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Spectrophotometric determination of iron (III) in tap water using 8-hydoxyquinoline as a chromogenic reagent

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Abstract
A simple, rapid and sensitive spectrophotometric method was developed for the determinationation of trace amounts of iron (III) using 8-hydroxyquinoline as a chromogenic reagent. The proposed method was based on the reaction of iron (III) with 8-hydroxyquinoline in chloroform solution to form a metaloxine complex having a maximum absorption at 359 nm. Beers law was obeyed in the range of 1 to 14 ug/ml Fe3+. The recovery was between 98.60 and 103.30% with a coefficient of variation of 1.209%. The method was successfully applied to tap water samples and comparison with standard method showed the new method to be accurate and precise as the more sophisticated AAS commonly used for Fe determination.

Keywords: Chloroform, hydroxyquinoline, iron, spectrophotometry, oxine, spectrophotometry, tap water

Introduction
Water is the basis of life, an ecological resource for the earth's flora and fauna and a fundamental necessity for human life. If there is no adequate supply of safe water, we have no hope of improving the health of the people in our partner countries. It has been estimated that 80% of all diseases are in some way, connected with contaminated water (WHO, 2007). It is difficult to achieve productive human activity without a functional water supply be it agriculture, forestry, trade or industry. Adekunle *et al*. (2007) [4] highlighted some problems associated with lack of adequate and quality water resources in India; they include threat to health of about 40 million people. Globally, pathogenic contamination of drinking water poses the most significant health risks to humans. There have been countless numbers of disease outbreaks and poisoning throughout history resulting from exposure to non pathogenic, toxic contaminants that are often globally ubiquitous in drinking water (Ritter *et al*., 2002). The latest World Bank studies suggest that it would cost an excess of USD 109 a year to reduce the problems if ground and surface water contamination goes unchecked. Iron is an objectionable constituent of potable water. Presence of iron in water impacts a bitter and metallic characteristic taste and oxidized precipitate. Water containing significant qualities of iron may appear clear when drawn and change quickly upon exposure to air. This process is called oxidation and it involves the conversion of ferrous ion to ferric ion. Iron stains everything with which it comes in contact; 0.3 ppm standard is often recommended and is based on preventing taste and staining problems for humans (Damron and Eldred, 2002) [5]. The World Health Organization (WHO) recommends 0.3 mg/l (WHO, 1993) as maximum concentration limit of iron in drinking water. Federal and state regulations limit the iron content of drinking water to <1 ppm, though iron is easily determined in contaminated water containing > 1 ppm (1mg/l). Iron in excess of 0.2 to 0.3 mg/l may cause nuisance even though its presence does not affect the hygienic quality of water (0 to 0.3 mg/l acceptable. 0.3 to 1.0 mg/l satisfactory and over 1.0 mg/l unsatisfactory).

Iron is commonly present in surface and drinking water. This occurs from dissolution processes of ironstones in nature and partially from the corrosion processes in pipes (Pitter, 1990). Industrial wastewater is also a significant source of iron. The iron concentration in water can vary over several orders of magnitude from tens of µg/l (sea water) to tens of mg/l (source). The concentration of iron in most ground and surface water is not very important from the hygienic point of view; its content influences, however the organo leptic properties of drinking water, mainly the color, taste and turbidity.

Iron concentration thus becomes a parameter for the quality of drinking water. However, wastewater also has concentration limits, but their limit varies for various industrial branches. From the literature, maximum limits are set between 5 and 10 mg/l (Pavael *et al*., 1997).

The iron content of water can cause technical faults rather than sanitary problems; iron concentration of just 0.5 mg/l can lead to turbidity of water. Small amounts of Fe (II) can cause iron bacteria growth leading to the clogging of pipes and an offensive odour of water. Several analytical methods have been used for the determination of iron in water. They include HPLC methods (Nagosa *et al*., 1991; Yi *et al*., 1992), which are based on the separation of various complexes of iron and other cations offering the possibility of multi element analysis. Necessity of sample pre-treatment such as SPE or solvent extraction is the demerit of these methods.

Various spectrophotometric procedures have also been employed; some of them are combined with flow injection analysis (Pavel *et al*., 1997). Atomic absorption spectrometry and inductively coupled with plasma spectrometry have also been applied for the determination of iron in water and other cations (Abollino *et al*., 1995) [2]. Iron has been separated by capillary isotachophoresis as an EDTA complex for the simultaneous determination of EDTA-Fe (III) and free EDTA not in water but in wash liquids used for the desulfurization of gases. EDTA-Fe (III) complex has also been used for the determination of EDTA in manganese (Ito *et al*., 1980). However, an intensely colored complex must be formed to detect the presence of these low levels of iron spectrophotometrically. Usually, when trace quantities of an analyte are being measured, cleanliness of equipment, glassware etc. is essen-tial to prevent positive determinate errors due to laboratory contamination (Skoog *et al*., 2010).

Furthermore, in analytical chemistry, special attention is given to development of methods simultaneously combining Pre concentration and determination. A promising technique therefore, is the use of ready analytical methods (Gavrilenco and Markova, 2008). Methods are continually being developed to improve the selectivity, sensitivity and specificity of an analyte. It is equally important to consider new methods in terms of cost, convenience and the number of sample throughout. The precision and accuracy of methods are also critical factors in any analysis. Spectrophotometric methods for the determination of elements are based on the absorption of visible and near ultraviolet radiation. Visible spectrophotometry was often called colometry and such definitions as colorimetric photometric or absorptometric methods are sometimes used in the literature, as equivalent to the terms spectrophotometric method (Sandwell, 1985). The basis of spectrophotometric methods is the simple relationship between the absorption of radiation by the solution and the concentration of species in the solution. In order to determine a species or analyte in the solution spectrophotometrically, it is usually converted into a colored complex. The colour of the analyte or itself is utilized much less often (Sandwell,1985). When the determinant is not coloured, or forms no coloured compound, indirect spectrophotometric method may be used for its determination or ultraviolet spectrophotometry may sometimes be used. Spectrophotometric methods are remarkable for their versatility, sensitivity and precision. Almost all are direct and can be used for all the elements except for the noble gases. A very extensive range of concentration may be covered from macro quantities to trace (10-8 to 10-6%). Spectrophotometric methods are among the most precise instrumental methods of analysis (Edisbury, 1990) [6]. This study was aimed at determining iron in tap water collected from Dav College and some selected locations in Abohar, and some nearby villages using the proposed spectrometric method.

Materials and Methods

Chemicals and apparatus used were 8-hydroxquinoline (oxine), sulphuric acid, chloroform, distilled water, ferric ammonium sulphate, analytical grade aluminum sulphate, separating funnel, beaker, test tubes, pipettes, volumetric flasks, measuring cylinder and UV spectrophotometer (single beam).

Preparation of stock solutions

8.635 g of ferric ammonium sulphate was weighed and dissolved in distilled water containing 3 ml of concentrated H_2SO_4, the solution was diluted to 1L in standard flask. Working solutions were obtained by suitable dilution of the stock solution with dilute sulphuric acid (0.005 M).1.0 g of oxine was accurately weighed and dissolved in 100 ml acidified water with pH 1 to 2. 1.0 g of 8-hydroxquinoline was weighed and dissolved in chloroform in a 100 ml volumetric flask and made up to the mark with chloroform. The flask was covered immediately to avoid evaporation.

Determination of absorption maxima for metal-oxine complex

A solution containing 10 ppm of iron standard was placed in a separating funnel, 10 ml of oxine-chloroform was added and the content was properly shaken for 3 to 5 min. The aqueous layer was allowed to separate by a clear partition between the 1 upper layer (the aqueous layer) and the denser chloroform oxine-metal complex layer. The oxine-metal complex was separated into a test tube and covered immediately. A blank was prepared by adding 10 ml of distilled water to 10 ml of chloroform in a separating funnel. The 2-layers were properly shaken for 3 to 5 min and then separated into test tubes. The absorbance of the 10 ppm oxine-metal complex was scanned at various wavelengths in order to establish the maximum absorbance (Amax).

Standard solution preparation

The stock solution was diluted serially by 0.005 M sulphuric acid. To each of the standard solutions (1 to 10 ppm), 10 ml of chloroform oxine reagent was used for the extraction of the metal oxine complex and their absorbances were recorded. Oxine was prepared in deionised water by weighing and 100 g oxine was accurately weighed and dissolved in 100 ml acidified water (pH 1 to2).

Preparation of calibration curve

Series of iron (III) standard solution were prepared from 1 to 14 ppm. To 10 ml of the solution containing each of the standards,about 0.5 ml of the oxine solution was added. Their corresponding absorbance was measured.

Interference studies

Using oxine chloroform reagent and acidified water solution

To a solution containing l0 ppm of iron (lll) standard, l0 µg/ml of Al (III) was added; the solution was made up to 10 ml. The extraction was carried out the same way as described earlier. For acidified water solution, a solution containing 10 ppm of iron (III) standard,10 µg/ml of Al^{3+} was added, and the solution was made up to 10 ml in a volumetric flask. 0.5 ml of acidified oxine reagent was added to form a greenish color metal oxine complex.

Determination of iron (III) in water samples

To 10 ml each of the water samples, a drop of sulphuric acid was added. It was later treated the same as the standard.

Levels of iron were also assessed in the water sample using atomic absorption technique for comparison.

Recovery and stability studies of the proposed method

To 10 ml aliquot of the water sample, 3 ppm of Fe (III) standard solution was added. The extraction procedure was carried out as described earlier. The absorbance was measured. The standard concentrations were studied for about 72 h by continually monitoring the absorbance. The absorbance of the standard concentration was recorded against time.

Standard addition recovery studies on 10 ppm

To 10 ml of the water samples, 10 ppm of Fe (III) standard was added. The procedure to develop the color was as described earlier.

$$\% \text{ Recovery} = \frac{\text{Amount recovered}}{\text{spiked time}} \times 100$$

Results and Discussion

Preliminary studies had shown that oxine could react with iron III to form a colored complex that could be monitored and developed into a simple spectrophotometric method for the determination of Fe in samples. Efforts were therefore made to optimize the reaction conditions. The spectrum of the complex has a maximum absorption at 359 nm (Figure 1) and this Amax was used in subsequent experiments.

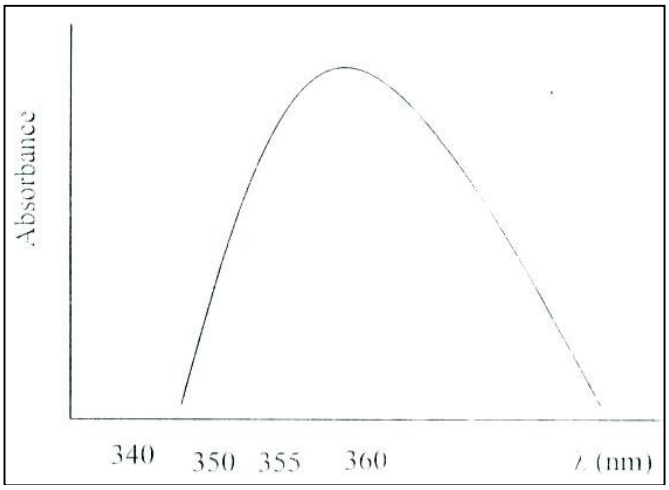
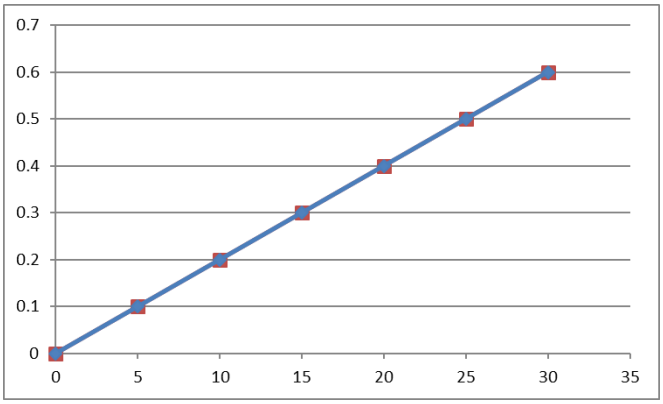


Fig 1: Absorption maxima for iron-oxine complex.



X axis =Concentraion in ppm
Y axis= Absorbance

Fig 2: Calibration curve using oxine chloroform solution

Table 1: Comparison of proposed method result with AAS (ppm).

Sample	Proposed method	AAS
W1	1.02	1.04
W2	1.50	1.52
W3	1.04	1.07
W4	1.96	1.20
W5	2.54	2.55

Table 2: Result of reproducibility of oxine methods.

Sample	Absorbance
1	0.480
2	0.474
3	0.470
4	0.479
5	0.480
6	0.478
7	0.476

Mean value: 3.321; standard deviation: 0.006; coefficient of variation: 1.209%.

Table 3: Recovery studies of spiked Iron (III) from proposed quantitative method.

Sample	Amount added	Amount recovred	Percentage recovery
1	3.00	3.10	103.33
2	3.00	3.00	100
3	10.00	10.05	100.50
4	10.00	9.85	98.50

Preparation of calibration curve

As shown in Figures 2 and 3, the calibration curves were rectilinear up to 10 ppm on investigation using oxine chloroform reagent and up to 14 ppm using acidified oxine as reagent. As presented in Table 1, the mean values for iron in the samples were similar with the results involving the more expensive AAS determination.

Recovery studies

The results of recovery of spiked amounts of iron III in water samples are shown in Table 3. As indicated in Table 3, the method gave very good recovery and the determination of iron (III) using the proposed method would be quantitative.

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