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Development and Validation of Solvent Extraction Method for Spectrophotometric Determination Cu(II) by Using $SALENH_2$ Ligand

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ABSTRACT

In present investigation we demonstrated first time the green chemistry route of synthesis of N,N'-bis (salicylaldehyde) ethylenediimine ligand (SALENH2). The ligand has four coordinate sites and form stable complex with Cu(II) ion at pH 8 to 11. The complex is yellow coloured and insoluble in water but highly soluble in $CHCl_3$. The complex was found to stable in $CHCl_3$ for long time and is violet coloured. The absorbance spectra of complex show broad peak in visible region with maximum absorbance at 558 nm. The calibration curve method was developed for selective analysis of Cu(II) and was validated in context with AAS method. The sample analysis showed that developed method by is highly efficient and selective for the analysis of Cu(II) in presence of other metal ions.

Keywords: Solvent extraction, Cu- SALEN, analytical method development, spectrophotometry

I. INTRODUCTION

Solvent extraction is the method of quantitative separation of substances usually from aqueous phase to organic phase (extracting solvent) which is immiscible with water. The extracting solvent is so selected that compound is highly soluble in it and can be easily separable from aqueous phase [1]. Solvent extraction methods are applicable for selective separation as we as quantitative analysis of substances. The technique is well demonstrated for selective separation of metal ions from aqueous phase in to organic phase and for quantitative analysis by spectrophotometry.[2] In the technique, the importance is choice of suitable ligand which form stable complex with metal. Furthermore complex should be highly soluble in organic phase. Transition metal complexes are usually coloured hence after extraction they can be easily quantitatively analysed by spectrophotometry. For selective determination of metal ion of interest from mixture of metal ions, suitable complexing agent must be selected which show distinct colour with analyte metal ion from other metal ions in the sample. Such method spectrophotometric method possesses high importance as sensitivity of these methods is high and low cost of equipment.[3] Thus, herein we demonstrate the synthesis of salicylaldehyde ethylenediamine Schiff's base ($SALENH_2$) ligand by green chemistry route and its application for the solvent extraction and quantitative analysis of Cu(II) metal ion from micronutrient sample.

II. METHODS AND MATERIAL

a. Synthesis of Ligand:

Ligand was synthesized by green chemistry route under ambient condition. To 5 ml pure salisylaldehyde liquid 2 ml ethylenediamine was added drop wise with constant stirring. This resulted into formation dark yellow coloured crystalline

product. The product is the ligand of interest i.e. $SALENH_2$. Ligand is purified by crystallization using methanol as a solvent. TLC was recorded in pure methyl acetate. Melting point was recorded by usual method.

b. Analytical Method Development for Cu(II) analysis by using $SalenH_2$ ligand:

In first step of analytical method development standard Cu(II) 0.01M concentration was used. In first step effect of pH was studied on percent extraction.[5] Into 2 mL Cu(II) solution 2 mL ligand solution and 10 ml water was added and pH was adjusted to 5. Similarly six more solutions were prepared of pH was adjusted to 6, 7, 8, 9, 10 and 11. Each of this solution was extracted with 5+2 ml chloroform (two times) and absorbance spectrum was recorded in visible region. From observed absorbance optimum pH for extraction was decided.

c. Quantitative analysis:

Standard stock solution (0.01M) of Cu(II) was prepared from A.R. Grade $CuSO_4 \cdot 5H_2O$ was prepared in water and used in further studies for the preparation working standards of Cu(II). Linearity range and detection limit was obtained by reported method. Afterword, the known solution of Cu(II) was analysed by calibration curve method so as to validate the method. Finally copper alloy sample was analysed by validated spectrophotometric method and results were confirmed by reported atomic absorption spectroscopic (AAS) method.

III. RESULTS AND DISCUSSION

a. Synthesis and purity of Ligand:

Synthesised by ligand was purified by crystallisation method using in 1:1 water methnol mixture. M.P was recorded which is found to be 123°C which matches with reported value (125° C). Further purity was checked by TLC (Fig.1). The synthesised product shows Rf value is 0.53 which is different from starting material (solvent system) methyl acetate using silica gel as stationary phase and N-hexane + Methyl acetate as mobile phase.

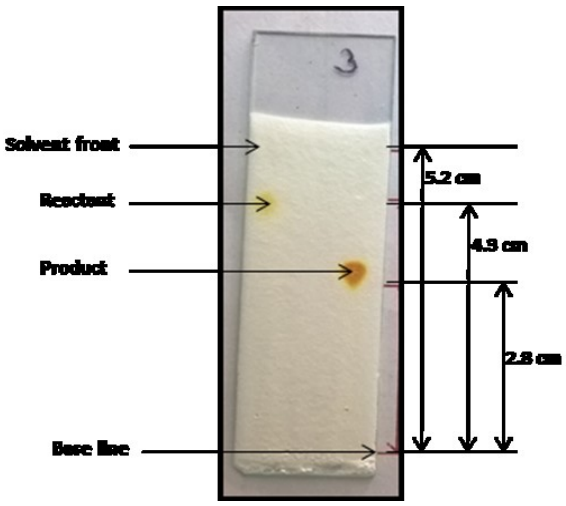

Figure 1. TLC of ligand and starting material

The purity of product was quantitatively checked by non aqueous titration method. 100 mg of synthesised ligand was dissolved in 10 ml Glacial acetic acid and titraed against 0.1M $HClO_4$ using crystal violet as an indicater. The observed purity of ligand was found to be 98.72% for which structure is represented in fig.2.[5]

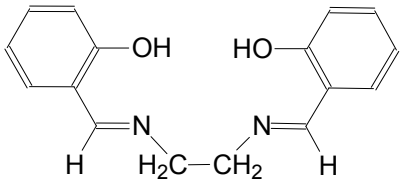

Figure 2. Structure of ligand

b. Effect of pH on Extraction of Cu(II):

In 1st step we have studied the effective pH on complexation of ligand with Cu(II).[6] The complaxation was performed at the different pH (5, 6, 7, 8, 9, 10,11). The complex was extracted into chloroform ($CHCl_3$) and absorbance was recorded 560 nm. 560 nm is a wavelength at which Cu-$SALENH_2$ ligand show maximum absorbance (Fig.3). The result of effect of pH on extraction of complex represented in Fig-4.

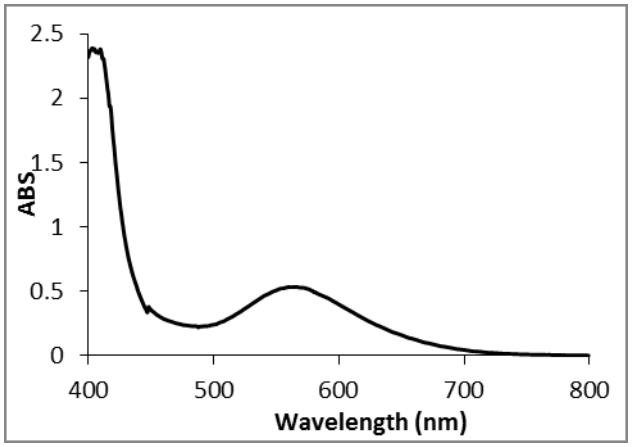

Figure 3. Absorbance spectra of Cu-SALEN Complex

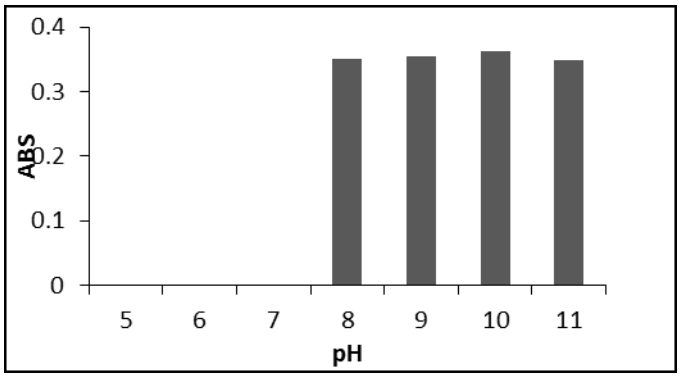

Figure 4. Effect of pH on extraction of Cu-SALEN complex

The results clearly indicate that alkaline pH is good for extraction of this complex into $CHCl_3$ and pH 10 is best for the quantitative extraction of the complex.

c. Linearity Rang:

Beers law (A= εbC) is obeyed over the limited range of concentration of analyte. In most of the cases good relation in concentration and absorbance is observed over the range of absorbance from 0.1 to 0.9.[7] Thus we have selected the concentration range of Cu (II) which gives the absorbance near to this range. For Cu (II) concentration it was found to be 0.0002 to 0.001 M. Over this concentration of Cu (II) absorbance was found in the range of 0.139 to 0.650 (fig. 5). The linearity in absorbance is indicated by R^2 value which is 0.998 i.e. well above the 99.8% confidence level as required for quantitative analysis, theoretically.

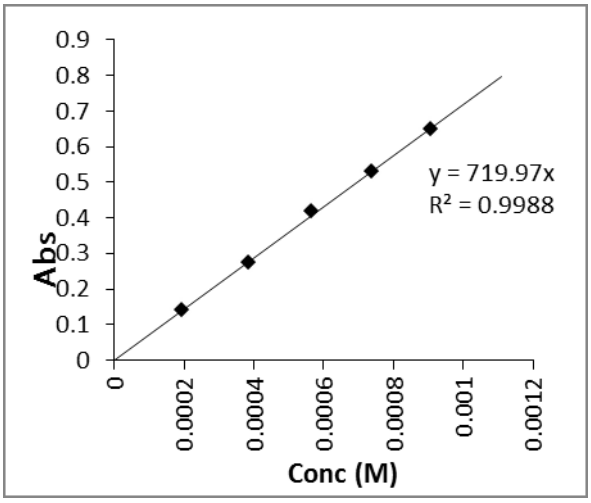

Figure 5. Determination of Linearity range

d. Method validation by calibration curve method:

For method validation we have used calibration curve method.[8] Calibration curve was set by using 0.2 ml to 0.1 ml Cu(II) and extracted with 5+2 ml $CHCl_3$. Sample of known concentration was used. From stock sample 0.3 and 0.5 mL solution i.e. 0.000291M and 0.000476 M Cu(II) was used for extraction. By using calibration curve method conc. of Cu(II) in sample were calculated. The observed amount was found to be 0.000287 and 0.000466 M. These values lie in the range of 95 to 105% of expected value i.e. in the 98% confidence limit. This indicate that the developed method of Cu(II) analysis by using $SALENH_2$ ligand can be used for the quantitative analysis of Cu(II).[9]

e. Analysis of sample and its confirmation by AAS:

We have analysed the sample of plant micronutrient supplement for the Cu(II) content by developed method. 1 g Micronutrient powder sample was dissolved in 250 ml water and 0.5ml H_2SO_4 solution was prepared. From this solution 5 ml was used for analysis. Calibration curve method was used. Calibration curve was set by using 0.2 ml to 1.0 ml of Cu(II) of 0.01M. Absorbance of extracted solution was recorded at 560 nm. The observed content in micronutrient sample was 3.63±0.08% by developed method. The same sample was analysed by known AAS method. By AAS percentage of Cu(II) in sample was found to be 3.64±0.06%. The result of analysis of

developed method is very close to AAS method which is well known and precise method of analysis of trace metal in presence of other metals.

IV. CONCLUSION

The ligand can be synthesized by very simple and rapid method by mixing molar proportions of salicyladehyde and ethylendiamine which is environment friendly method with high yield of product. Solvent extraction method for selective analysis of Cu(II) was developed and validated by using $SALENH_2$ ligand of which validation was done by comparing results by AAS method. The results of analysis shows that developed method is highly efficient for the analysis of Cu(II) in presence of other metal ions.

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