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# Electrochemical Analysis of Cu (II) Complex Derived from Sulpha Drugs-Based Azomethine Ligand: Cyclic Voltammetry and Impact of Concentration and Scan Rate

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## Abstract

*A Cu (II) azomethine complex was synthesized using 2-hydroxy-2-methylpropiophenonesulphamethizole (HMPSMO) ligand and a copper salt. The ligand and its corresponding metal complex were characterized through elemental analysis and infrared (IR) spectroscopy. The electrochemical properties of the Cu (II) complex were examined via cyclic voltammetry using a glassy carbon electrode in a dimethylformamide (DMF) solution with 0.1 M NaClO4 as the supporting electrolyte. Cyclic voltammograms revealed metal-centered electroactivity within the potential range of -1.2 to +0.7 V. The electrochemical studies indicated a one-electron quasi-reversible reduction wave in the cathodic region for the Cu (II) mononuclear complex. Cathodic peak current intensities increased with scan rate, consistent with the Randles-Sevcik equation. A linear relationship between peak current and the square root of the scan rate confirmed diffusion-controlled electrochemical processes. The influences of concentration and scan rate on the cyclic voltammograms were also evaluated.*

**Keywords:** *2-Hydroxy-2-methyl Propiophenone sulphamethizole, Cyclic voltammetry, Glassy carbon electrode, Quasi-reversible.*

## 1. INTRODUCTION

Schiff bases or azomethines are well-known in coordination chemistry for their distinct characteristics, which are attributed to the chelating activities of the imine group -C=N. As a result of their chelation activity, the ligands and their metal complexes have diverse biological properties [1-4]. Schiff base chelates exhibit extraordinary features, including optical nonlinearity [5], photochromic behavior [6], catalytic activity [7], and high stability. Furthermore, by shielding metal surfaces from corrosive chemicals [8], these solid chelates operate as good inhibitors and antioxidants, absorbing reactive oxygen species. These favoured ligands were explored due to their versatility, selectivity, and sensitivity to the core metal atom [9-11], which endows them with a diverse role in research and technology. In Schiff base ligand, the azomethine (imine) group (-N=CH-) and other donor atoms, such as oxygen, nitrogen, sulfur, etc., help them chelate with metal ions [12-14]. Because of their chelating ability, they have a variety of biological effects on microorganisms [15-18].Electrochemical techniques are powerful tools for studying the properties and behaviour of molecules at the electrode-solution interface. Among these techniques, cyclic voltammetry (CV) is one of the most widely used and versatile methods [19]. Cyclic voltammetry is an electrochemical technique that measures the current response of a system as a function of the applied potential. In CV, the potential is swept linearly with time, first in the forward direction and then in the reverse direction, forming a cycle. This technique provides valuable information about the redox properties, electron transfer kinetics, and chemical reactivity of molecules [20-22].CV is used to develop electrochemical sensors, Material science, biological systems and pharmaceutical analysis [23].

Many researchers have focused their attention on the electrochemical behaviour of Schiff base metal complexes. However, there is limited information in the literature about the electrochemical behaviour of sulfa drugs' Schiff bases and their transition metal complexes. The current study focused on the electrochemical behaviour of the Cu (II) complex containing the HMPSMO ligand in order to evaluate several essential parameters and the influence of concentration and scan rate on cyclic voltammograms.

## 2. EXPERIMENTAL METHODS

### 2.1. Chemicals

All chemicals were analytical grade and of the highest available purity, and were used without further purification. For the preparation of Cu (II) complex $(CH_3COO)_2Cu.H_2O$, and Methanol were obtained from Sigma-Aldrich. For the preparation of the stock solution for cyclic voltammetric studies, DMF, $NaClO_4$, were also obtained from Sigma-Aldrich. Double-distilled water (DDW) and purified solvent were used for cyclic voltammetric studies.

### 2.2. Equipment and Voltammetric Parameters

Cyclic voltammetric measurements were performed using a computerised constant current source (Cyclic Voltammetry System) provided by Retrace Electronics, which is fully digitally controlled. By the use of the instrument's programming capabilities, the whole test set was automated and managed. Toper form cyclic voltammetry, a specially created electrochemical cell was utilized. Three-component cells made of glassy carbon (GC) as the working electrode, a reference electrode Ag/AgCl (saturated KCl) and a Pt wire as an auxiliary electrode were used for electrochemical research. The digital pH meter (Elico) was used to measure the pH of solutions. The infra-red spectra of synthesized compounds were recorded on KBr pellets in the range 4000-400 $cm^{-1}$ on an FT-IR spectrometer (Schimadzu).There are some kinds of significant variables that characterize the CV. The diagnostics invented by Nicholson and Shain for examining the electrochemical response are centered on four of these observables, especially the two peak currents (Epc, Epa) and the two peak potentials (Ipc, Ipa).

### 2.3. Sample Preparation and Procedure

The complex stock solution has been prepared in DMF. For this purpose, an appropriate amount of complex was dissolved in DMF to yield a 0.01M solution. 1.0 M $NaClO_4$solution is employed as a supporting electrolyte; 1 ml of stock solution (.01M) and 1ml of supporting electrolyte were taken in an electrochemical cell, and this solution was made up to 10 ml with DMF.The experimental solution was then placed in an electrochemical cell with three electrodes and deoxygenated with dry nitrogen gas for 15 minutes before being used to record potential current curves and thereafter, used for recording CV curves.

### 2.4. Synthesis of Cu (II) complex of HMPSMO

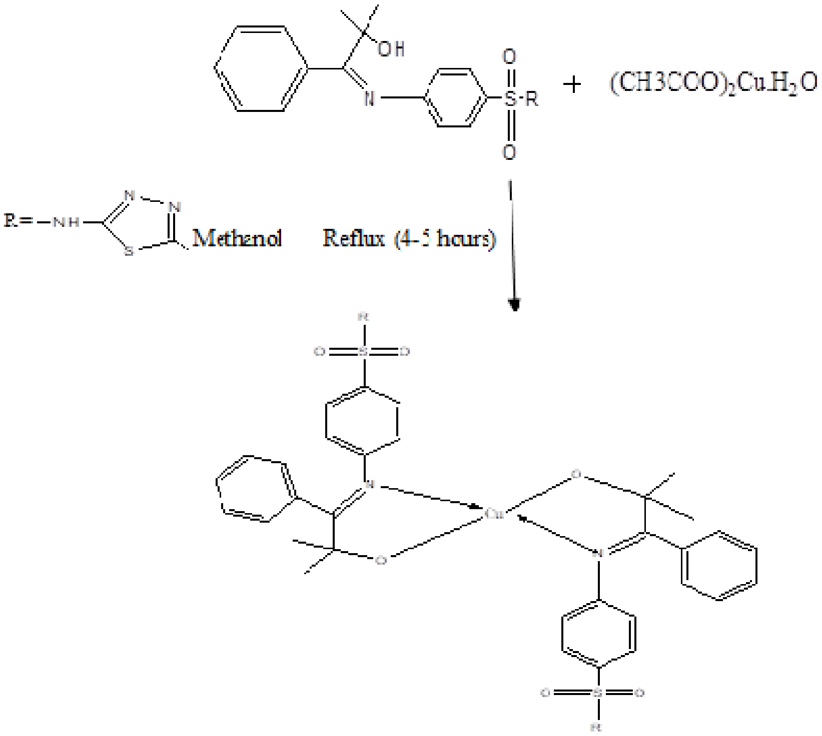

**Figure 1.** *Synthesis reaction of Cu (II) complex of Schiff base ligand*

The required ligand was prepared by condensation reaction of2-Hydroxy-2-methyl Propiophenone and sulphamethizole, signified as HMPSMO (mentioned previous research article)[24-25].The Copper (II) complex with prepared ligand was synthesized by adding a methanolic solution of $(CH_3COO)_2Cu.H_2O$ with vigorous stirring to prepared ligand methanolic solution in ratio of 1:2. The obtained mixture was heated under reflux with stirring for 4-5 hours at 60$^{O}$C and then allowed to evaporate into its one third volumes and cooled [26-27]. The obtained deep green colored complexwas separated and washed with methanol and dried in a vacuum over anhydrous calcium chloride .The formation reaction and structure of the prepared complex are shown in Figure 1.

## 3. RESULTS AND DISCUSSION

The structure of the novel Cu (II) complex was elucidated by several analytical and physical techniques. The obtained analytical and physical results were in good agreement with the suggested composition of the complex. The synthesised metal (II) complex is colored, stable in air and insoluble in water, but completely soluble in DMSO and DMF solvents.

**Table 1.** *The physico-chemical data of the synthesized complex*

| S. No. | Composition of Cu(II) complex | Colour & state | Yield (%) | M. Wt. Found (Cal.) | Elemental Analysis (%): found (Cal.) | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | C Found (Cal.) | H Found (Cal.) | N Found (Cal.) | Cu Found (Cal.) |
| 1 | **$C_{38}H_{38}CuN_8S_4O_6$ (HMPSMO-Cu)** | Dark green solid | 81 | 894.51 (894.56) | 50.97 (51.02) | 4.20 (4.28) | 12.49 (12.53) | 6.98 (7.1) |

### 3.1. FT-IR spectral Studies

The infra-red spectra of Cu (II) complex of HMPSMO were recorded on KBr pellets in the range 4000-450 cm$^{-1}$ on an FT-IR spectrometer (Schimadzu). The binding site of the ligand in metal (II) complexes can be determined a lot by comparing the infrared spectra of the free ligand and its metal (II) complexes. Table2 lists the selected IR vibrations and approximate assignments for the metal (II) complex [28-29]. The frequency at 1630 cm$^{-1}$ for (-CR=N) bond decreased by 13-20 cm$^{-1}$ for Cu (II) metal complex, indicating coordination of the nitrogen atom of the ligand to Cu (II) ion. The broad and strong band due to υ (OH) 3410 cm$^{-1}$ in ligand spectra disappear in the complex formation, showing oxygen coordination to the metal ion by deprotonation. The appearance of new bands in regions 450-465 cm$^{-1}$ and 560-570 cm$^{-1,}$ assignable to υM-N and υM-O, respectively attributed to the bonding of Cu(II) ions to oxygen and nitrogen atoms in Cu(II).Water vibration bands were absent in Cu (II)complex spectra, indicating that it was not present in the Cu(II) complex. This conclusion was further reinforced by the results of the CHN analysis.

**Table 2.** *IR data of Cu (II) complex of HMPSMOSchiff base ligand*

| S. No. | Syn. compound | ν (C=N) (cm$^{-1}$) | $\nu_{asym}(SO_2)$ (cm$^{-1}$) | $\nu_{sym}(SO_2)$ (cm$^{-1}$) | ν(OH) (cm$^{-1}$) phenolic | ν(C-O) (cm$^{-1}$) phenolic | ν(NH) (cm$^{-1}$) | ν(M-O) (cm$^{-1}$) | ν(M-N) (cm$^{-1}$) |
|---|---|---|---|---|---|---|---|---|---|
| 1. | **$C_{19}H_{20}N_4S_2O_3$ (HMPSMO)** | 1630 | 1332 | 1140 | 3410 | 1460 | 3360 | - | - |
| 2. | **$C_{38}H_{38}CuN_8S_4O_6$ (HMPSMO-Cu)** | 1615 | 1332 | 1140 | - | 1440 | 3358 | 563 | 460 |

### 3.2. Cyclic Voltammetric studies of Cu (II) complex of HMPSMO Schiff base ligand

The cyclic voltammetry technique was used to investigate the electrochemical properties of Cu (II) complex of sulpha drug ligand. The redox behaviour of Cu (II) metallic compound has been studied in a DMF solvent with 0.1 M $NaClO_4$ as a supporting electrolyte. A single electrochemical compartment cell with the previously mentioned three-electrode assembly is used for electrochemical studies. The cyclic voltammograms were recorded in the potential window of +0.7 to 1.2V at various scan rates of 100,150,200,250,300mVs$^{-1}$.The cyclic voltammograms and their parameters are shown in Figure 2 and in Table 3, respectively. The cyclic voltammograms show the negative potential of complex ranging from -0.50V to -0.63V,-0.52 to 0.65V and 0.63V to 0.69V and in reverse scan, a corresponding anodic wave occurs in the range of -0.15V to- 0.14V,0.17V to 0.14V and 0.17V to 0.14V at 1mM, 2mM and 3mM concentrations, respectively.

**Table 3.** *Cyclic voltammogram parameters of the Cu (II) complex of HMPSMO Schiff base ligand*

| Complex Conc. | v(mV/s) | $E_{pc(mV)}$ | $Epa_{(mV)}$ | ΔEp | $E_{1/2}$ | $Ipc_{(\mu A)}$ | $Ipa_{(\mu A)}$ | Ipa/Ipc | Ipc/ $v^{1/2}$ |
|---|---|---|---|---|---|---|---|---|---|
| 1Mm | 100 | -502 | -150 | 352 | -370 | 0.0088 | 0.0065 | 0.738636 | 0.000880 |
| | 150 | -560 | -130 | 430 | -392 | 0.0101 | 0.0078 | 0.772277 | 0.000825 |
| | 200 | -580 | -140 | 440 | -400 | 0.0105 | 0.009 | 0.857143 | 0.000742 |
| | 250 | -600 | -145 | 455 | -410 | 0.011 | 0.0097 | 0.881818 | 0.000696 |
| | 300 | -630 | -140 | 465 | -440 | 0.0129 | 0.0101 | 0.782946 | 0.000745 |
| 2Mm | 100 | -520 | -170 | 350 | -370 | 0.0109 | 0.0098 | 0.899083 | 0.001090 |
| | 150 | -550 | -160 | 390 | -392 | 0.0127 | 0.0111 | 0.874016 | 0.001037 |
| | 200 | -620 | -150 | 470 | -420 | 0.0133 | 0.0113 | 0.849624 | 0.000940 |
| | 250 | -640 | -145 | 495 | -450 | 0.0152 | 0.0115 | 0.756579 | 0.000961 |
| | 300 | -650 | -170 | 465 | -430 | 0.0156 | 0.0123 | 0.788462 | 0.000901 |
| 3Mm | 100 | -630 | -170 | 460 | -430 | 0.0205 | 0.0146 | 0.712195 | 0.002050 |
| | 150 | -650 | -160 | 490 | -450 | 0.0127 | 0.0111 | 0.874016 | 0.001037 |
| | 200 | -680 | -150 | 530 | -470 | 0.0226 | 0.016 | 0.707965 | 0.001598 |
| | 250 | -690 | -145 | 545 | -500 | 0.022 | 0.0139 | 0.631818 | 0.001391 |
| | 300 | -696 | -155 | 465 | -510 | 0.0248 | 0.0164 | 0.66129 | 0.001432 |

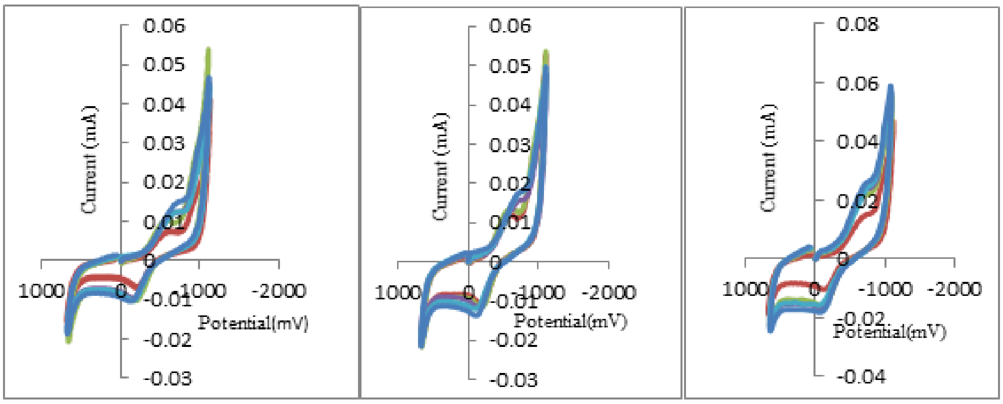

**Figure 2.** *Cyclic voltammograms of Cu (II) complex of HMPSMO in DMF-NaClO$_4$ at different concentrations, 1mM(A),2mM(B) and 3mM(C)*

The CV of complex shows a quasi-reversible peak corresponding to the formation of Cu(II)/Cu(I) couple at a given potential. The peak separation (ΔEp) varies from 0.352V to 0.465V at 1mM concentration, and the ratio between the anodic and cathodic peak current is less than unity (IPa/IPc<1), corresponding to a simple one-electron transfer process confirms the redox couple's quasi-reversibility[30-31]. A linear dependence between cathodic peak current (Ipc) and the square of the scan rates ($\upsilon^{1/2}$) has also been observed (Figure.3B).This fact implies that these electrochemical processes are mainly diffusion-controlled [32].The ligand does not show cathodic and anodic potential as in the above range, so the redox process is assigned to the copper centre only.

### Effect of scan rate on the voltammogram of Cu (II) complex

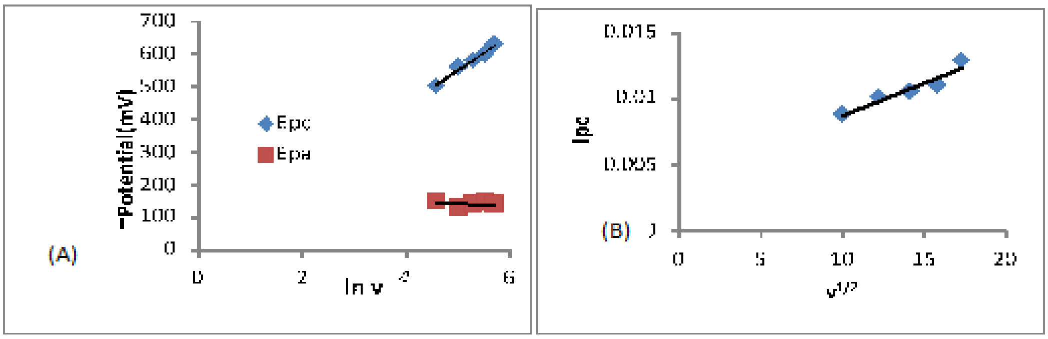

**Figure 3.** *Agraphical representation, potential (Epc&Epa) vs lnv(A) and Ipcvs $v^{½}$ (B) for Cu (II) complex at 1mM concentration*

The CV graphs of Cu (II) complex showed that both cathodic and anodic peak currents linearly increase with the increase of scan rate [33]. The Epc and Epa values slightly shifted towards negative and positive directions, respectively, with the scan rate (Fig. 3A). The variation of Ipc values with the scan rate is represented in Figure. 3B. It confirms the quasi-reversible redox behaviour of the copper complex.

### 3.3. Effect of concentrations on the voltammogram of Cu (II) complex

Electrochemical characteristics included a progressive rise in peak current and cathodic peak potential with increasing Cu(II) concentration(Figure. 4 A&B), suggesting the presence of multiple electro-active species on the electrode surface [33].

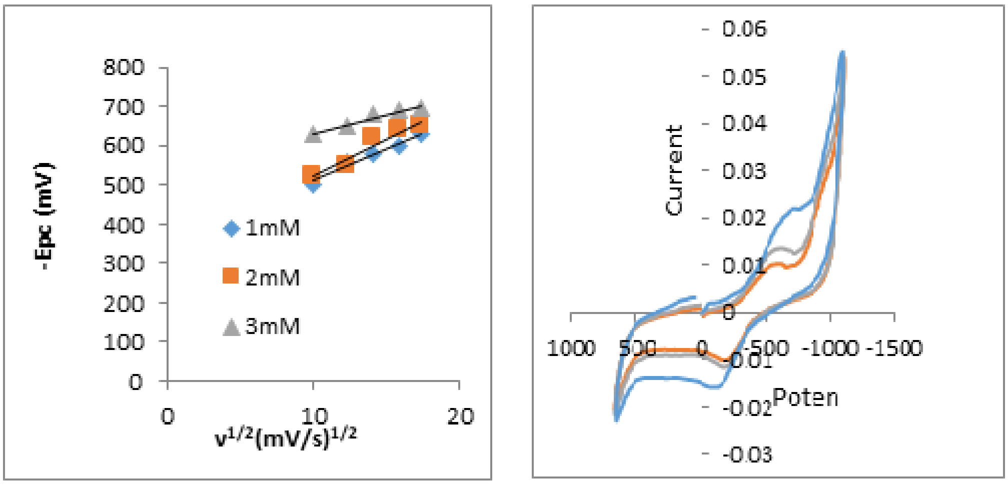

**Figure 4.** *Graphical representation Epc vs ln v for Cu (II) complex at different concentrations* ***(A)*** *and cyclic voltammograms of complex at1mM-3mM conc in DMF-NaClO$_4$ at 150 scan rate* ***(B)***

## 4. CONCLUSION

A novel Cu (II) complex with the bidentate Schiff base ligand HMPSMO (L) was synthesized and characterized, forming a square planar CuL$_2$ complex through facile coordination with Cu (II). Cyclic voltammetry studies demonstrate that the Cu (II) mononuclear complex exhibits a one-electron, quasi-reversible, diffusion-controlled electrochemical process. The effects of concentration and scan rate on the cyclic voltammograms were investigated, revealing that both peak current and cathodic peak potential increase with higher concentrations and scan rates.

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