

Novel and Easy Accessible Method for the Estimation of Copper Spectrophotometrically in Water and Soil Samples

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Abstract

A simple and sensitive spectrophotometric method has been established for the determination of copper in water and soil sample. The method simply involves the chelation of Dimethylglyoxime ligand with Copper under optimal conditions. The method is based on the formation of 1:2 complexes between Copper and Dimethylglyoxime. The optimal condition for the determination of copper was established. The Beer's law was applicable in the range 0.05 to 0.995 $\mu\text{g/ml}$. The molar extinction coefficient of the said complex was found to be $1.2 \times 10^5 \text{ mol}^{-1} \text{ cm}^{-1}$. The method was successfully applied for the determination of Copper in the water and soil samples.

Keywords: Copper; Dimethylglyoxime; Chelate complex; Spectrophotometry; Mole ratio method

Introduction

Copper is one of the most important metals in all the biological systems and occurs naturally in many vegetables, meat and grains. The increased concentration of copper in drinking water may cause increase in lead levels which in turn may be associated with liver damage and kidney diseases [1]. Copper kills a variety of potentially harmful pathogens. On 2008 the United States EPA registered 275 alloys having anti-microbial properties possessing more than 65% nominal copper content [2]. Copper being playing an important role in biological and environmental areas, an attempt has been made to study copper by spectrophotometry by using piperazine as a reagent. The method was also applied for copper alloys and results were co-related with the standard samples [3]. Cobalt, copper and iron are all trace essential elements for human bodies. Lack of these essential elements can induce some diseases while it is harmful and deleterious for overtaken [4]. Co, Cu and Zn from the essential group of metals required some metabolic activities in organisms. Toxicological effects of large amounts of Co include vasodilatation, flushing and cardiomyopathy in human and animals [5]. It has been reported that out of 110 known elements, 81 were present in living organism which were then biologically classified [6]. Medicinal uses, excess or deficiency of 81 elements [7] was reported to affect at least 235 diseases or functions of the body.

Estimation of copper

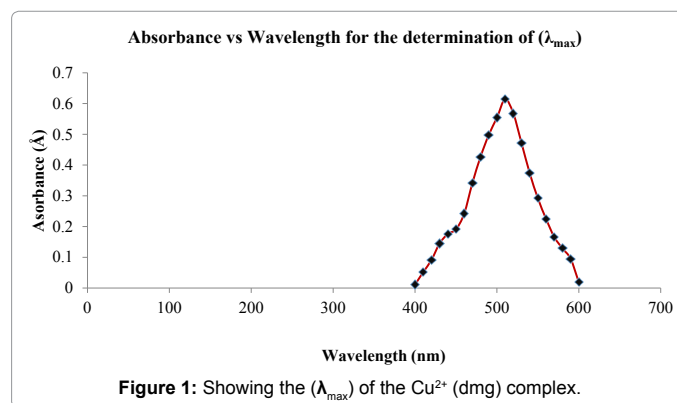
All reagents were purchased from local market. The stock and standard solutions were prepared freshly throughout the experiment. Under recommended conditions, Cu (II) metal was determined in various water samples following UV-VIS Spectrophotometer. An Elico pH meter was used for the pH adjustments.

Procedure

Copper sulphate solution: 0.01 g of copper sulphate solution was dissolved in 10 ml of deionized water.

Dimethylglyoxime solution: 1 g of Dimethylglyoxime was dissolved in 100 ml of ethanol.

Determination of (λ_{max}) of a copper dimethylglyoxime complex solution: The procedure described by Lee et al. [8] was modified and adopted for the determination of (λ_{max}), 5 ml of copper sulphate solution, 5 ml of Dimethylglyoxime, 2 ml of buffer and 8 ml of deionized water was taken in a test tube. The absorbance of the solution was taken in



the visible range from 400-800 nm at regular intervals of 10 nm. The graph was plotted between wavelength and the absorption, and a graph was obtained as shown in the Figure 1. From the line graph the (λ_{max}) for copper dimethylglyoxime was found to be 517 nm. Also, the color formation is instantaneous and the absorbance values remain constant up to 72 h. No significant change occurs when the order of addition of the reaction of the reactants is altered.

Effect of pH on the (λ_{max}) of a solution: Dissolve 0.25 g of copper sulphate (R) and 4.5 g of ammonium acetate R in dilute acetic acid R and dilute to 100.0 ml with the same solvent. Copper (II) solution was transferred into a series of test tubes marked 1 to 10. In each test tube, different concentration of copper sulphate solution was transferred in a ratio, 1 to 10 ml. to each test tube 5 ml of Dimethylglyoxime solution and 2 ml of buffer solution was added. The volume in each test tube was made up to 20 ml with deionized water. The test tube solutions were kept in warm water bath at a temperature range of about (30°C) for 10

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minutes. Finally, the absorbance was determined for each test solution by setting up of the instrument at 517 nm (λ_{\max}).

Effect of pH: The effect of pH on the formation of Cu (II)-dimethylglyoximine complex was studied to find out the optimum pH for Cu (II)-dimethylglyoximine complex stability. The studies were carried out keeping the 0.5 mL of copper (II) solution and 1.0 mL of dimethylglyoximine solution constant and varying the pH values of aqueous phases from 4.0 to 11.0 using suitable buffer solutions. The volume of each aqueous phase was adjusted to 10.0 mL with double distilled water. The plot between pH and the absorbance is shown in Figure 2. From the graph, it is observed that the development of the color of metal complex increases with increase of pH from 4.0 to 11. Below pH 4 and above pH 11, no any change in the color of the complex was noticed. But at pH 10.0 Cu (II)-dimethylglyoximine complex gives maximum absorbance values. Hence, pH 10.00 was chosen for further investigations for the determination of the stoichiometry of the complex.

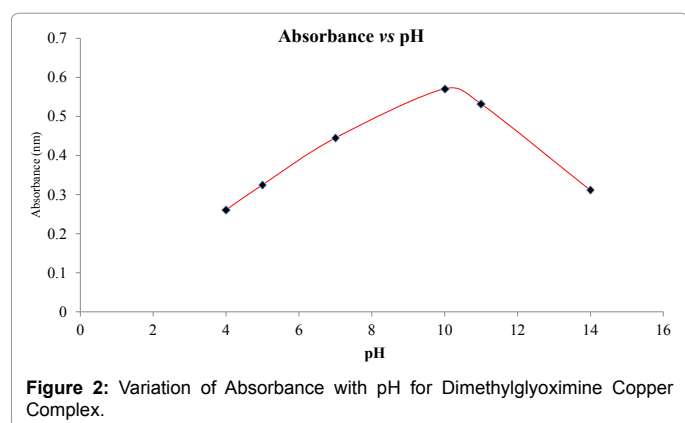


Figure 2: Variation of Absorbance with pH for Dimethylglyoximine Copper Complex.

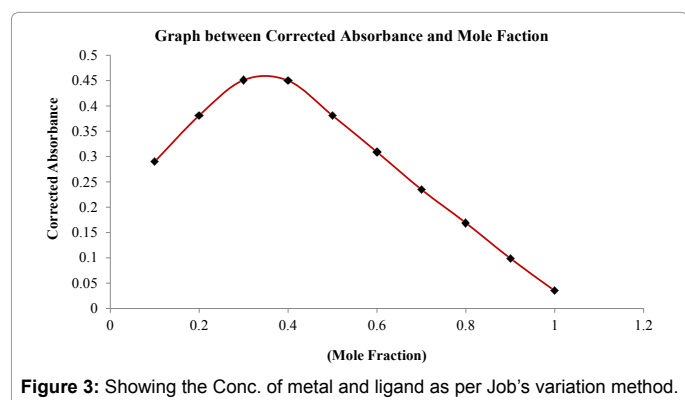


Figure 3: Showing the Conc. of metal and ligand as per Job's variation method.

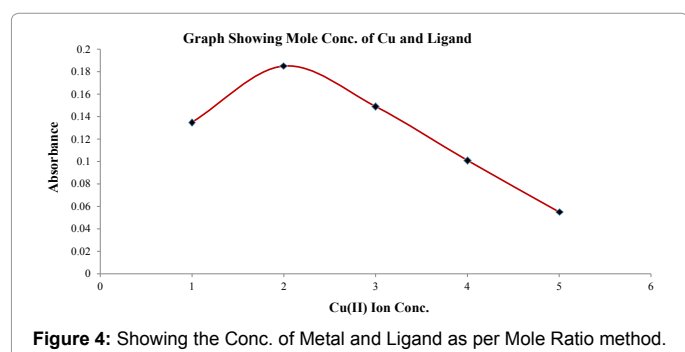


Figure 4: Showing the Conc. of Metal and Ligand as per Mole Ratio method.

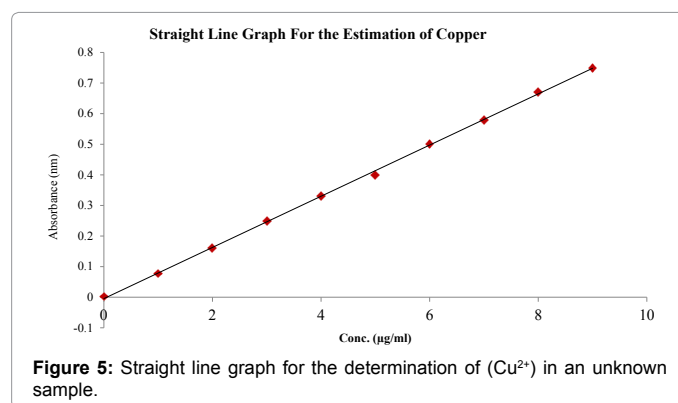


Figure 5: Straight line graph for the determination of (Cu²⁺) in an unknown sample.

Job's method of continuous variation

The stoichiometric ratio of dimethylglyoximine to Cu(II) in the complex was determined by Job's method [9]. A Series of solutions of varying mole fractions of Cu (II) and dimethylglyoximine were prepared keeping the total volume constant. In each case 1 ml of (3M) ammonium acetate was added and the final volume was made up to 10 ml with distilled water. The test tubes were shaken well and the absorbance values in each case were measured at 517 nm. A graph was plotted between corrected absorption and mole fraction of the ligand and from the graph it is observed that 2 moles of copper chelates with 4 moles of dimethylglyoximine indicating that the compositions of the complex as 1:2 in Cu-dimethylglyoximine complex (Figure 3).

Mole ratio method

The procedure described by Nardo and Dawson [10] was followed with little modifications was used for determining the stoichiometry of the complex spectrophotometrically, is especially applicable to determine stoichiometry of weak complex. To 1.0 ml of 0.05 M cupric solution, 1 ml of 3M ammonium acetate and required volume of dimethylglyoximine solution were added and shaken well. The absorbance of the complex was measured at 517 nm. The experiment was repeated with different volumes of the reagent ranging from 1 ml to 5 ml and the absorbance values were noted. A graph was plotted between volume of ligand and absorbance and it is observed that 1 mole of copper chelates with 2 moles of dimethylglyoximine (Figures 4 and 5).

Applicability of Beer's law

The set of test tubes were taken and into each test tube 5 ml of dimethylglyoximine, 2 ml of buffer solution and different concentrations of the copper solution are transferred. The final volume was made up to 10 ml with the deionized water. The solutions were shaken and kept at room temperature for 10 minutes and the absorbance was measured at 517 nm. A graph was plotted between copper concentration ($\mu\text{g/ml}$) and the absorbance. A straight-line graph was obtained, from which the concentration of unknown copper ion was determined.

Comparison of methods

The spectroscopic data of the present study and the already known method (by piperazine) for the estimation of copper were subjected to variance ratio test (F-test at 95% confidence level) to test the precisions of the results. The estimated copper in the water samples by the concerned method and the already known piperazine method are shown in Table 1. The experimental F-value in each case turned out far less than the F-distribution Table values suggesting that there was no significant difference in the precisions of the spectroscopic data, (Tables

S. No	Water samples	Piperazine method (µg/L)	Concerned method (µg/L)
1	Sample 1	0.15	0.16
2	Sample 2	1.5	1.4
3	Sample 3	1.2	1.21
4	Sample 4	0.93	0.85
5	Sample 5	0.85	0.88

Table 1: Data value for the determination of copper in various water samples.

Wavelength (Å)	Absorbance (nm)	Wavelength (Å)	Absorbance (nm)
400	0.011	510	0.614
410	0.052	520	0.568
420	0.091	530	0.472
430	0.145	540	0.374
440	0.176	550	0.292
450	0.191	560	0.225
460	0.242	570	0.166
470	0.341	580	0.129
480	0.427	590	0.095
490	0.498	600	0.019
500	0.554		

Table 2: Showing Absorbance of Copper dimethylglyoximine complex verses wavelength applied.

S. No.	Metal	Ligand	Mole Fraction	Corrected Absorbance
Set 1	1	9	0.1	0.29
Set 2	2	8	0.2	0.381
Set 3	3	7	0.3	0.451
Set 4	4	6	0.4	0.45
Set 5	5	5	0.5	0.381
Set 6	6	4	0.6	0.309
Set 7	7	3	0.7	0.235
Set 8	8	2	0.8	0.169
Set 9	9	1	0.9	0.099
Set 10	10	0	1.0	0.035

Table 3: Showing the metal and ligand concentration, Mole fraction and Corrected absorbance.

2-5). The T-test evaluation on the volumetric data gave the like leads on the precisions of the results due to Test-method and the Reference method.

Conclusion

The determination of copper ion was carried out by the using dimethylglyoximine as a coordinating ligand. The concerned method so for developed seems cost effective, simple and accurate. The method gives clear cut information about the composition of the complex so the concentration of copper ion.

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Mixture	A	B	C	D	E
Cu(II)	1	2	3	4	5
Dimethylglyoximine	5	4	3	2	1
Buffer solution	4	4	4	4	4
Absorbance	0.135	0.185	0.149	0.101	0.055

Table 4: Concentration of metal and ligand and their absorbance as per Mole Ratio Method.

S. No	Conc. of Copper (µg/ml)	Absorbance (nm)
1	0	0.002
2	1	0.077
3	2	0.16
4	3	0.25
5	4	0.33
6	5	0.4
7	6	0.5
8	7	0.58
9	8	0.67
10	9	0.75

Table 5: Concentration and the absorbance of Copper Solution for the determination of unknown copper in the water samples.

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