotometry. Talanta. 1967; 14(5): 611-2. https://doi.org/10.1016/0039-9140(67)80249-2
29. Abd Wannas F, Azooz EA, Ridha RK, Jawad SK. Separation and micro determination of zinc (II) and cadmium (II) in food samples using cloud point extraction method. Iraqi J Sci. 2023; 64(3): 1049-1061. https://doi.org/10.24996/ijs.2023.64.3.2
30. Kirkbright G, Smith A, West T. An indirect sequential determination of phosphorus and silicon by atomic-absorption spectrophotometry. Analyst. 1967; 92(1096): 411-6. https://doi.org/10.1039/AN9679200411

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التقدير الطيفي لمركب 4-إيثيل فينول باستخدام استخلاص نقطة الغيمة في عينات مياه مختلفة

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الخلاصة

تصف هذه الدراسة تطوير تقنية سهلة ورخيصة ودقيقة وسريعة لقياس 4-اثيل فينول وتنطوي الطريقة الأولية على تحويل -3 نيترو انيلين إلى ملح ديازونيوم ثم التفاعل مع 4 - إثيل فينول في وسط قلوي.المعقد المتكون هو أصفر اللون وله امتصاص عند اعلى طول موجي عند 426 nm. ويتبع قانون بير في مدى خطي قدره 5-12 µg mL^{-1} مع معامل ارتباط قدره 0.994 وامتصاص مولاري ^{3}^10x6.0024 L.mol^{-1}.cm^{-1} وتم استُخدِام تقنية نقطة السحابة لقياس كميات قليلة جدا من الفينول باستخدام TritonX-114 كمفاعلات خافضة للتوتر السطحي، ومن ثم تم القياس باستخدام مقياس طيفي للأشعة فوق البنفسجية. وكانت قيمة معامل الارتباط 0.998، وكان الامتصاص المولاري هو 1.0476 × 10^{4} L.mol-1.cm-1، وكان المدى الخطي 2-11 µg mL^{-1} . وحُددت حدود الكشف والتحديد الكمي بأنها 0.42103 و0.140345 µg mL^{-1}، على التوالي. وقد استخدمت الطريقة المقترحة بنجاح لتحديد الفينول في العينات البيئية المختلفة.

الكلمات المفتاحية: الاستخلاص بنقطة الغيمة ، تقدير ، عينات المياه البيئية ،4-إيثيل فينول، قياس الطيف الضوئي.