Chemical Methodologies 6 (2022) 569-570

Chemical Methodologies

Journal homepage: http://chemmethod.com

Original Research Article

Micro Spectrophotometric Determination and Cloud Point Extraction of Aspirin with Iron (III) in Pure Form and Pharmaceutical Drugs

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ARTICLE INFO

Article history

Submitted: 2022-03-03
Revised: 2022-04-12
Accepted: 2022-05-17
Manuscript ID: CHEMM-2203-1449
Checked for Plagiarism: **Yes**
Language Editor:
Dr. Behrouz Jamalvandi
Editor who approved publication:
Dr. Zeinab Arzehgar

DOI:10.22034/CHEMM.2022.332256.1449

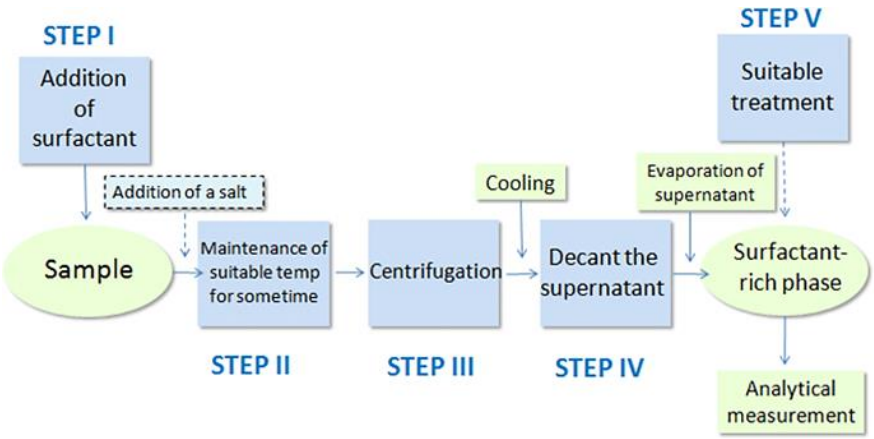
KEYWORDS

Aspirin
Spectrophotometry
Ferric Ion
Cloud pint extraction

ABSTRACT

Cloud-point extraction and molecular spectrophotometry in tandem was developed and suggested as a method for detecting aspirin and iron (III) ions in pharmaceutical. In a dilute acidic medium, aspirin interacted with Fe(III) ions to produce a brightly colored [Fe(III)-aspirin] complex, which was originally as a mediated extractant, extracted into Triton X-114 micelles, then determined by spectrophotometry at a wavelength maximum of 527 nm. For target analytes, all experimental variables were previously adjusted to accomplish this purpose. The results showed that pre-concentration factors of 80 for aspirin resulted in a detection limit of 0.23 g mL^{-1} having a linear range of (5-120) gmL^{-1} (r= 0.9998). For aspirin, a mean recovery percentage of 98. ± 1.09% was recorded, with precision (RSD %) ranging from 0.04-0.66 %.

GRAPHICAL ABSTRACT



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Introduction

Aspirin (acetylsalicylic acid) is one of the most affordable and commonly available drugs used for the alleviation of headaches, heat, and muscular pains all over the world (Scheme 1).

Salicylate drugs can reduce the incidence of a variety of malignancies, including lung and colon cancers, due to their antioxidant properties and ability to neutralize radicals [1, 2].

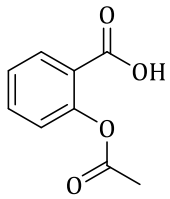
In light of this, and given the paucity of published work in this area, this study aimed to contribute to the development of new scope in analytical chemistry by developing analytical methods based on present chemical reaction systems. Aspirin analysis is commonly performed using direct titration and back-titration procedures [3]. Furthermore, an electrochemical sensor for determining acetylsalicylic acid has recently been developed; also, to improve sensitivity and precision, a composite of multi-wall carbon nanotubes (MWNT) and carbon black (CB) was created, with ferrocene (Fc) serving as a built-in correction molecule [4]. Additionally, Wang *et al.,* produced a Nano-sensor based on the structure-switching technique. The aptamer had a strong affinity for the salicylic acid SA [5].

The complexometric reaction system chosen in this study is the reaction of aspirin with Fe(III) ion in acidic medium to form a soluble Fe(III)-ASP complex. The main reason for selection of this reaction is that the drug ASP and Fe(III) have great importance to humans.

Several processes are not straightforward for routine analysis and involve expensive or specialized instruments, along with a heating step, poor selectivity, and low sensitivity. Visible spectrophotometry is perhaps the most extensively described method for determining aspirin in medicines [6]. To tackle this challenge, there has recently been a significant increase in interest in cloud point extraction-spectrophotometric approach to detect numerous organic compounds of medicinal value [7].

The mix is perfect of molecular spectrophotometry and cloud point extraction (CPE) [8]. It has been reported to have appealing qualities that leads to analytical approaches [8]. The routine analyses of organics and metal ions in a variety of matrices have been simplified and made more convenient [8].

Instead of relying on complex and costly instruments [9]. UV-Vis absorbance spectroscopy was used to investigate the purple SA-Fe (III) combination for providing light on the mechanism of Salicylic acid SA's inhibitory effects on MIL-53(Fe) activity [10, 11]. This research was based on the possibility of the reaction of drug (aspirin) with iron (III) ions to form a coloured complex that can be extracted by one of extraction methods; thereby the drug and iron ions are determined by the matching procedure.



Scheme 1: Structure of aspirin

Materials and Methods

A Shimadzu double-beam UV-Vis Spectrophotometer model UV-1800 (Kyoto, Japan) with a 10 mm optical path cell was used to analyse the absorbance spectra and absorbance of both analytes. The residual iron in aqueous solution was examined using a double-beam Shimadzu AA-6300 Atomic Absorption Spectrophotometer with a titanium burner unit (Shimadzu, Japan) Premixed for an air/acetylene flame that had been air-cooled (10 cm slot). The pH of the solution was monitored using a microprocessor-based portable pH meter (HANNA, Germany). For CPE studies, the experts from England provided a thermostatic water bath (WNB7-45). Sartorius (0.0000) electric balance made in Germany was used and Triup International Corp. TRIU 800 centrifuges made in Korea was applied.

Preparation of Standard Solutions

The reagents and materials used in this study were all of the highest purity possible. To dissolve and make solutions, distilled water was employed to prepare and dissolve solutions. Aspirin 1000 g. ml^{-1} stock solution was made by 0.1 g dissolved in

a very small amount of distilled water, then diluted to 100 mL with distilled water. Daily working solutions were made from this solution using appropriate water dilutions, maintained in the refrigerator. Acros Organics, based in New Jersey, provided Triton X-114 (purity >99.9%). Diluting 10 mL of Triton X-114 in 100 mL water gave a 10% (v/v) solution in a volumetric flask with a capacity of 100 mL; in (0.050 M) sulfuric acid, 0.8634 gram of pre-dried ammonium ferric sulfate (BDH) was dissolved (BDH) to make a stock solution of Fe^{3+} (1000 $mg.L^{-1}$). To ensure complete dissolution, this solution is used after at least 24 hours. In a 1 L calibrated flask, 5.43 mL of 98 percent H_2SO_4 (1.84 g/mL, BDH) was diluted with distilled water to make a 1 M sulphuric acid solution with 0.1 M from sulphuric acid Ethanol bought from Abo Teeba Co. Iraq. Throughout this project, we used deionized and double-distilled water.

General CPE procedure for preparation of Fe(III)-aspirin complex

Standard amount of aspirin solution has been mixed with 1.0 mL ferric ion solution (100 gmL^{-1}) and 0.25 mL H_2SO_4 (1×10^{-4}M) in a 10 mL volumetric flask. Then one percent Triton X-114 (10% v/v) has been added, stirred, and diluted with water. The contents of the flask were transferred to a 10 mL centrifuging test tube and heated at 60 °C for 25 minutes in the thermostatic bath. Centrifugation at 6000 rpm for 15 minutes separated the phases. Next, to improve viscosity, the surfactant-rich phase was cooled in an ice bath. Inverting the tube made pouring the aqueous phase quite simple. The complex's absorbance at 527 nm was measured in comparison to a reagent blank made in comparable circumstances after the surfactant-rich phase containing the complex was dissolved with an appropriate of ethanol. Traditional spectrophotometry was used to evaluate the extraction efficiency (E) and the distribution ratio (D) of aspirin in aqueous solution, with a maximum wavelength of 293 nm (%E).

Pharmaceutical sample preparation for aspirin detection

The suggested approach was to use Fe(III) and mix it with aspirin-containing pharmaceutical formulations. This approach has been utilized to determine aspirin in a tablet of 100 mg aspirin (Taj Pharmaceuticals Ltd., India). After dilution with water, the tablets were filtered and tested to the CPE approach in general, where the aspirin content was evaluated spectrophotometrically at a maximum of 527.

Results and Discussion

Absorption spectra

The production of in an acidic medium, Fe(III) ions and aspirin interact to form the complex in the presence of the surfactant was confirmed using spectrophotometers. 3×10^{-4} M aspirin solution, 1×10^{-4} M Fe(III) solution, and Fe(III)-aspirin complex (10 mL aqueous solution containing 2.5 $\times10^{-4}$ M aspirin) absorption spectra, 1.5×10^{-4} M H_2SO_4, 1×10^{-4} M Fe(III). A spectrophotometric was used to measure 1 mL of 10% ethanol TX-114 versus blank solutions between 190 and 1000 nm. At 527 nm, there was a prominent absorption band with a shoulder. The development of a compound between Fe(III) ions and aspirin was indicated. The absorption maxima for pure aspirin solution were found to be at 293 nm. At 300 nm, one distinct band can be seen in the Fe(III) ion solution. Figure 1 depicts all of these spectra. As a result, the Fe(III)-aspirin complex's wavelength maximum of 527 nm was used throughout this work.

Optimization of CPE procedure

A set of studies were carried out with the objective of determining the impact of major elements on using a typical optimization, *i.e.* the extraction efficiency of cloud points. A classic optimization method is to look at the effect of one component at a time on the instrumental response (OFAT) while leaving the other parameters fixed. As a result, the measurement's sensitivity can be improved. Triton X-114 amount, Fe(III) concentration, equilibration temperature, H_2SO_4 concentration, and all of the incubation periods were investigated in this regard.

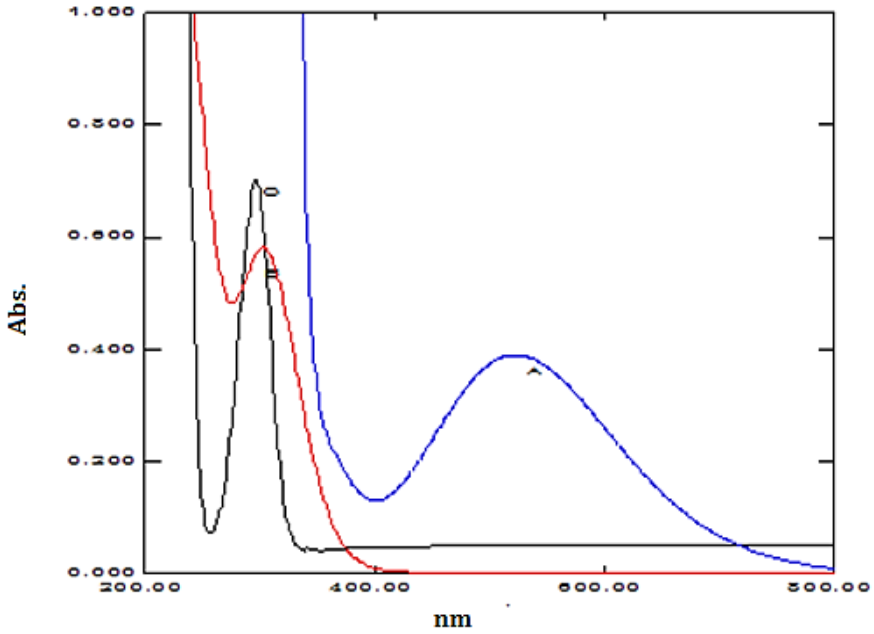


Figure 1: (a) Fe(III)-aspirin complex (b) Fe(III) solution and (c) reagent aspirin

Influence of H_2SO_4 amount

The concentration of H_2SO_4 was found to play a significant role in the complication reaction between aspirin drug and Fe(III), then it is in charge of the complication's stoichiometry [12]. Furthermore, the acidic medium has a significant impact on the synthesis and stability of chelate extracted. The influence of H_2SO_4 concentration upon the formation of the (Fe-aspirin) complex was analysed in Triton X-114 media by measuring 527 nm absorbance signals across the limited values of H_2SO_4 concentrations (1×10^{-2} -0.5×10^{-4} M). 10 gmL^{-1} Fe(III), 80 gmL^{-1} aspirin, and 1% (10%by volume) Triton X-114 were used in the experiment. The results are shown in Figure 2. It was noted that as the concentration of H_2SO_4 was increased, the absorbance increased and reached at 1.5×10^{-4} M H_2SO_4. After that, it decreased rapidly at a high-level concentration, which could result in the case of complicated dissociation and inadequate micelle extraction as a result of a shift in the formation response to the left. Consequently, a concentration of 1.5×10^{-4} M H_2SO_4 was chosen as the best for the entire production of the Fe(III)-aspirin complex, corresponding to an ionic strength of 0.75×10^{-3}.

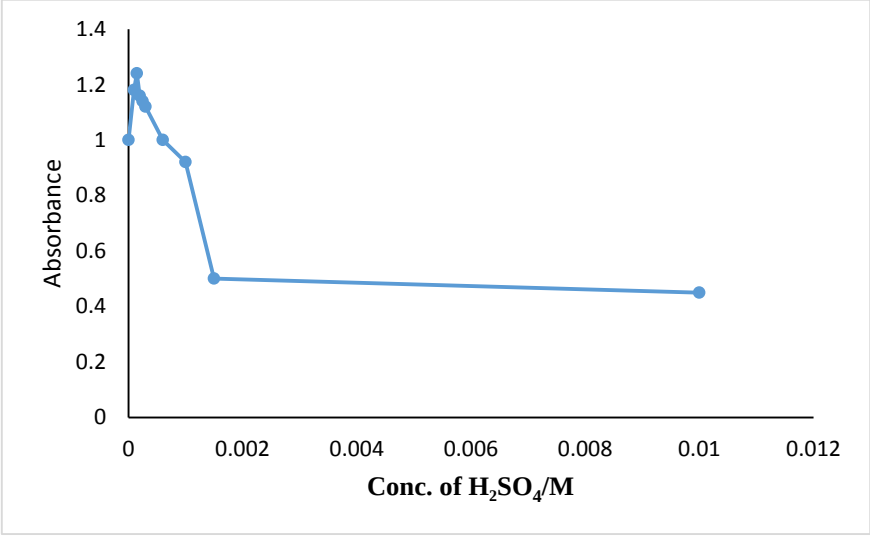


Figure 2: Influence of H_2SO_4 concentration on the Fe (III)-aspirin complex formation

Influence concentration of Fe(III) ions

The influence of the Fe(III) ion concentration was investigated by taking measurements of the absorbance signals using the CPE procedure for a solution containing 80 g mL^{-1} aspirin, 1.5×10^{-4} M H_2SO_4 for the aspirin complex, 1 percent (v/v) ten percent Triton X-114, and a Fe(III) concentration ranging from 0.1-15 g mL^{-1}. The results are shown in Figure 3.

The Fe(III) ion concentration that delivered the highest absorbance for high-efficiency complex formation and extraction in the cloud point layer (CPL) was 10 g mL^{-1}.

The responses were decreased as the amount of ferric ion solution increased, and the extraction efficiency was reduced as a result of a shift in the equilibrium in the direction of reversion according to the principle of mass action. There is no potential for complex formation completion at lower metal ion concentrations than optimal, resulting in less drug complex extracted into the CPL. Following that, it was observed that 10 g mL^{-1} was sufficient because of the complicated formation of Fe(III)-aspirin, hence throughout this study, it was used as the best option.

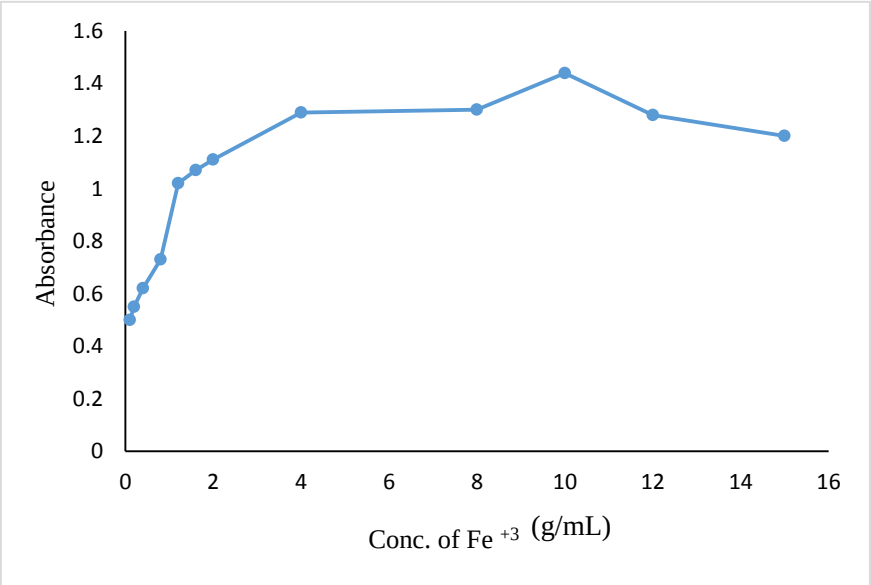


Figure 3: Influence concentration of iron (III) in the (Fe-Asp) complex by CPE

Influence amount of Triton X-114

The surfactant content has a significant impact on the efficiency of extraction when using the (CPE) method as a result of its significance capacity to increase extraction efficiency. This is due to reducing Vs/Va in the phase volume ratio and enhancing its ability to concentrate. To demonstrate the role played by the concentration of triton X-114 in the absorbance of the recovered Fe(III)-aspirin complex, the issue was investigated in 10 percent (v/v) triton X-100 surfactant in a volume range of (0.1-2.5 mL) while all additional variables were held constant. As shown in Figure 4, it was observed that increasing the amount of Triton X-114 to 1.0 mL of 10% (v/v) enhanced the complex's absorbance, which then sharply dropped at higher concentrations. As a result, a volume of 1.0 mL with a 10% concentration TritonX-114 is the maximum amount that can be achieved for the extraction process, a state of equilibrium, resulting in the production of a point cloud layer with a higher viscosity and lower volume. Low sensitivity or extraction efficiency is caused by the gathering's inability to quantitatively. When the amount was increased, the detection signal decreased, resulting in ineffective e for modest levels of triton X-114, entrap the hydrophobic compound traction. As a result, the optimum volume of Triton X-114 was 1.0 mL at a concentration of 10% (v/v).

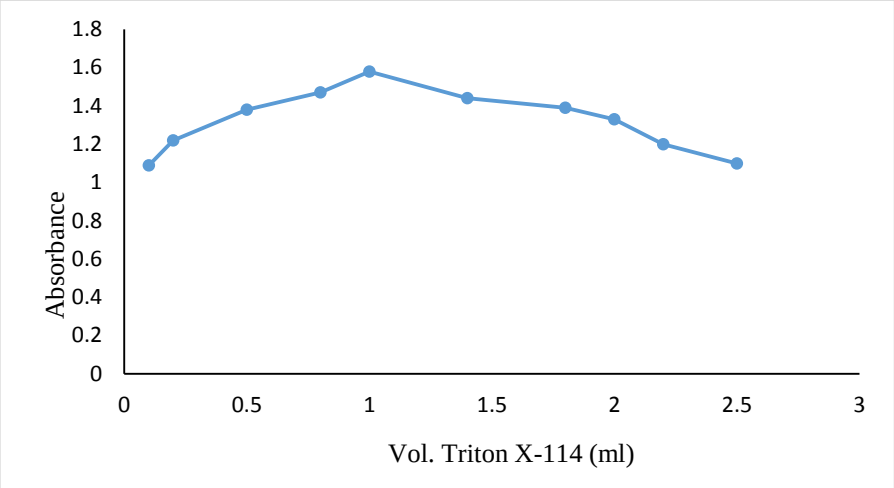


Figure 4: Influence of Triton X-114 amount on the analytical of Fe^{+3}-aspirin

Influence of time and temperature

To investigate temperature's Influence on Extraction of the Fe(III)-aspirin complex, a collection of tests was conducted using a 10 mL aqueous solution with all of the ingredients under previously optimal conditions; a temperature range of 25 to 85 °C was used at 20 minute incubation period. The highest absorbance for the Fe(III)-aspirin complex was found to be at 55 °C (Figure 5). The target complex's CPE efficiency was then reduced as the temperature was increased. As a result, to obtain the most out of the Fe(III)-aspirin complex, the temperature of 55 °C was chosen as the best temperature of equilibration.

In addition, the CPE methodology requires enough time to achieve two phases in equilibrium (surfactant-rich and bulk aqueous phases) by collecting surfactant micelles. As a result, to extract all reagents from 10 mL solutions, a number of experiments were carried out under optimum circumstances, containing all reagents, but with incubation times ranging from 5 to 40 minutes at 55 °C. As demonstrated in Figure 6, a 20-minute incubation time was sufficient to achieve the highest absorbance of the extraction of Fe^{+3}-aspirin complex. The effects of centrifugation rate and time were also considered. It was observed that a 15-minute centrifugal time at 6000 rpm was enough to separate two phases

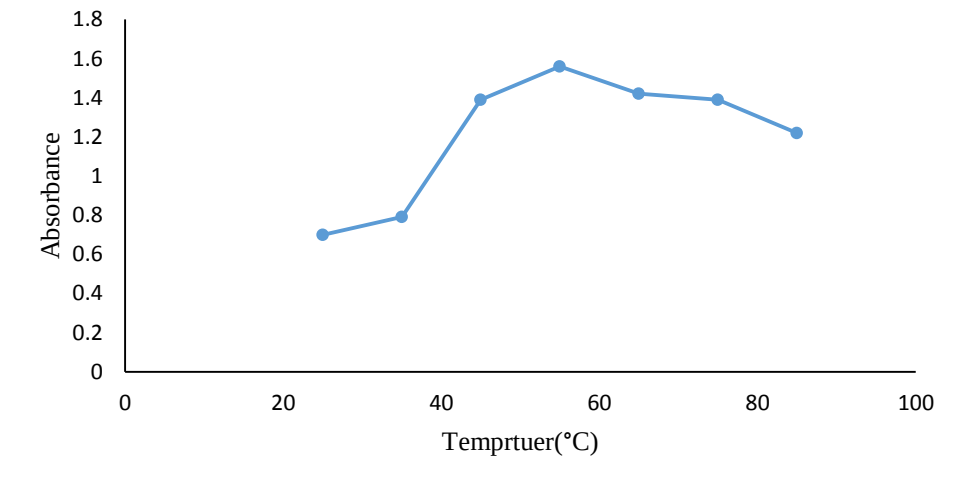


Figure 5: Influence of equilibration temperature on the CPE of Fe(III)-aspirin complex

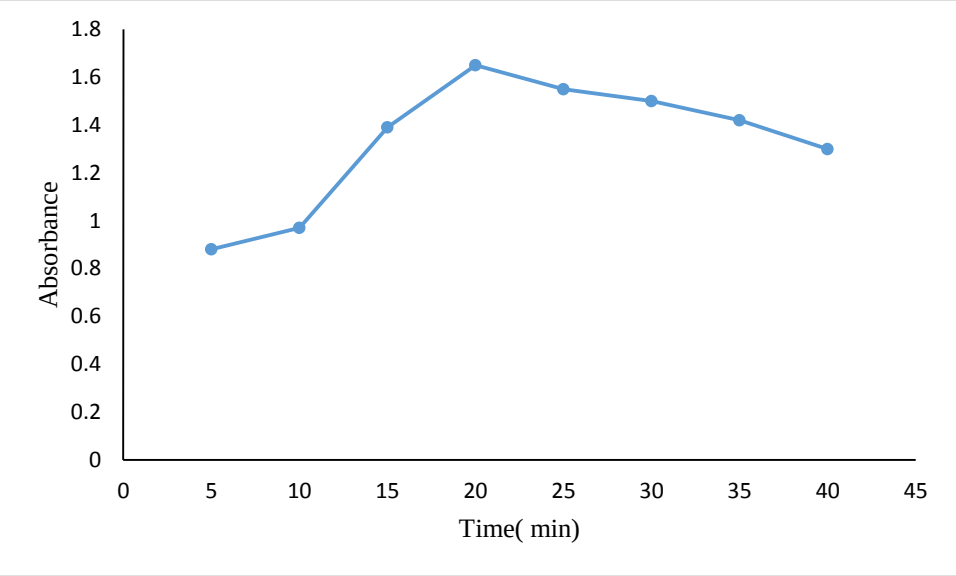


Figure 6: Influence of incubation time on the CPE of the Fe(III)-aspirin complex

Stoichiometry of the Fe $^{+3}$-aspirin complex

The mole-ratio approach is used to determine the composition of azo dye in an acidic medium in a solution of aspirin and Fe(III) ion, where the amount of aspirin remains constant but the amount of Fe(III) ion varies. Absorption is measured at 527 nm, which is the maximum absorbance. In the presence of H_2SO_4, the mole-ratio plot of azo dye between aspirin and Fe(III) ion is shown in Figure 7. The results indicated that a drug-to-reagent ratio of 1:2 was produced. The Job plot (continuous variation approach) provided a result that is similar to the molar ratio method, suggesting that there is the mole fraction close to 0.30, suggesting that the complex's iron(III): drug ratio is (1:2) (Figure 7). As a result, the proposed reaction path for the synthesis of azo dye may be predicted based on the mole ratio and Job plot data, as depicted in Figure 8.

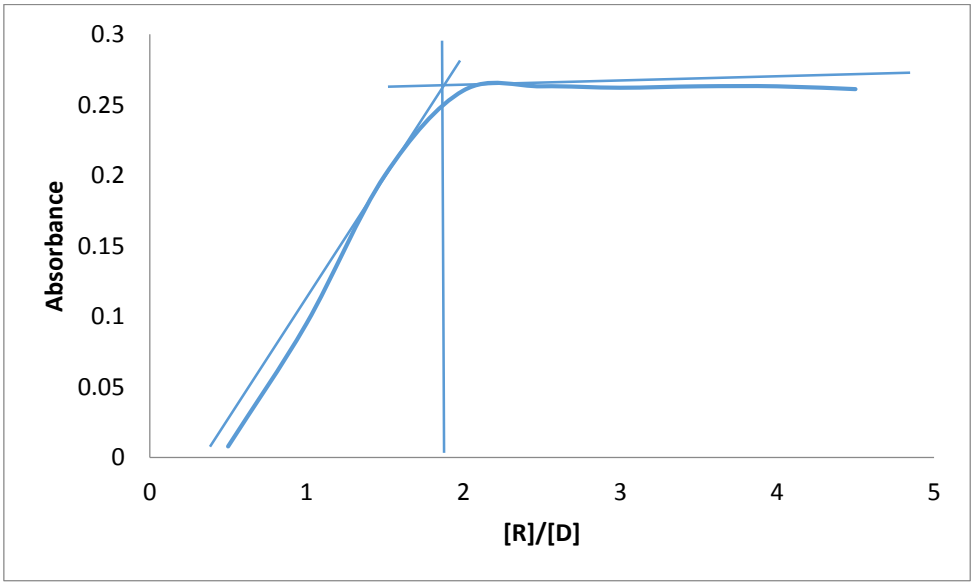


Figure 7: Fe (III)-aspirin complex mole ratio method

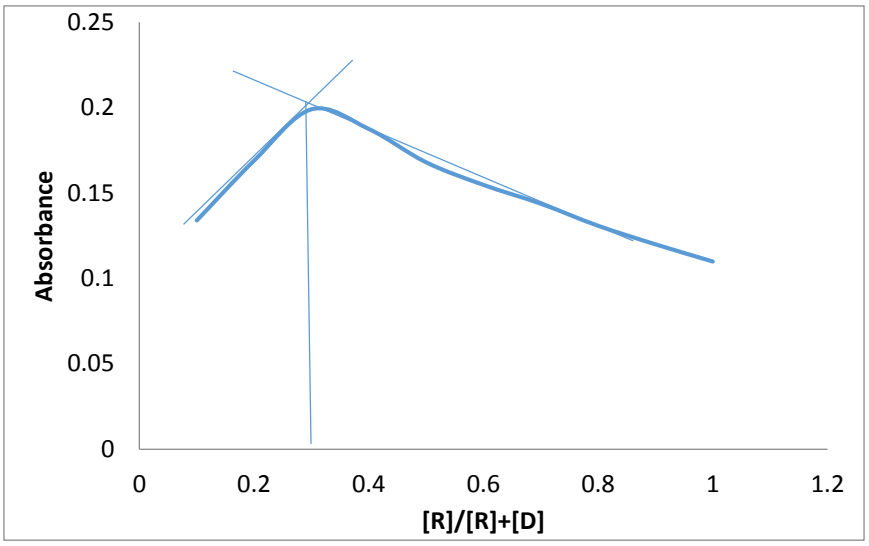
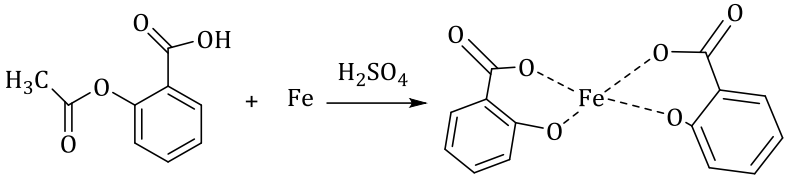


Figure 8: For the Fe(III)–aspirin combination, a continuous variation technique was used

Our results were consistent with those of Idrees *et al* [13], Sultan and Suliman [14, 15], but they disagree with those of other workers [16] where a (1:1) complex was obtained, when iron(III) and aspirin were complexed at acidities greater than 0.025 M. Using the given results, the stability constant (K_f) of the Fe(III)-aspirin complex may be measured using the approach used elsewhere 29 and found to be 2.12×10^{10} at 527 nm from a thermodynamic standpoint [12]. So, the reaction between iron(III) and aspirin in acidic media was shown in the **Scheme 2.**



Scheme 2: Calibration curve for aspirin and iron(III) ions

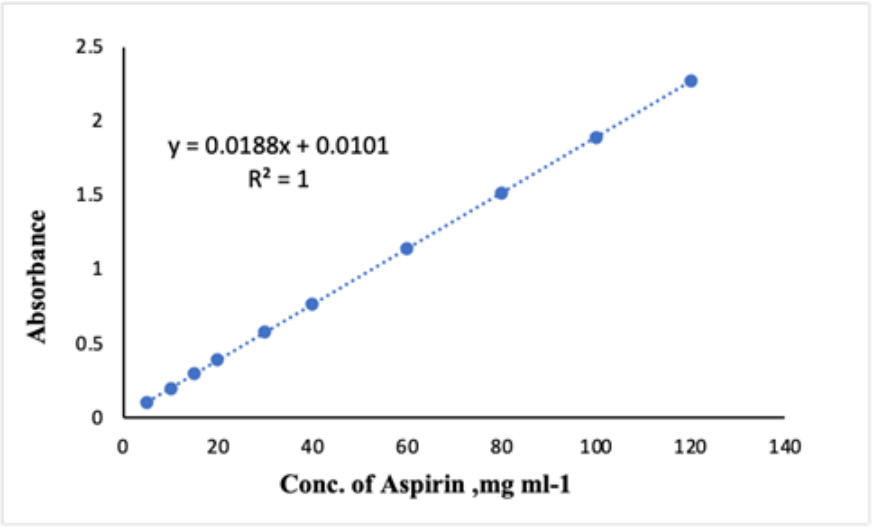


Figure 9: Calibration curve for aspirin by combined CPE-spectrophotometry

The calibration curves for aspirin using Fe(III) were constructed under optimal conditions. The statistical evaluation of the curve is described in Table 1. It shows target analytes and it includes the calibration curve of the investigated aspirin using Fe(III) after CPE. The calibration curve was

linear (r=0.9998, 10 points) over the concentration range of (5-120) μgmL^{-1} (Figure 9). With a 60-fold preconcentration factor, a limit of detection (LOD) by 0.23 μgmL^{-1}was attained (Table 1). Concerning LOD, our results were superior to those obtained by Singh [17], Wagh *et al*. [18], Shinde *et al*. [19], Abou-Taleb *et al*. [20] and Pant *et al*. [21]. However, our results are in agreement with those of Patel *et al*. [22] and Hanwen *et al*. [23]. It was, however, worse than the results reported by Sher *et al*. [24] and El-Brashy. A M, [25]. HPLC was used, accompanied by UV and fluorescence detectors. Tables 1, the proposed approach has a high level of sensitivity and extractability.

Accuracy and precision

To determine the precision of the suggested method for detecting aspirin in terms of freedom from systematic mistakes, three blank samples had been spiked with 10, 20, and 30 μg ml^{-1}of aspirin taken from Aspirin (300 μg ml^{-1}) solution, as well as additives such as water. The three spiked samples were subjected to the standard CPE aspirin methodology. Because there are no systemic errors, the results demonstrated in Table 2 provide a high level of precision in terms of percent recovery that may be reached inside the limits of 98.96-1.08 %. In the meantime, each spiking sample was tested five times for RSD percent precision and the results were found to be in the range of 0.04 to 0.66 %, indicating that the suggested procedure is accurate and precise.

Determination of aspirin in the pharmaceuticals

The suggested method was used to identify the presence of aspirin in an aspirin-containing medicinal product, such as 100 mg aspirin tablets. Table 3 displays the results. The result was statistically compared with the quoted value reported by the manufacturer to ensure that the suggested process is applicable. Table 3 shows that the calculated t-value at 95 percent level of assurance and (n-1) degree of freedom was smaller than the critical (t=4.304), showing acceptance of the manufacturer's claim (Ho=0.3 percent). At the 95 percent confidence level, systematic or random errors are not supported by evidence.

Table 1: The statistical data and analytical figures of merits for determining aspirin using Fe(III)-CPE spectrophotometry

Parameter	Value
λmax nm	527
CPE method and regression equation	y = 0.0188x+ 0.0101
Correlation coefficient(r)	0.9998
Coefficient of determination (R^2)	99.98%
C.L. for the slope (b± tsb*)* at 95%	0.0188±0.000243
C.L. for the intercept (a± tsa) at 95%	0.0101±0.016512
Concentration range, μgmL-1	5-120
Limit of Detection, μgmL-1	0.23
Limit of Quantitation, μgmL-1	0.67
Sandell's sensitivity (mg cm-2/0.001A.U)	0.053
Molar absorptivity, L.mol-1.cm-1	1.3×10^4
Composition of complex (Fe-Aspirin)*	1:2
RSD% (n=4) at 80 μgmL^{-1}	0.46
Preconcentration factor	60
Enrichment factor	280.6
Distribution ratio(D)	28.56
Extraction efficiency(%E)	96.63

** Methods for calculating mole ratios and jobs*

Table 2: The proposed method for determining aspirin using Fe(lll) has high accuracy and precision

Amount of Aspirin taken, µgmL-1	Amount of Aspirin found, µg mL-1	Recovery %	Erel %	RSD % (n=5)
10	9.87	99.21	-0.800	0.665
20	19.83	99.23	-0.820	0.332
30	29.65	98.39	-1.57	0.042

Table 3: The proposed method for determining aspirin in pharmaceutical formulations and the statistical comparisons with stated values

Commercial name and content	Practical content (proposed* method) ($x^* \pm ts/\sqrt{n}$) at 95% C.I.	$t=(x^*-\mu)\sqrt{n}/s$ Proposed method* *versus* claimed value at 95% C.I.	Erel %	RSD %
300 µg mL^{-1}[1] Aspirin	0.31 0.28 0.30 Ave: 0.303±0.029%	tcal=0.449 0.449<4.303	1.00	1.3

**Mean of three determinations*

Conclusion

The level of aspirin in pharmaceutical items was determined using a new CPE approach developed for this investigation. The approach is based on the interaction of aspirin with Fe(III) ions in a dilute acidic media utilizing Triton X-114 as a non-ionic surfactant to form a brightly coloured [Fe(III)-aspirin] complex, which was measured using spectrophotometry at a wavelength maximum of 527 nm. The proposed approach provides an efficient and cost-effective method for determining aspirin in pharmaceutical products, as well as avoiding toxic solvent extraction by utilizing a little volume of solvent (500 µL per sample), making it environmentally friendly. The recovery results and all statistical parameters clearly show that this approach is accurate and reproducible, suggesting that it can be used as a good spectrophotometric method.

Acknowledgments

I would like to thank the department of chemistry/ college for science for women. I would like to thank my husband for his contribution in the production of this work.

Funding

This research did not receive any specific grant from fundig agencies in the public, commercial, or not-for-profit sectors.

Authors' contributions

All authors contributed toward data analysis, drafting and revising the paper and agreed to responsible for all the aspects of this work.

Conflict of Interest

We have no conflicts of interest to disclose**.**

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HOW TO CITE THIS ARTICLE

Noora Saad Mubder, Maha Al-Tameemi, Hawraa Mahmood, Nahlah Kadhim Salman, Haider Al-Neshmi. Micro Spectrophotometric Determination and Cloud Point Extraction of Aspirin with Iron (III) in Pure Form and Pharmaceutical Drugs. *Chem. Methodol.*, 2022, 6(7) 569-570

https://doi.org/10.22034/CHEMM.2022.332256.1449

URL: http://www.chemmethod.com/article_150156.html