

Original Research Article

Spectrophotometric Determination of Zn(II) and Cu(II) in Analytical Samples Using Murexide Reagent

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ARTICLE INFO

Article history

Submitted: 2022-04-20

Revised: 2022-05-10

Accepted: 2022-06-03

Available online: 2022-06-03

Manuscript ID: PCBR-2204-1222

DOI: 10.22034/pcbr.2022.338475.1222

KEYWORDS

Spectrophotometric methods

Murexide

Zinc

Copper

Stability constant

ABSTRACT

The current research aims to develop a new spectrophotometric method for the determination of zinc(II) and copper(II) using a colorimetric reagent, as murexide ion. The complexation of Zn(II) and Cu(II) with murexide has been studied spectrophotometrically at absorption maxima of 450 and 470 nm for Zn-murexide and Cu-murexide, respectively. The murexide reagent interacts with Zn(II) and Cu(II) instantaneously at pH 7 and pH 5.5, respectively, and the absorbance of the solution is stable for 220 and 120 minutes, respectively. Using Job's continuous variation method, the stoichiometry of the complexes was found to be 1:2 metal to ligand ratio for Zn and Cu, respectively. The continuous variation approach was used to estimate the stability constants (K_{stab}) values, which were found to be in the order 1.35×10^{16} and 2.30×10^7 for Zn and Cu complexes, respectively. The proposed spectrophotometric methodology established that zinc(II) and copper(II) could be estimated at levels of 0.2-2.0 and 0.5-5.0 ppm, which has molar absorptivity values of 1.95×10^4 and 6.55×10^3 $\text{l.mol}^{-1}.\text{cm}^{-1}$, respectively. Likewise, the formed complexes were stable at different pH values, allowing the simultaneous estimation of the two metals.

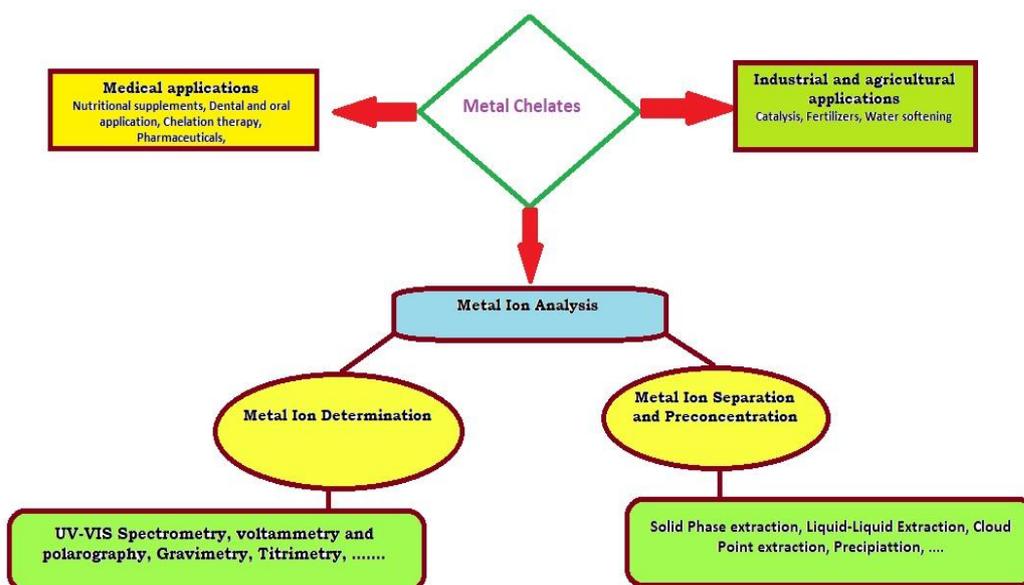
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GRAPHICAL ABSTRACT


1- Introduction

Contamination of living organisms and ecosystems with heavy metal ions is a major public health hazard around the world [1]. Heavy metal ions cause a variety of diseases in biological systems, including Minamata, itai-itai, arsenicosis, mental retardation, and Wilson's disorders [2,3]. Heavy metal ions detection and removal has become one of the more difficult tasks in recent years [4–7]. To determine the heavy metal content in their compounds, a range of methodologies can be adopted. Atomic absorption spectrometry [8-12], inductive coupled plasma-emission spectrometry [13,14], potentiometry [15], derivative potentiometric stripping analysis [16], flow injection catalytic photometric method [17,18], and inductive coupled plasma-mass spectrometry [19] are some of these techniques. Several researches on spectral estimation of heavy metals have been published.

A number of methods for determining zinc and copper have been developed [18,20-24], with many of them based on the synthesis of coordination compounds with attractive optical characteristics. Transition metal complexes are

of particular importance in industry, medicine, and biology, with several applications in thermochromism, antioxidation, vulcanization acceleration, painting protection, catalysis, and anti-bacteriology [25]. Murexide, a reddish purple ligand, has attracted the interest of researchers due to its use in chemical analysis and spectrophotometric disciplines [25,26]. Some metals are determined using it as a chromogenic reagent in standard spectrophotometric methods [27,28]. In the literature, this indicator has a limited potential, with only a few studies looking at surface features [22], stability constants of its complexes [28,29], and complex formation kinetics [30,31]. The main objective of this work is to prepare a metal-murexide complex that will be used in a new method that is more sensitive, easy, and low cost for determining zinc(II) and copper(II) ions from environmental samples, as well as to study all the conditions that affect the determination process, such as the pH of the reaction solution, the time required to complete the complex formation reaction, and the effect of some interference ions that will affect the complexation reaction.

2- Experimental

2-1- Chemical reagents

All of the chemical reagents utilized in this study are of high purity and are supplied either by companies B.D.H, Fluka, and Merck without any kind of purification processes, as well. Murexide indicator, hydrochloric acid, sodium hydroxide, copper chloride, zinc chloride, potassium chloride, potassium nitrate, and potassium sulphate were used throughout this work.

Murexide (5.0×10^{-3} mol. L⁻¹) and metal chloride (5×10^{-3} mol. L⁻¹) stock solutions were made by dissolving the appropriate weighted quantities of pure solid materials in double distilled water and diluting to the mark in 25.0 mL volumetric flasks.

2-2- Instrumentation

Jenway Spectrophotometer model 6305 was used to record the electronic absorption spectra, which ranged from 400 to 650 nm. For pH measurements, a Jenway model 3505 pH meter was utilized as well as a composite glass electrode were utilized.

2-3- Analysis

2-3-1- The spectrum of murexide

In a 10 mL volumetric flask, 100 μ L standard stock solutions of murexide (5×10^{-3} M) were diluted with double distilled water. The solution's spectrum was then measured between 400 and 650 nm, and the λ_{\max} of murexide solution was identified.

2-3-2- The spectra of complexes

100 μ L aliquots of metal ion solution [5×10^{-3} M] were mixed with 100 μ L aliquots of murexide solution [5×10^{-3} M]. The resultant solution was transferred to 10 mL volumetric flask and the volume was filled to the mark with double distilled water. The prepared solutions' spectra were measured between 400 and 650 nm, and the λ_{\max} of the two complex solutions was determined.

2-4- Study of pH effect

Mixing 250 μ L aliquots of metal ion stock solutions with 250 μ L murexide stock solution produced a series of solutions. After adjusting the pH to 2.0-9.0, the solutions were transferred to 25 mL volumetric flasks and the volume was made up to the mark with double distilled water. The absorbance of these solutions was measured at 450 nm for the Zn complex and 470 nm for the Cu complex, with the reagent solution behaving as a blank.

2-5- Study the effect of time

100 μ L aliquots of metal ion stock solutions and 100 μ L aliquots of murexide stock solutions were mixed together. The resulting solutions were transferred to 10 mL volumetric flasks and filled with double distilled water to the desired volume. The absorbance of the prepared solutions was measured every 5 minutes for up to 200 minutes at 450 nm (for Zn complex) and 470 nm (for Cu complex).

2-6- Stoichiometry and formation constant determination

The stoichiometric ratio of M(II) to ligand in the complexes were evaluated by Job's continuous variation method as reported by Adedibu et al. [31] and Elsherif et al. [32,33]. The series of volumes (100, 200, 300, 400, 500, 600, 700, 800, and 900 μ L) of the 0.005 M of M(II) solutions were respectively pipetted into nine 25 mL volumetric flasks and aliquot (900, 800, 700, 600, 500, 400, 300, 200, and 100 μ L) of the 0.005 M ligand were added; keeping the mole fraction of the solution constant, and then the flasks were filled to the mark with double distilled water after adjusting the pH to the optimum value (which previously determined). The absorbance of each of the solution was measured at the wavelength of maximum absorbance of the complex which was previously determined.

2-7- The effect of interferences

Experiments were conducted to test the interference of three important anions: chlorides, nitrates, and sulphates. Several synthetic solutions with known concentrations of metal ions (1.0×10^{-4} M), murexide (1.0×10^{-4} M), and interference anions (0.01, 0.02, 0.05, 0.10, 0.20, 0.50 M) were prepared. The proposed method was used to determine the metal ion content of each mixture. The recoveries were estimated for each solution.

3- Results and Discussion

3-1- Absorption spectra of murexide and complexes

Figure 1 displays the spectra of murexide and its complexes with Zn^{2+} and Cu^{2+} ions in aqueous solution. The electronic spectra of murexide clearly indicate one primary band of absorption at 520 nm, attributed to π - π^* and n - π^* transitions [30,34]. As can be observed, the resultant complexes in both cases are differentiated by a relatively strong shift towards shorter wavelengths (450 and 470 nm), as compared with free ligand (520 nm). In general, the electrostatic interaction of a bonded metal ion would not have such a strong effect on the electronic structure of a dye molecule, and hence on its spectrum. The twisting of the two rings of the murexide molecule (Figure 2) relative to each other (around the N-bridge axis) caused by metal ion complexation is thought to be responsible for such spectral behaviour [35].

3.2- Optimization of reaction conditions

The pH effect

For metal complex solutions, pH levels in the range of 2-9 were shown to be appropriate. HCl and NaOH solutions were used to select the ideal pH value of metal-complex solutions. As demonstrated in Figure 3, the best pH levels for murexide- $Zn(II)$ and murexide- $Cu(II)$ complex formation were $pH=7$ and $pH=5.5$, respectively, according to the pH against absorbance graph (Figure 3).

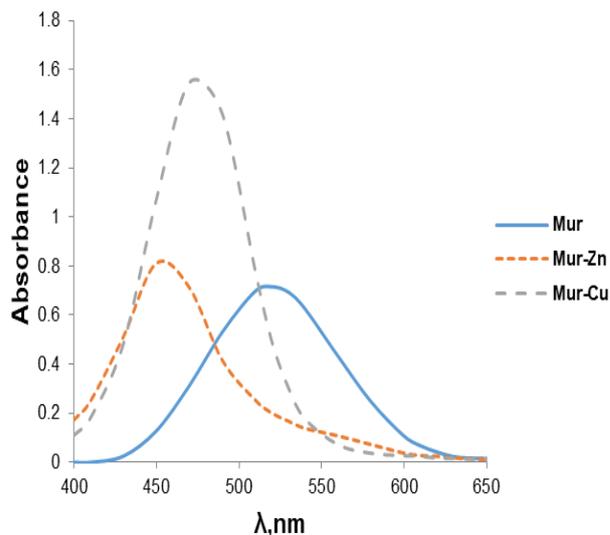


Fig. 1. The Spectra of murexide and its complexes with $Zn(II)$ and $Cu(II)$

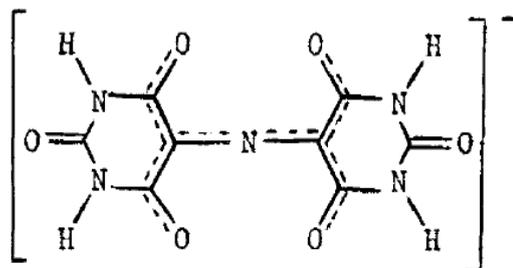


Fig. 2. Murexide Structure

When the pH was raised above 7 and 5.5, the possibility of the occurrence of zinc hydroxide and copper hydroxide rather than metal complexes increased [35]. When the pH decreased less than 3, the murexide ligand protonated and made the complex formation impossible [8].

3.3- Effect of time

Figure 4 indicates that the developed zinc complex had a high absorbance after 60 minutes of reaction time and remained steady for 220 minutes at ideal circumstances, however the formed copper complex had a high absorbance initially, remained constant for 120 minutes, and then decreases with time.

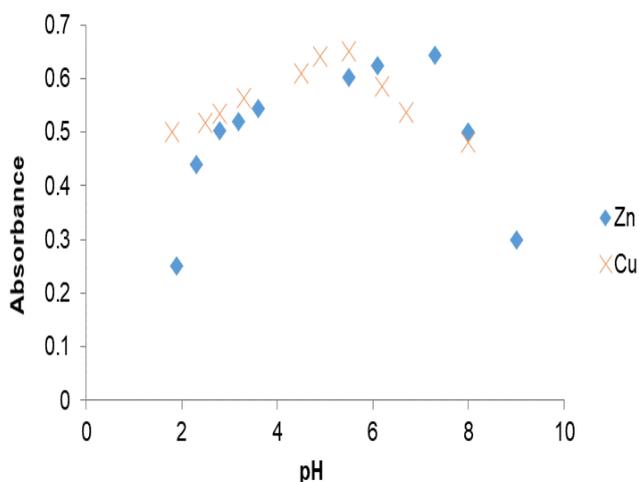


Fig. 3. Effect of pH on the complexes formation

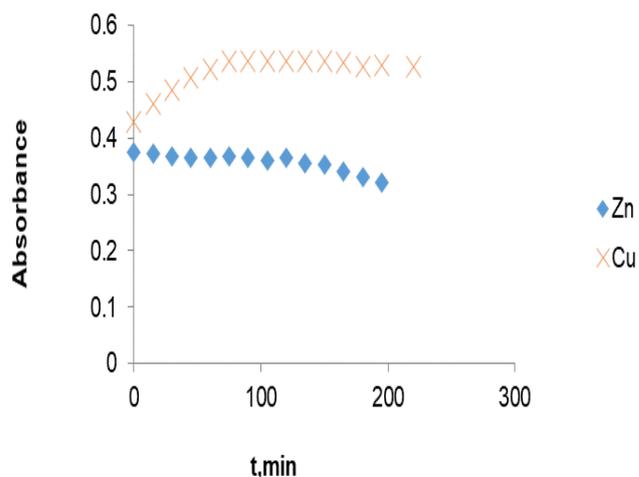


Fig. 4. Effect of time on the complexes formation

The results of this investigation demonstrated that the murexide ligand can be used to estimate zinc and copper concentrations by forming a stable complex, comparable to the previous reagents which have been used to estimate the concentration of other ions spectrally [8,23,36].

3.4- Effect of interferences

The effect of several interferences on the absorption of zinc and copper complexes with murexide was investigated by using a variety of negative ions, as illustrated in Figure 5. The

negative ions were revealed to either decrease or increase absorption. This is due to competition between these ions and metal ions on the bonding with murexide to form the complex, where part of the murexide is taken to be a new complex [8]. The low absorbance values resulted from their action as a masking reagent to remove the effect of the interference by returning the absorption values to the original calculated output without interference [37].

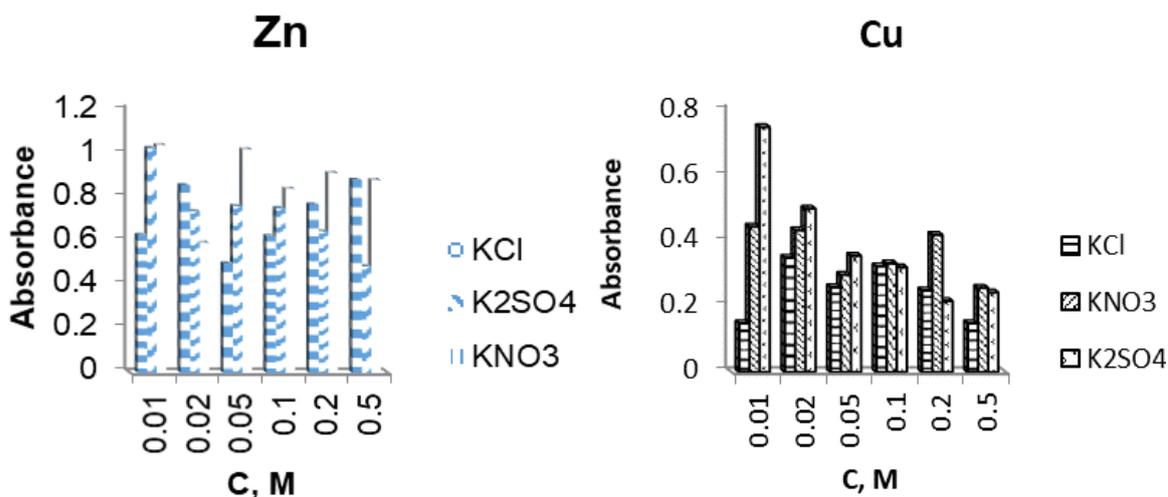


Fig. 5. Effect of interferences on the complexes formation

3.5-Composition and stability constant for the complex

The method of continuous variations was used to determine the stoichiometry of copper and zinc complexes with murexide in an aqueous solution (Figure 6). The metal ion to murexide mole ratio was found to be 1:1 in the case of the copper complex studied. It has already been observed that various metal ion-murexide complexes in aqueous solutions have a 1:1 stoichiometry [37,38]. In the case of zinc complex, however, the ratio was determined to be (1:2), i.e. one mole of metal to two moles of ligand. This observation is similar with that of Sethi et al. [39].

The values of the stability constants of the complexes were computed using the Job method, and the results are reported in Table 1. The values were found to change in the order of 1.35×10^{16} and 2.30×10^7 for Zn-murexide > Cu-murexide, confirming the complexes' high stability. The stability constants revealed that the zinc complex is more stable. The reason for this is that the higher the atomic number, the higher the nuclear charge, causing it to have more ability to attract ligands compared to the other ions in the same period of the periodic table, resulting in more electric field absorbed by the positive ions of the ligand, increasing the complex's stability [40].

Table 1. Various zinc and copper parameters

Ion	λ_{\max} (nm)	pH	M:L	ϵ (l.mol ⁻¹ .cm ⁻¹)	Linear range (ppm)	Sensitivity (ppm ⁻¹)	K_{stab}	R ²
Zn	450	7.0	1:2	1.95×10^4	0.2-2.0	0.2982	1.35×10^{16}	0.9994
Cu	470	5.5	1:1	6.55×10^3	0.5-5.0	0.1017	2.30×10^7	0.9999

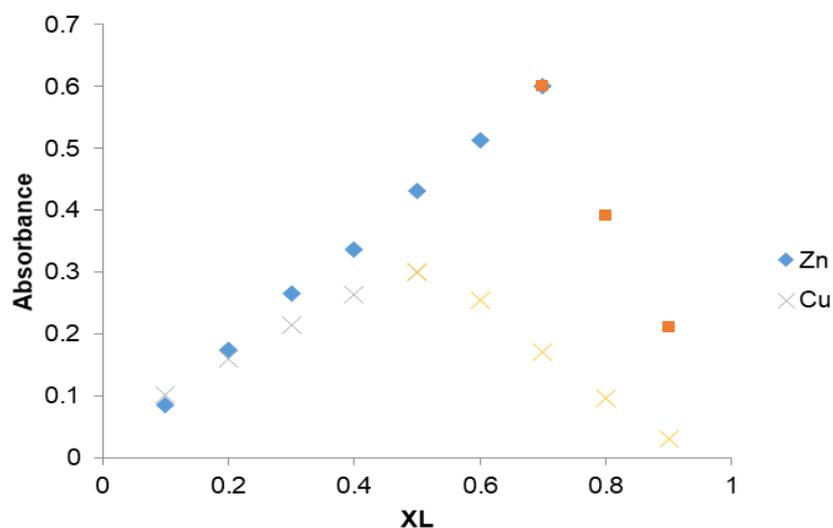


Fig. 6. Continuous variation plots for Zn²⁺ and Cu²⁺ complexes with murexide

3.6-Beer's law and sensitivity

Calibration curves (Figure 7) were developed at 470 and 450 nm for Zn and Cu complexes, respectively, against a reagent blank under the optimum conditions outlined above. Beer's law holds true for Zn concentrations ranging from 0.2

to 2.0 ppm and Cu concentrations ranging from 0.5 to 5.0 ppm. According to Sandell's definition, the molar absorptivity and sensitivities are as follows: Zn: 1.95×10^4 l.mol⁻¹.cm⁻¹, 0.2982; Cu: 6.55×10^3 l.mol⁻¹.cm⁻¹, 0.1017.

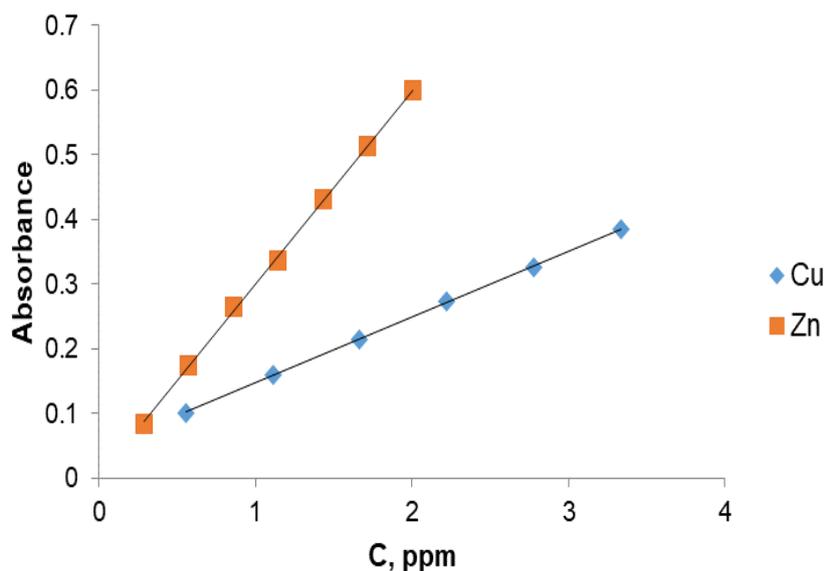


Fig. 7. Calibration for Zn²⁺ and Cu²⁺ complexes with murexide.

4-Conclusion

The complexes formed by murexide reagent and Zn(II) and Cu(II) were studied spectrophotometrically. The proposed approach demonstrated the maximum absorbance at 450 and 470 nm for Zn-murexide and Cu-murexide complexes when using a UV-VIS spectrophotometer, and optimum conditions at pH 7, pH 5.5, and 0.2-2.0, 0.5-5.0 ppm, respectively. In comparison to the conventional spectrophotometric approaches which involve the direct interaction between zinc and copper ions solution and the reagent solution used for quantification, this new analytical method was relatively simple, quick, and sensitive. Aside from the low limit of zinc detection, the analysis of a wide range of concentrations follows Lambert-Beer's Law.

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HOW TO CITE THIS ARTICLE

Khaled Muftah Elsherif*, Qamar Hadidan, khawla Alkariwi. Spectrophotometric Determination of Zn(II) and Cu(II) in Analytical Sample using Murexide Reagent, *Prog. Chem. Biochem. Res.* 5(3) (2022) 229-238.

DOI:10.22034/pcbr.2022.338475.1222

URL: http://www.pcbiochemres.com/article_152663.html

