

Spectroscopic Determination of Iron in soil sample using 1,10-Phenanthroline

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Abstract- This study investigated the spectroscopic determination of iron in soil samples using 1,10-phenanthroline as a complexing reagent. The method is based on the formation of an intensely coloured iron (II)-1,10-phenanthroline complex, which was quantified by measuring its absorbance at 510 nm using a UV-Visible spectrophotometer. A series of iron (II) standard solutions was prepared, and a calibration curve was established in accordance with the Beer-Lambert law. The resulting calibration equation was used to determine the concentration of soluble iron in four unknown soil samples. The measured absorbance values for Samples A, B, C and D were 0.32, 0.30, 0.24 and 0.24, corresponding to iron concentrations of 0.611, 0.570, 0.450 and 0.450 ppm, respectively. Sample A recorded the highest iron concentration, while Samples C and D had the lowest concentrations. The results demonstrate the applicability of the 1,10-phenanthroline method for the quantitative determination of iron in soil samples.

Key Word: Iron (II), 1,10-phenanthroline, Soil

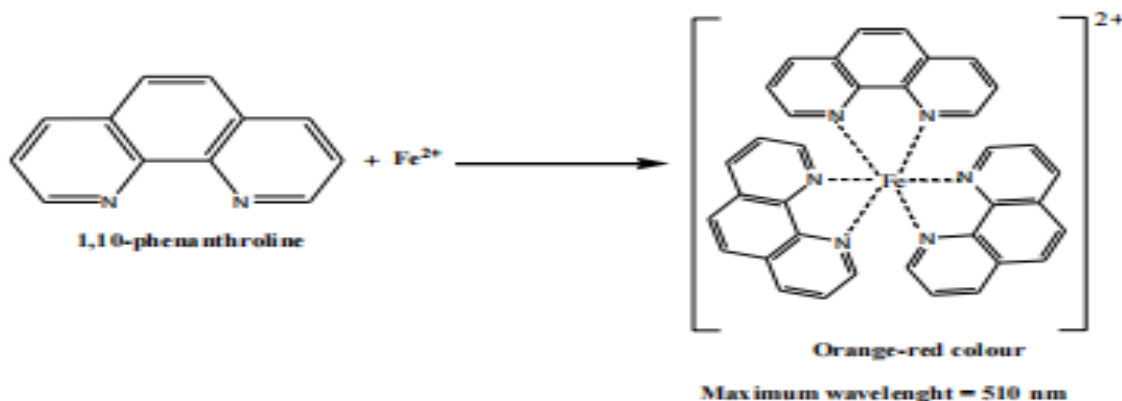
I. INTRODUCTION

Spectrophotometric methods provide simple and sensitive approaches for the determination of iron in environmental samples. Among the commonly used reagents, 1,10-phenanthroline forms a stable, intensely coloured complex with iron (II), making it suitable for colorimetric determination. The intensity of the colour produced is related to the concentration of iron and can be quantified according to the Beer-Lambert law using measurements at an appropriate [6].

Iron (III) is reduced to the ferrous state by hydroxylamine as shown in the equation below



The reduced Iron subsequently reacts with the 1,10-phenanthroline at pH 2 – 9 and forms an orange red complex known as ferroin with a maximum absorbance (λ_{max}) at 510 nm. The intensity of the colour is proportional to the concentration of Iron present in sample solution and obeys Beer's law [4]



Materials/Reagents

The reagents used in the analysis included 1,10-phenanthroline ($C_{12}H_8N_2$), Hydroxylamine Hydrochloride ($C_{21}H_{27}ClN_2O_2 \cdot 2HCl$): 10% aqueous solution, Ferrous ammonium sulfate hexahydrate [$(NH_4)Fe(SO_4)_2 \cdot 6H_2O$], Ammonium acetate ($C_2H_7NO_2$), Buffer solution, (CH_3OH), Acetic acid (CH_3COOH). All reagents were of analytical grade and were used as supplied without further purification.

Sample Area

The samples were collected from Leadtots Development Initiative Farmland located in Tula Kaltungo LGA of Gombe State, Nigeria, about 101 km away from the State Capital and 15 kilometers off the Adamawa-Yola highway. It is geographically located between Latitude: $9^{\circ}48'0''N$. and Longitude: $11^{\circ}30'00''E$ [2].

Sample Collection

The soil samples were collected by using grid sampling technique in polythene bags from four (4) different locations on the farm land, about from 0-20 cm deep using hoe. The samples were labelled as A, B, C and D [5].

Preparation of Soil Samples

The soil samples were air-dried, crushed, and sieved, passed through a 0.16mm sieve to remove gravel-sized materials and then homogenized and sealed in polyethene bags [3].

Digestion of Soil Samples

10 g was weighed and taken in digestion flasks, 40 cm³ of Aqua regia (Nitric acid and HCl in the ratio of 1:3) were added. The mixtures were then heated on a heating mantle to boil until the fumes became clear and were allowed to cool. The digested solution was diluted with deionized water and filtered through a filter paper (Whatman No. 1) into 100 ml volumetric flasks and was diluted to the mark with deionized water. The solutions were finally kept and used for the analyses. [1].

Preparation of Reagents

Stock iron (II) solution: A stock iron (II) solution was prepared by dissolving 0.351 g of ferrous ammonium

sulfate hexahydrate, [$NH_4Fe(SO_4)_2 \cdot 6H_2O$], in distilled water and making the solution up to 100 mL in a volumetric flask [4].

Hydroxylamine hydrochloride solution: A 10% (w/v) hydroxylamine hydrochloride solution was prepared by dissolving 10 g of hydroxylamine hydrochloride in distilled water and making the volume up to 100 mL [4].

Ammonium Acetate Buffer: The buffer solution was prepared by dissolving 62.5 g of ammonium acetate in 40 mL of distilled water, followed by the addition of 175 mL of glacial acetic acid [4].

1,10-Phenanthroline reagent: The reagent was prepared by dissolving 0.1 g of 1,10-phenanthroline monohydrate in distilled water and making the volume up to 100 mL. [4].

Preparation of Iron (II) Standard Solutions

0.2, 0.4, 0.6, 0.8 and 1.0 mg/L was prepared from the stock solution by pipetting 2 mL into 100 mL volumetric flasks, to each flask 2 mL of concentrated HCl, 1 ml of hydroxylamine solution, 10 ml of ammonium acetate buffer, 2 mL of phenanthroline solution were added and made up the solutions to the mark with distilled water [4].

Preparation of Blank Solution

A blank solution was prepared by transferring 50 mL of distilled water into a 100 mL volumetric flask, followed by the addition of the same quantities of hydrochloric acid, hydroxylamine hydrochloride, ammonium acetate buffer and 1,10-phenanthroline used for the standard solutions. The solution was then made up to the mark with distilled water. No iron solution was added to the blank. [4].

Determination of Wavelength of Maximum Absorption

The wavelength of maximum absorption (λ_{max}) of the iron (II)-1,10-phenanthroline complex was determined using the UV-Visible spectrophotometer. The selected standard solution was scanned over the wavelength range of 200–800 nm against the blank solution. The absorbance was recorded at the selected wavelengths, and a plot of absorbance against

wavelength was used to identify the wavelength corresponding to maximum absorption [4].

Spectrophotometric Determination of Iron

The absorbance of the prepared iron (II) standard solutions and the digested soil samples was measured at 510 nm against the blank solution. A calibration curve was constructed by plotting absorbance against the corresponding iron concentration of the standard solutions. The concentration of iron in the soil samples was determined from the calibration equation obtained from the calibration curve [4].

Absorbances taken at various wavelengths for Fe (II) Complex for Determination of Wavelength of Maximum Absorbance (λ_{max}).

Wavelength (λ) nm	Absorbance
200	0.063
300	0.012
400	0.079
500	0.150
600	0.025
700	0.020
800	0.047

II. RESULT/DISCUSSION

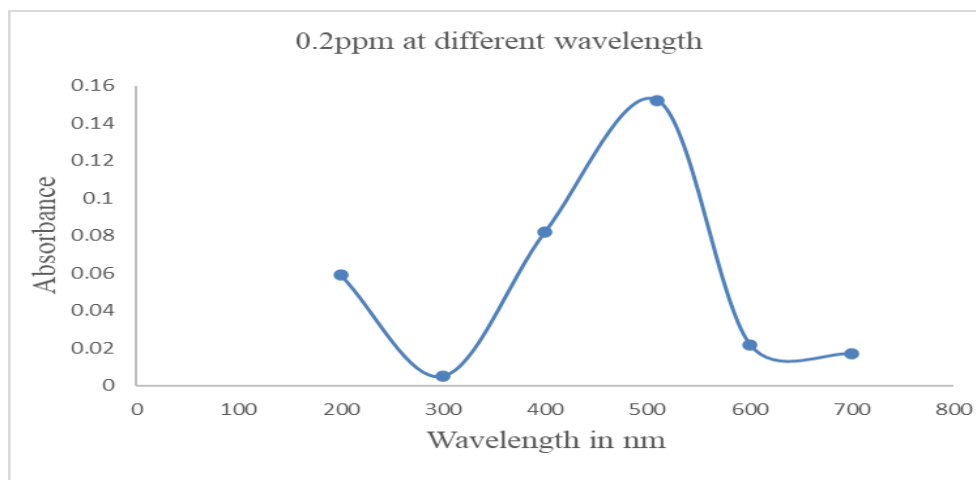


Fig 4.1: UV-Visible Spectrum of the Iron (II) – 1,10- Phenanthroline Complex showing the (λ_{max}) wavelength of maximum absorbance.

From Figure 4.1 the wavelength of maximum absorbance (λ_{max}) was confirmed to be 510 nm for the Fe (II)-1,10-Phenanthroline complex and this agrees with literature [4].

Calibration curve	
Concentrations (ppm)	Absorbances
0.0	0.000
0.2	0.101
0.4	0.211
0.6	0.321
0.8	0.421
1.0	0.523

Table 4.2: Absorbances and concentrations for calibration curve of Iron (II) Complex

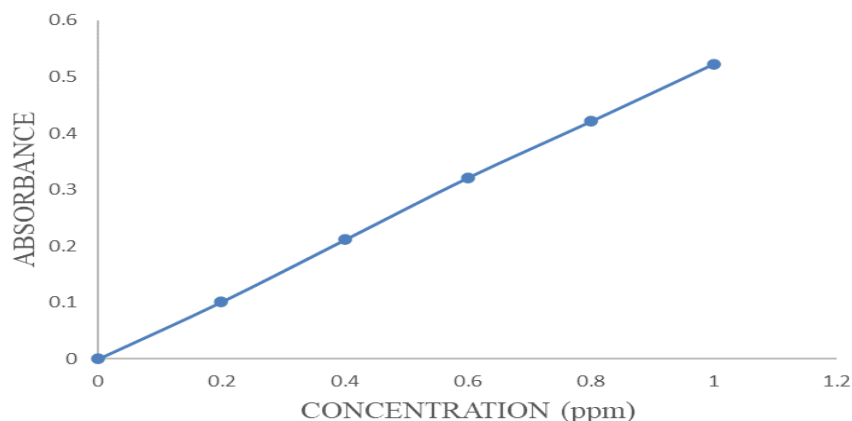


Figure 4.2: Calibration Curve for Fe (II) – 1.10-Phenantroline Complex.

The Calibration Curve from Figure 4.2 showed a linear relationship over concentration range of 0 -1.0 ppm and with a correlation factor of $r = 0.99986$, $R^2 = 0.99971$. absorbance, as described by the equation $A = 0.527C - 0.0008$, where 0.527 is the slope and -0.0008 is the intercept. The intercept was negligible, indicating minimal systematic error.

The concentration of the four unknowns was calculated using the formula from the calibration curve shown in the fig:4.2 $C = (A + 0.0008) / 0.527$ by accounting for the dilution factor. The findings achieved are as follows:

Sample A: $A = 0.32$, Concentration = 0.611 ppm

Sample B: $A = 0.30$, Concentration = 0.57 ppm

Sample C: $A = 0.24$, Concentration = 0.45 ppm

Sample D: $A = 0.24$, Concentration = 0.45 ppm

chemical properties of soil of similar lithology in three land uses types in J Ahiazu Mbaise, Imo State, Nigeria. *Journal of Agriculture and Crops* 1(3), 38 - 43.

- [6] Skoog, D. A., Holler, F. J., & Crouch, S. R. (2007). *Principles of instrumental analysis* (6th ed.). Thomson Brooks/Cole.

REFERENCE

- [1] AOAC (1995), *Official Methods of Analysis*. 16th Edition, Association of Official Analytical Chemists, Washington DC.
- [2] Gombe Geographic Information System, (2024). Plan No: CGS/70/2024
- [3] J. R. Boulding (1994), *Description and sampling of contaminated soils*. (2nd Edition), Lewis Publishers, New York, p. 230 – 240.
- [4] Rajbhandari, A. & Subedi, T. *Scientific World*, Vol. 11 No. 11, (2013).
- [5] S. U. Onwudike, E. E. Ihem, I. F. Irokwe and G. Onwuso (2015), *Variability in physico-*