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Experimental Studies on Spectrophotometric Extraction for Estimation of Fe (III) Using an Analytical Reagent

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Abstract In the present experimental studies, a new reagent 2, 4-dimethyl -3H- 1, 5 benzodiazepine (DBA) is devised for the extractive estimation of Fe (III) from given sample and by use of spectrophotometer as analysis tool. In the laboratory work, the fresh reagent was prepared and its characterization has been carried out with the help of mass spectrophotometer and IR, NMR, elemental analysis. The created analytical reagent (DBA) when reacts with iron produces red complex, this complex can be extracted by using n-butanol as a selected solvent, maintained at constant pH 7.8. The Beer's law is followed in the concentration of 1-10 µg Lit^{-1} of Fe (III) and the optimum values of maximum absorption, molar extinction coefficient, and sandal's sensitivity to the red complex, are observed to be 430 nm, 4954 Lit $mol^{-1}cm^{-2}$ and 0.01203 µg cm^{-2} respectively. Accordingly to which it turned out that the reagent is the best for the assessment for estimation of Fe (III) from various sources of samples.

Keywords: *Fe (III), Spectrophotometric determination, DBA reagent, molar absorptivity*

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1. Introduction

The traditional methods such as ion exchange, membrane separations, solvent extraction, and chemical adsorption for removal of heavy metals from industrial wastewater are being used [1,2]. The removal of heavy metals from industrial effluents, to control environmental pollution liquid-liquid extraction is one of the cheapest and effective techniques commonly employed [3-11].

Iron (Fe) is the commonly found metal, in the Pharmaceutical waste, chemical waste and industrial waste. Several research findings show various analytical methods developed for estimation Fe (III) [12-18]. A spectrophotometric extraction research work formation of a complex between Fe (III) and analytical reagent was studied by Micheal and Dermot [19]. Various analytical reagents were suggested by several researchers to estimate Fe (III) in various samples by spectrophotometric extraction [20,21,22,23].

The above literature survey shows that till date, no work has reported for predications of Fe (III) metal ions by using DBA as an analytical novel reagent. Hence, the attempt is made in present work on the 2, 4 dimethyl-3H-1,5 benzodiazepine fresh, novel reagent for the estimation of minute level quantities of Fe (III) using spectrophotometric extraction.

2. Methodology

2.1. Instrumentation for Experimental Research

During the experimentation the absorbance was measured by using calibrated UV, visible spectrophotometer (Shimadzu 2450 UV-Visible with 10 mm quartz cell) and pH was maintained by using calibrated digital pH meter (Elico LI-120). The parameters maintained during experimentation are depicted in Table 1.

Table 1. Experimental parameters

Parameters	Values
Maximum Absorbance	430 nm
Solvent used	n- butanol as best among studied
pH required	7.8
Equilibration time observed	60 seconds
Stability of Fe(III)- reagent	48 hrs
Optimum Beer's concentration limit	1 to 10 mg cm^{-3}
Molar exction coefficient	4954 lit $mol^{-1}cm^{-2}$
Sandell's sensitivity	0.01203 mg cm^{-2}
Mole ratio of Fe (III) : DBA	1:1

2.2. Preparation of Analytical Reagent

The analytical reagent is prepared by mixing 1 mole of o-phenylenediamine and 2 moles of Acetyl acetone in the

Ethanol as solvent. The mixture obtained is then refluxed for the duration of 2 hrs in the round bottom glass flask. The obtained solution from above process is poured on ice to get crystals. This results a solid product, which is crystallized by using ethanol as a solvent. Further, it is then synthesized and characterized and used as analytical reagent for spectrophotometric identification of Fe (III) ions. A stock solution of above analytical reagent concentration of 0.05% was made ready in a methanol. The reaction scheme is as indicated in Figure 1.

2.3. Stock Solution

An accurately measured amount of ferric chloride was added to purified water, and then diluted to desired quantity.

2.4. Effect of pH on Extraction

In this study, different Buffer solutions were used for Fe (III) extraction with varied range of pH from 1 to 11, maintaining constant molar ratio 1:1 between organic phase and an aqueous phase. Figure 2 indicates the variation of absorbance with pH. It is observed from this figure that absorbance increases with increasing pH and touches maximum to the corresponding value of pH 7.8 and thereafter rise in pH, reduces absorbance significantly. Hence, the value of pH 7.8 was used in further investigations

2.5. Selection Solvent

Different organic solvents were tested in this experimental work to identify the suitability of solvent and presented in Figure 3. The n-butanol is seems to be suitable solvent as indicated in the same figure.

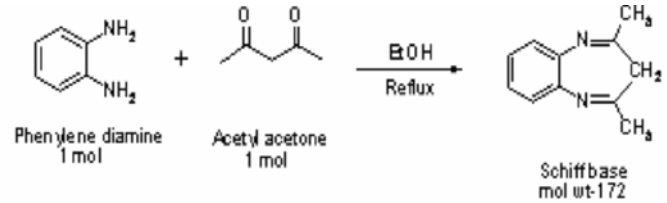

Figure 1. Reaction for preparation of analytical Reagent 2, 4-Dimethyl -3h- 1,5 benzodiazepine (DBA)

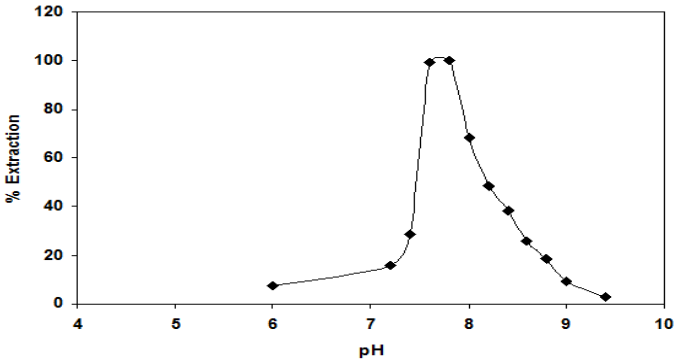

Figure 2. Variation of Absorbance with pH

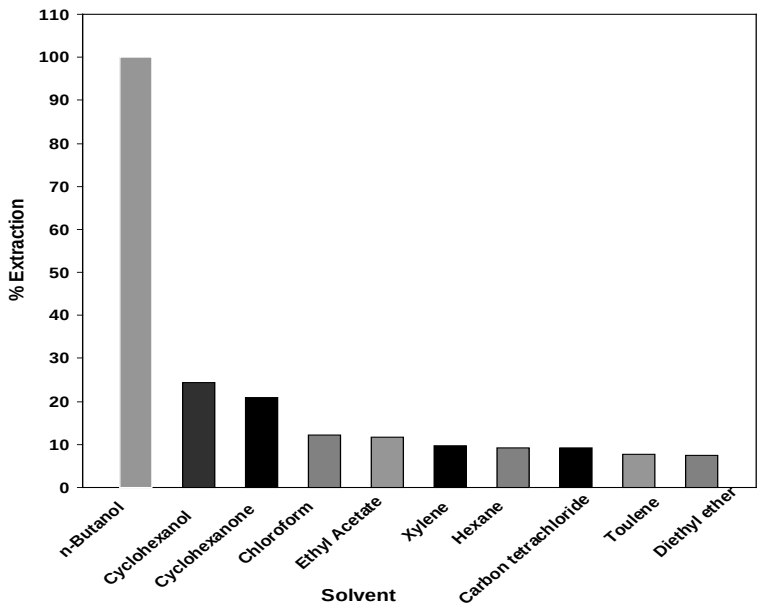

Figure 3. Selection of Solvent

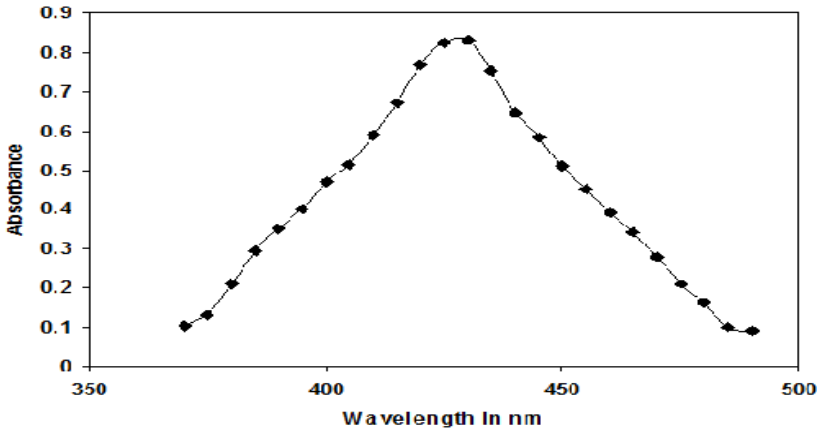

Figure 4. Variation of Absorbance with Wavelength

2.6. Selection of Wavelength

We will notice the absorbance in a Figure 4 that increases with increasing wavelength and attains a maximum value of absorbance at the corresponding wavelength of 430 nm for n-butanol as a solvent. Further, increase in wavelength reduces absorbance. This value of wavelength was recommended for further investigations

3. Experimental Procedures

In a beaker mix 1 ml solution of ferric chloride (1 to 100 mg), and 0.05% reagent in methanol is thoroughly mixed. The pH is then adjusted to 7.8 by adding the buffer solution. The above solution is then added to separating glass funnel with 10 ml n-butanol. The organic and aqueous phases were separated. The organic phase is then subjected to spectrophotometer at a wavelength of 430 nm.

3.1. Development of Calibration Curve

Figure 5 shows the variation of absorbance with concentration of Fe (III) ions. By following the same method as demonstrated in experimental procedures, the samples of Fe (III) with different concentrations were prepared and its absorbance was measured for development of Calibration curve.

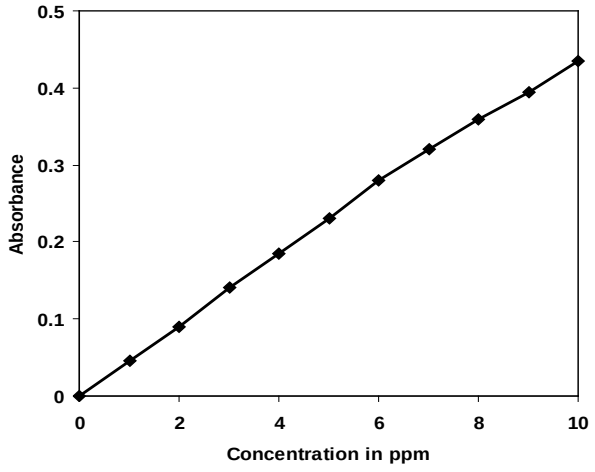

Figure 5. Development of Calibration Curve

3.2. Selection of Molar Ratio between Fe (III) and Analytical Reagent

Figure 6 shows the selection of molar ratio between Fe (III) and analytical reagent using various methods. The Job's continuous variation method was extensively used for fixation of the composition ratio of the extracted species, and the same results were further validated by using, mole ratio method and slope ratio method. From, these methods a molar ratio between Fe (III) and analytical reagent to be fixed as1: 1.

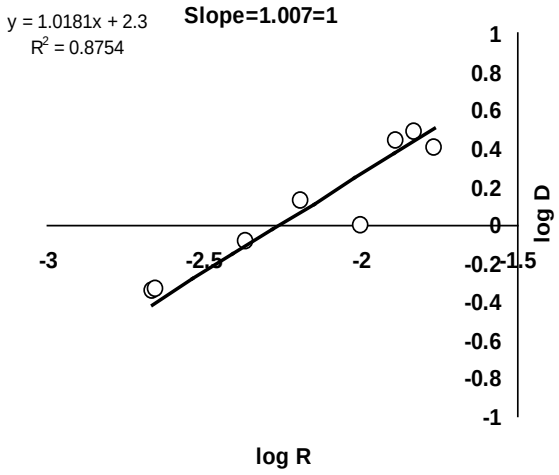

Figure 6. Selection of molar ratio between Fe (III) and analytical reagent

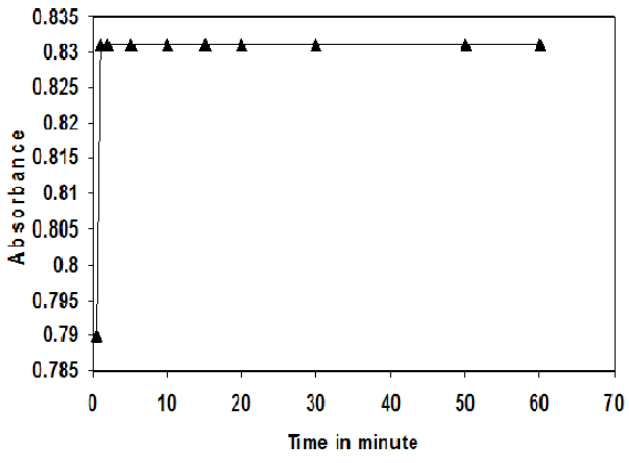

Figure 7. Study of Equilibrium time on Fe (III): DBA

3.3. Study of the Equilibrium Time

The influence of pulsating time on the extraction was studied in the range of few seconds to 60 minutes. From Figure 7 it was observed that the quantitative extraction was achieved after 10 seconds. The further shaking time has no effect on the percentage extraction of the Fe (III).

3.4. Stability of the Complex with Time

Figure 8 presents the effect of time on stability of extracted species; the stability of extracted species is noticed up to 36 hours. Due to this reason, during the experimentation absorbance measurement was done within 1.25 hour time span.

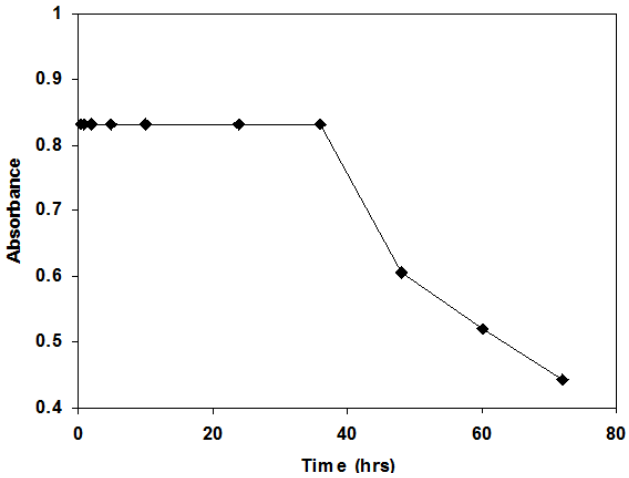

Figure 8. Influence of time on the stability of extracted species

3.5. Effect of Diverse Ions

Various ions interfere at different concentration range estimation of Fe (III).The positive and negative ions were tested to find their limit in the prediction of Fe (III).The details of such ions and their tolerance limit with minimum error are reported in Table 2. These ions are further suppressed during actual prediction of Fe (III) and presented in Table 3.

Table 2. Influence of diverse ions

S. No.	Diverse ions	Acceptability
1	Per-chlorate	8
2	Per-iodate	11
3	Mn(II), Sn(II),Ti(IV),Cr(II)	13
4	W(VI)	15
5	K+, NH_4+	17
6	Zinc, Ni^{+2}Mn(II), Zr(II), Co(II), Cu(II), EDTA	Interferes all cations

Table 3. Suppressing reagents

Interfering Ion	Added Suppressing agent
Mn (II)	Sodium Iodide
Ag +	Heated with Conc. HNO_3
EDTA	Boiled with Conc. HNO_3
Cr (VI)	Thiourea
Cd (II)	Sodium flouride
Ce (IV)	Potassium Iodide

3.6. Analytical Reagents and Their Limitations

Table 4 presents the comparison between different analytical reagents used in the earlier study and proposed analytical reagent used in the present work. This table clearly indicates that the proposed analytical reagent is appears to perform well over the earlier reported data.

Table 4. Analytical reagents and their limitations

Analytical Reagent	Disadvantages
2- Mercapto pyridine- 1-oxide	Copper interferes
Pyridazine-3,6-diol	Pink coloured complex.
Phenanthraquinone monoxime	Sandell sensitivity is 27 µg cm^{-2}
5,5' – thiodisalicylic acid	Violet coloured complex. M:L ratio is 1:1
2-(1,2,3- triazolylazo)-5-diethyl aminophenol	Interference of Cu can masked by using thiourea

4. Uses of Present Study

This experimental investigation for detection of Fe (III), find a wide application in the areas of industrial, food, and environmental. The proposed analytical reagent seems to be superior as compared to earlier reported data.

Table 5. Use of present study

SN	The sample tested	Quantity of Fe(III) (standard method)	Quantity of Fe(III) (present method)
1	Ferrochrome	57%	55.9%
2	Hemitite	69.9%	69.12%
3	Water sample (per Lit)	0.11 µg//gm	0.096 µg//gm

5. Conclusion

In the present experimental studies, a new organic compound 2, 4-dimethyl -3H- 1, 5 benzodiazepine (DBA) was devised to estimate Fe (III) by using spectrophotometer. The fresh organic compound is observed to be more effective over literature data. The present method is found to be best, simple and effectively applied for estimation of Fe (III) from various sources of samples.

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Conflict of Interests

With reference to publication of the present paper, author hereby state that no sort of conflict of interest associated to this work.

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