Development of method for extractive spectrophotometric determination of Fe (ll) of 2-{4-(1h benzimidazole-2-yl) phenyl imino}-2-hydroxy-4-methoxy benzaldehyde as an analytical reagent

Ritima Singh, Department of chemistry, The Institute of Science, Mumbai, India, ritimasingho2@gmail.com

Ratnamala P. Sonawane, Department of chemistry, The Institute of Science, Mumbai, India

Atul Singh, Department of chemistry, The Institute of Science, Mumbai, India

Abstract: A spectrophotometric method has been developed for the determination of Fe (II) using 2-{4-[1h benzoimidazole-2-yl] phenyl imino}-2-hydroxy-4-methoxy benzaldehyde (BPIHMB) as an acquired reagent. BPIHMB with Fe (II) formed red coloured complex at the range of P^H 9.8 in an n-butanol solvent and showed maximum absorption at 370nm. This method complied with Beer's law within 1 to 100ppm. Sandell's sensitivity and molar absorptivity were obtaining 0.16636 $lmol^{-1}cm^{-1}$ and $0.2357x10^{4,}$ respectively. The introduced work is profound precipitant and selective. This method employed for alloy, synthetic mixture and commercial samples successfully.

***Keywords* — Iron, Spectrophotometric determination, n-butanol solvent, commercial sample, 2-hydroxy-4-methoxy(2H4M), BPIHMB.**

I. INTRODUCTION

Iron is one of the most plentiful elements. It is the 6th most readily available element in the universe. It is cheap to produce and has a large number of different uses [1]. A significant amount of iron oxides are present on the surface of the earth. It has played a very important role in our lives. Most of the iron used to manufacture steel. This is used for the construction of buildings, vehicles, ships, tools, electrical equipment oil and rail transportation. It is also very important nutrient for both plants as well as for animals. It is part of haemoglobin [2] which transport the oxygen in the body.

In-plant iron is present in chloroform which is important for photosynthesis, and it also works as micro-nutrient in the plant, so it is used in agriculture for growth of the plant. Iron has some unique properties as compared to other metal which keeps it in high demand. A deficiency can cause serious health problem in humans like anaemia [3], vomiting, diarrhoea, coma, and abdominal pain.

A rich source of iron in food includes red meat [4], beans, fish, and green leafy vegetables. Ferrous fumerate, ferrous gluconate and ferrous sulfate are the tablets which containing iron compound are used for older people and children, catalase [5], lipoxygenases [6] and IRE-BP [7] are enzymes which contain iron and vital for life.

The soluble salts of Fe have various pigments and treat the wastes of industries and sewage [8]. A minute fence which is made by iron used for prevents insect entry into the home. Iron catalysis is used for the production of ammonia in the Haber process.

The iron is important for all but excessive iron in the human body can cause damage to organs [9]. In the woman, due to menstruation and by the other types of bleeding causes a lack of iron in them. Therefore, it is essential to emulate accurate, selective and sensitive analytical method for the determination of iron at notch level.

A numerous reagent [10-21] used for determination of iron like hydrazine, thiosemicarbazone, oxime. But these methods are bear from some limitations as equilibrium time requirement of some masking agent.

The present exploration is free from limitation, simple, rapid, sensitive and also precise for the analytical method. It will be applied for the determination of synthetic mixture alloy and for pharmaceutical. Solvent extraction is used for separation of metal ion due to its simplicity and rapidity.

EXPERIMENTAL METHOD

The reagent 2-{4-[1h benzoimidazole-2-yl] phenyl imino}-2-hydroxy-4-methoxy benzaldehyde has been prepared through the mentioned process. Firstly the

 DOI : 10.35291/2454-9150.2019.0579 © 2019, IJREAM All Rights Reserved.

preparation of 4-(1H Benzimidazole-2yl) aniline. Take 0.05mol of o-phenylediamine and 0.05mol of p-aminobenzoic acid were contracted in a round-bottomed flask with 50ml 4N HCl Refluxed for 1hours. Subsequently the brownish coloured of the mixture was cooled and filtered off and then recrystallised by ethanol.

Then the equimolar quantity of the prepared product and 2H4M contracted in another clean round bottom flask with 40ml of ethanol to refluxed for 2 hours. Later on, it was cooled and drained off. The novel product was recrystallised from absolute alcohol. The yellow colour product conformed by TLC and by M.P. M.P- 220^{0}C

The preparation of Fe^{2+} has been readied by dissolving 0.07024gm of its $(NH_4)_2SO_4.FeSo_4.6H_2O$ into 100ml of double-distilled water. It contains dilute sulfuric acid. It has been diluted to the necessary volume using double distilled-water and then standardised through o-phenanthroline. The estimation of Absorbance and pH were found using a UV-Visible spectrophotometer with 1cm quartz cells along with combine glass electrode of P^H-meter.

A. Extraction procedure

Firstly, 1.0ml aqueous-solution was blended in 50ml beaker. The solution contains 0.1mg of Iron metal and 1 ml of reagent. The pH of the solution adapted to 9.8 with the help of 0.2M boric acid and potassium chloride by fixing 10ml volume. The solution then shifted onto 100ml separator-funnel. The beaker has been cleaned two times with n-butanol. It was then shifted to the same funnel.

The two different phases had been agitated in three minutes. It was kept to segregate. Then, the organic phase was compiled in 10ml measuring-flask. It was made up to the mark with organic-solvent, if necessary. After segregation, pH of aqueous phase as well as Fe (II) in every phase has been estimated through o-phenanthroline.

II. RESULT AND DISCUSSION

Reagent BPIHMB made dark reddish coloured complex along with Fe (II) that has been extracted as organic-phase. The extraction of Fe (II) makes aqueous-phase from BPIHMB in n-butanol. This extraction was analyzed across a range of experimental conditions. The results from the observations have been explained in the following section.

A. Extraction with pH variation

The extraction of Iron with 2-{4-[1h benzoimidazole-2-yl] phenyl imino}-2-hydroxy-4-methoxy benzaldehyde was explored over 1-10 pH range.

It was found that extraction is maximal at pH 9.8 (Figure 1).

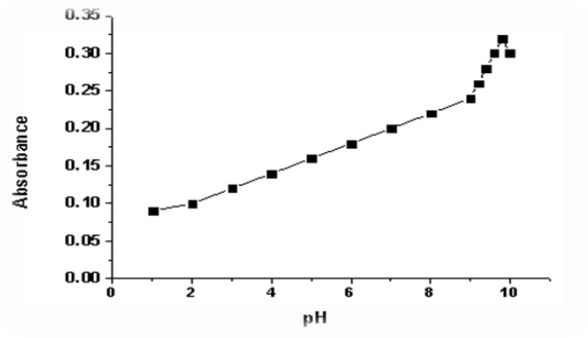

Fig.1. Percentage Extraction as a function of pH

B. Absorption spectrum

Fe (II) with BPIHM in n-butanol shows utmost absorption at 370nm. At this wavelength, the absorption due to BPIHMB is nearly negligible (Figure 2).

Therefore the absorption measurements at 370nm were selected for further work.

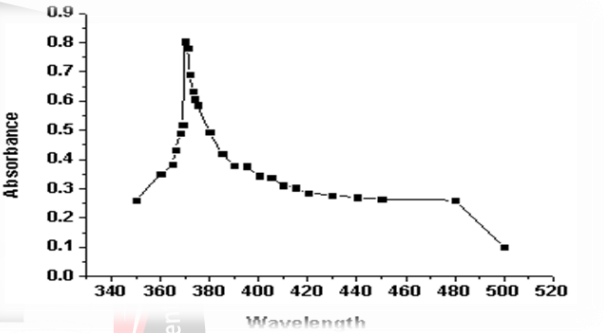

Fig.2. Absorbance spectrum of Fe (II) with BPIHMB

C. Influence of diluents

Ethyl acetate, hexane, toluene, n-butanol, chloroform, xylene, pentane, benzene, methyl iso-butyl ketone, ethyl methyl ketone are the different organic solvent that was used for the analysis of congruence of diluents.

The extraction of Fe (II) shows good influence with BPIHMB in n-butanol, so consequently, n-butanol was selected as forwarding studies; Moreover, it indicated superior and intense phase-separation.

D. Effect of salting-out agent

The effect of the presence of 0.1M salts of different alkali as well as alkaline metal over absorbance of Fe (II): BPIHMB complex extract were negligible. Hence, the salting-out agent was not needed in extraction.

E. Effect of Reagent-concentration

Sample solution having 60µg of iron at respective pH values were mixed with different volume of 0.1% reagent solution.

It was found that the absorbance stayed approximately constant if the volume of reagent solution was higher than 1ml. Hence, 1ml of 0.1% reagent has been selected for the

 DOI : 10.35291/2454-9150.2019.0579 © 2019, IJREAM All Rights Reserved.

quantitative finding of metal.

F. *The consequence of equilibrium-time and the stability of complex*

Variation in absorbance with a change in equilibrium time extraction of the complex into organic solvent has been studied. It was observed that that equilibrium-time of 60s is adequate in quantitative extraction of Iron.

Stability in the colour of the Fe (II): BPIHMB complex with respect to time has indicated that the absorbance from extracted species is stable up to 72 hours. However, after that, a small reduction in absorbance was witnessed. (Figure 3).

The measurements were performed within 1hr of extraction of iron during experimentation from practical conveniences.

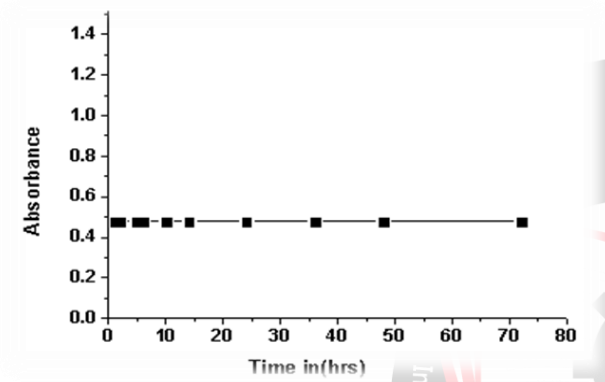

Fig.3. Stability of Fe (II) with BPIHMB

G. *Calibration plot*

A calibration plot demonstrates the concentration of the substance. For a calibration plot, the prepared solutions containing a fixed concentration of BPIHMB solution and different concentration of Fe (II) solution in the range of 1 to 10 ppm (Figure 4).

The graph plotted against Fe (II): BPIHMB was linear and reproducible. It indicates that it obeys Beer's law. The molar absorptivity is obtained as 0.2357×10^{4} $L.mol^{-1}.cm^{-1}$ whereas Sandell sensitivity as 0.16636 $\mu g/cm^{-2}$.

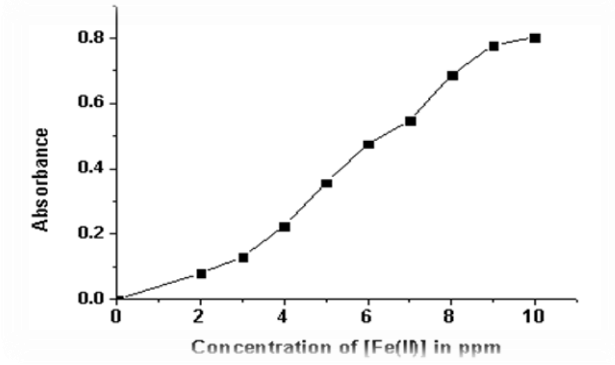

Fig.4. Calibration plot of Fe (II) with BPIHMB

H. *Nature of extracted species*

Use of different method such as Job's continuous variation method (Figure 5), Slope ratio method (Figure 6) and by Mole ratio method (Figure 7) found that the erection of extracted species. It shows the molar ratio of Fe (II): BPIHMB complex was 1:2.

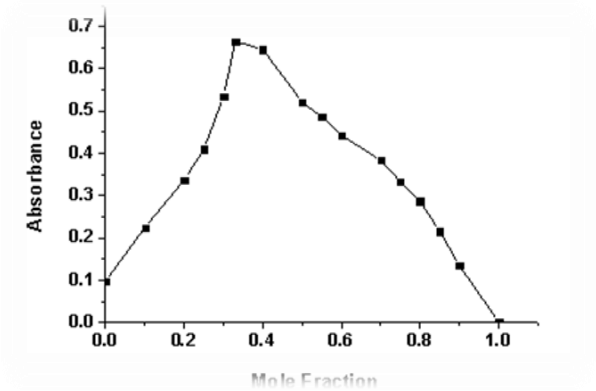

Fig.5. Method of Job's Continuous variation

Fig.6. Slope ratio (Fe (II) with BPIHMB)

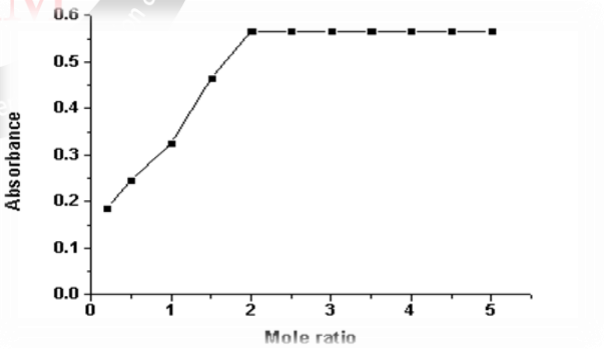

Fig.7. Mole ratio (BPIHMB: Fe (II))

I. *Effect of divalent ions and foreign ions*

No interference in the spectrophotometric estimation of 60 µg of iron (Table 1) was observed from the other ions presents in various amount.

The ions that exhibited interference in the spectrophotometric estimation of iron have been subdued with the help of necessary masking agents.

 DOI : 10.35291/2454-9150.2019.0579 © 2019, IJREAM All Rights Reserved.

Table 1: Effect of divalent ions and foreign ions

Sr. No.	Ion	Added amount in Fe	Absorbance on 370 nm
1	---	---	0.476
2	Cl^-	14.0	0.476
3	Br^-	16.0	0.476
4	I^-	10.0	0.476
5	SO_4^{-2}	18.0	0.476
6	PO_4^{-3}	7.0	0.476
7	NO_3^-	14.0	0.476
8	NO_2^-	16.0	0.476
9	$O_2O_4^{2-}$	12.0	0.476
10	CH_4N_2S	19.0	0.476
11	SO_3^-	13.0	0.476
12	CN^-	12.0	0.476
13	F^-	9.0	0.476
14	Zn (II)	6.0	0.476
15	Ba (II)	7.0	0.476
16	Ca (II)	10.0	0.476
17	Ni (II)	11.0	0.476
18	Cu (II)	7.0	0.476
19	Pb (II)	14.0	0.476

J. Precision and Accuracy

The precision and accuracy of the presented spectrophotometric method were studied by analysing five different solutions. Each solution contains 80 µg of iron in its aqueous phase.

The average of five calculations was 80.05. The variation from mean at 99% confidence limit was ± 0.1872.

K. Applications

The postulated method was effectively employed for the estimation of Iron from different alloys and synthetic mixtures.

The findings were observed to be equivalent to those estimated from the standard method (Table 2).

Table 2: Determination of Fe (II) Using BPIHMB from different samples

Sr. No.	Sample	Amount of Fe (II)	
		Standard method	Present method
1	**Alloys** Steel Hematite	 67.2% 35.0%	 67.16% 34.96%
2	**Capsule/ tablets** Austrin Ferium XT Globiro R.B Tone	 32.86mg 100 mg 50.0mg 100 mg	 32.85mg 95.94mg 49.96mg 96.10 mg
3	**Synthetic mixture** Fe (II) + Zn (II) Fe (II) + Mg (II)	 4.99 ppm 4.99 PPM	 4.89 ppm 4.97 ppm

III. CONCLUSION

The introduced method in present work is highly sentient and selective as compared to other reported method BPIHMB with Fe (II) metal is vastly easy to prepare than others. It formed red coloured at pH 9.8. It gives progression, such as durability and reproducibility.

This method is very facile fast and sensitive, instant colour development and tolerate from scanty interference. The estimation of Fe (II) at trace level in medicine, alloy and for synthetic mixture has been prosperously applied.

REFERENCES

[1] Venugopul. B.; Luckey, T.D. Metal Toxicity in Mammals-2, plenum press, New York, Vol.2, 3rd ed., 1662, 1981.

[2] Kikuchi,G.; Yoshida, T.; Noguchi, M. "Heme oxygenase and heme degradation." Biophysical Research communications. 2005, 338(1): pp. 558-67.

[3] Beguin, Y; Aapro, M; Ludwig, H; Mizzen, L; osterborg, A "Epidemiological and non clinical studies investigating effect of iron in carcinogenesis- a critical review" critical Reviews in oncology/ Hematology.2014, 89 (1):pp. 1-15.

[4] Micronutrient Information center, Linus pauling Institute Oregon State University, Corvallis, Oregon. April 2016. Retrieved 6 March 2018.

[5] Boom EM, Downs A, Marcey D, "Proposed Mechanism of Catalase". Retrieved 11 February 2007.

[6] Boyington JC, Gaffney BJ, Amzel LM "The three-dimensional structure of an arachidonic acid 15-lipoxygenase". Science. 1993, 260(5113):pp. 1482-86.

[7] Gray, N.K.; Hentze, M.W. "Iron regulatory protein prevents binding of the 435-translation pre- initiation complex to ferritin and Ealas Mrnas" EMBO J. 1994, 13(16):pp. 3882-91.

[8] M. Jamaluddin Ahmed, Uttam Kumar Roy; Turk J Chem, 2009, 33:pp. 709-726.

[9] Ellis J T, Schulman J, Smith CH Generalized Siderosis with fibrosis of liver and pancreas in Cooley's anaemia with observation on the pathogensis of the Siderosis and fibrosis. Americans Journal of Pathology. 1954, 30(2):pp. 287-309.

[10] Gao, H.W.; Chen, C.; Yang, Q.Z.J of AOAC Int., 88, 2005, pp. 1060-1164.

 DOI : 10.35291/2454-9150.2019.0579 © 2019, IJREAM All Rights Reserved.

[11] Zarei, K.; Atabati, M.; Malek shabuni, Z. Anal. Chem. Acta., 556, 247 , 2006.

[12] Adel S.Orabi, Adel Marghany, Medhat A.Shaker, Alaa E. Ali."Spectrophotometric determination of Fe (II), Cu(II) and Uo2(II) ions by a new analytical reagent derived from condensation ofMonoethanolamine and acetyl acetone." Bulletin of the chemist and Technologists of Macedonia, 2005,Vol. 24 No. 1, pp 11-19.

[13] Banerje, D. Tripathi, K.K. "Spectrophotometric determination of Iron and Copper with Methyl-2-Pyridyl Ketoxime and Their Simultaneous determination in Mixture", Anal. Chem., 1960, 32(9), pp 1196-1200.

[14] Nahid Pourreza, Saadat Rastegarzadeh, Ali Reza Kiasat and Hossein Yahyav, "Spectrophotometric determination of Iron (II) after Solid phase extraction of its 2,2' Bipyridine Complex on Silica Gel-polyethylene Glycol." Journal of spectroscopy. 2013, pp 1-6, ID 548345.

[15] Basheer K. Adebayo, Segun Ayejuyo, Hussein K. Okoro and B. J. ximba "Spectrophotometric determination of iron (III) in tap water using 8-hydroxy quinoline as a chromogenic reagent." Afr. J. Biotechnology. 2011, Vol 10(71), pp. 16057.

[16] Ratnamala P. Sonawane, Ritima singh "Development of Method for Extractive Spectrophotometric determination of Fe(II) with 2-[{4-(1,3 Benzoimidazole-2-yl) phenyl}Imino]-5- Nitro phenol as an analytical reagent. International Research Journal of Nature and Applied Sciences, 2015, Vol.2, ISSUE 10, pp 71-81.

[17] Mandhare D.B.; Barhate V.D. "Extractive specatrophotometric determination of Fe(II) with [N-(o-hydroxy benzylidene) pyridine -2- amine]." Int J. curr pharm Res. 2016, vol 4, ISSUE 1, pp. 32-35.

[18] Shigeroku Yamaguchi and Katsuya Uesugi, "extraction Spectrophotomtric determination of iron (III) with 2-hydroxy-1- naphthaldehyde Oxime". Analyst. 1984, ISSUE 11, 109, pp. 1393-1395.

[19] Kulieve Kerim A. Plotnikava Svetlana E., Gourbumova Elena M "Extractive Spectrophotometric of iron (III) using 2,6-Dithiol-4- Methyl ethanol and Amino phenols." Journal of Advanced applied scientific Research, Vol-1 ,1984.

[20] Shawket Kadhim Jawad, Mousa Umran kadhium Ebaa Adnan Azoz "Separation and Spectrophotometric determination of Fe(III) and Mercury(II) via cloud point Extraction with New Azo-derivatives ." Eurasian Journal of Analytical Chemistry. 2018, 13(5).

[21] G.H. Jeffery, J. Bassett and R.C. Denney, Vogel's Textbook of quantitative Chemical Analysis 5th Edn . ELBS, London 1991.

 DOI : 10.35291/2454-9150.2019.0579 © 2019, IJREAM All Rights Reserved.