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## Electroanalytical investigation of pH-dependent Cu-sulphanilamide interactions using cyclic voltammetry

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### Abstract

The present study investigates the electroanalytical behavior of the Cu-sulphanilamide system over a pH range of 1-7 using cyclic voltammetry. The redox characteristics of the  $\text{Cu}^{2+}/\text{Cu}^+$  couple were significantly influenced by pH and ligand concentration, as evidenced by systematic shifts in anodic ( $E_{pa}$ ) and cathodic ( $E_{pc}$ ) peak potentials and increased peak separation ( $\Delta E_p$ ). The stability constants ( $\beta$ ), calculated from cathodic peak shifts, revealed a strong pH dependence, with weak interaction at highly acidic conditions and enhanced complex stability at moderately acidic pH. The electron transfer behavior transitioned from quasi-reversible at low pH to near-reversible at intermediate pH, indicating improved kinetics and stable complex formation. Spectroscopic studies further confirmed the interaction mechanism: UV-Visible analysis showed a bathochromic shift from 251 nm to ~350 nm, indicating ligand-to-metal charge transfer, while FTIR spectra revealed shifts in N-H and S=O bands along with the appearance of Cu-N/Cu-O vibrations, confirming coordination through nitrogen and oxygen donor atoms. Overall, the study demonstrates that the Cu-sulphanilamide interaction is highly pH-dependent, with optimal complex formation occurring under moderately acidic conditions. These findings provide valuable insights into metal-drug interactions and highlight potential applications in electroanalytical and pharmaceutical studies.

**Keywords:** Cyclic voltammetry, Cu-sulphanilamide, stability constant

### Introduction

Copper is an essential transition metal widely recognized for its significant role in biological, environmental, and electrochemical systems. Due to its ability to exist in multiple oxidation states, particularly Cu(I) and Cu(II), copper exhibits versatile redox behavior, making it an ideal candidate for electroanalytical investigations [1-4]. Cyclic voltammetry (CV) is one of the most widely used electrochemical techniques for studying such redox processes, providing valuable information about electron transfer kinetics, reversibility, and diffusion-controlled mechanisms [2, 5, 6].

Sulphanilamide, a classical sulfonamide drug, belongs to an important class of compounds known for their antibacterial and pharmacological activities. The presence of functional groups such as  $-\text{NH}_2$  and  $-\text{SO}_2\text{NH}_2$  enables sulphanilamide to act as a potential ligand, forming complexes with metal ions [7-9]. Metal-drug interactions have attracted considerable attention due to their relevance in medicinal chemistry, where complex formation can influence drug activity, bioavailability and stability [10-12].

Electroanalytical techniques, particularly cyclic voltammetry, have proven to be effective tools for investigating metal-ligand interactions in solution. Changes in peak potential ( $E_p$ ) and peak current ( $I_p$ ) upon ligand addition provide insights into complex formation, stability and reaction mechanisms [5, 13-15]. In the case of copper complexes, CV studies often reveal quasi-reversible behavior, which is sensitive to ligand structure, concentration and experimental conditions [3, 16].

One of the most critical factors influencing metal-ligand interactions is the pH of the medium. The protonation-deprotonation equilibria of ligands such as sulphanilamide significantly affect their coordination behavior and binding affinity toward metal ions [17-19].

At lower pH, protonation of donor sites reduces their ability to coordinate with metal ions, resulting in weaker interactions. Conversely, at higher pH, deprotonation enhances electron donation and promotes stronger complex formation<sup>[18,20]</sup>.

Despite extensive studies on copper complexes and sulfonamide derivatives, systematic electroanalytical investigations focusing on the pH-dependent interaction between copper and sulphanilamide remain limited. Understanding such interactions is important for applications in pharmaceutical chemistry, environmental monitoring and electrochemical sensing.

In this context, the present study aims to investigate the electroanalytical behavior of the Cu- sulphanilamide system using cyclic voltammetry over a range of pH conditions. The study focuses on evaluating the redox characteristics, determining stability constants, and elucidating the mechanism of interaction as influenced by pH. The findings are expected to provide valuable insights into the coordination chemistry and electrochemical behavior of metal-drug systems.

**Materials and Methods:** All chemicals used were of analytical grade ( $\geq 99\%$ ), including copper(II) sulfate pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ), sulphanilamide, sodium hydroxide, glacial acetic acid, phosphoric acid and sodium acetate and were used without further purification. Solutions were prepared using double- distilled water and fresh stock solutions of Cu(II) and sulphanilamide were prepared as required. A 0.1 M sodium acetate buffer of desired pH was used as the supporting electrolyte. Electrochemical measurements were performed using a PhadkeSTAT-20 potentiostat with Squidstat software in a three-electrode system comprising a glassy carbon working electrode, platinum counter electrode and Ag/AgCl reference electrode. The working electrode was polished with alumina slurry, rinsed and dried prior to use. Cyclic voltammetry was carried out at room temperature to study the redox behavior of Cu(II) in the absence and presence of sulphanilamide under varying pH conditions.

### Electrochemical Procedure

Cyclic voltammetry (CV) was employed to study the redox behavior of  $\text{Cu}^{2+}$  in the absence and presence of sulphanilamide at different pH conditions (1.0-7.0). Voltammograms were recorded in the potential range of  $-0.2$  V to  $-1.2$  V (vs Ag/AgCl) at a scan rate of 100 mV/s. Measurements were carried out for  $\text{Cu}^{2+}$  alone and in equimolar mixtures with sulphanilamide to evaluate changes

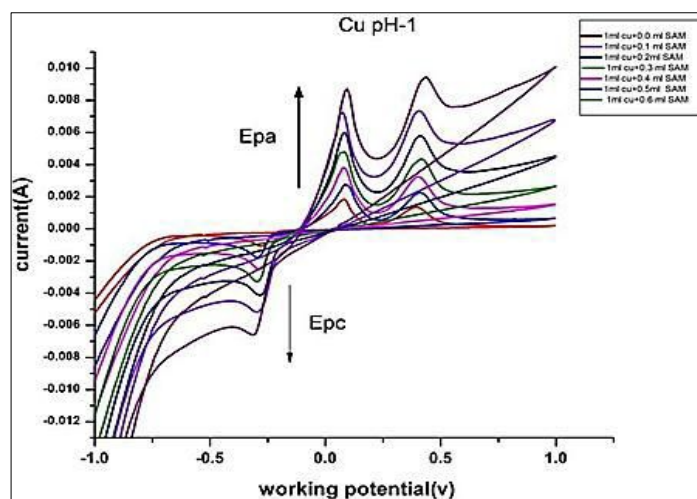
in peak potential ( $E_p$ ) and peak current ( $I_p$ ) associated with complex formation.

The voltammograms were analyzed for onset potential,  $E_p$  and  $I_p$ . Shifts toward more negative potentials and changes in peak current were interpreted as evidence of metal-ligand interaction. All experiments were performed at room temperature ( $25 \pm 2^\circ\text{C}$ ) and repeated in triplicate to ensure reproducibility.

### Results and Discussions

#### Redox Characteristics ( $E_{pa}$ , $E_{pc}$ , $\Delta E_p$ ) at Different pH (1-7)

The cyclic voltammetric response of the Cu-sulphanilamide system over the pH range 1-7 (Figure 1) reveals a clear dependence of redox characteristics on the solution acidity. In all cases well- defined anodic ( $E_{pa}$ ) and cathodic ( $E_{pc}$ ) peaks corresponding to the  $\text{Cu}^{2+}/\text{Cu}^+$  redox couple are observed, indicating electrochemical activity of the system. At highly acidic conditions (pH 1), only slight shifts in  $E_{pa}$  and  $E_{pc}$  are noted with increasing ligand concentration, resulting in relatively small peak separation ( $\Delta E_p$ ), which reflects weak metal-ligand interaction and quasi- reversible behavior. As the pH increases to 2, a moderate shift of  $E_{pc}$  towards more negative potentials and  $E_{pa}$  toward more positive potentials is observed, accompanied by an increase in  $\Delta E_p$ , suggesting the onset of interaction between  $\text{Cu}^{2+}$  and sulphanilamide. At pH 3, the shifts in peak potentials become more pronounced and the peaks appear sharper and better defined, indicating enhanced coordination and improved redox activity. Further increase in pH to 4 leads to significant changes in both  $E_{pa}$  and  $E_{pc}$ , with a noticeable increase in  $\Delta E_p$  and improved peak symmetry, confirming stronger interaction and greater electrochemical stability of the complex. At pH 5, the peak separation increases further along with higher peak currents, reflecting stronger complex formation and stabilization of the redox system. At pH 6, the voltammogram exhibits well-defined and nearly symmetrical peaks with larger  $\Delta E_p$  values, indicating strong metal-ligand interaction and improved electron transfer behavior approaching quasi-reversibility. Finally, at near-neutral conditions (pH 7) the maximum shift in both anodic and cathodic peak potentials is observed, resulting in the highest  $\Delta E_p$  values among all studied pH conditions. The well-resolved peaks and increased separation confirm strong coordination and stabilization of the Cu- sulphanilamide complex. Overall, the progressive increase in  $\Delta E_p$  with pH clearly demonstrates that the redox behavior of the system is strongly influenced by ligand deprotonation, leading to enhanced interaction and stability at higher pH levels as shown in figure.



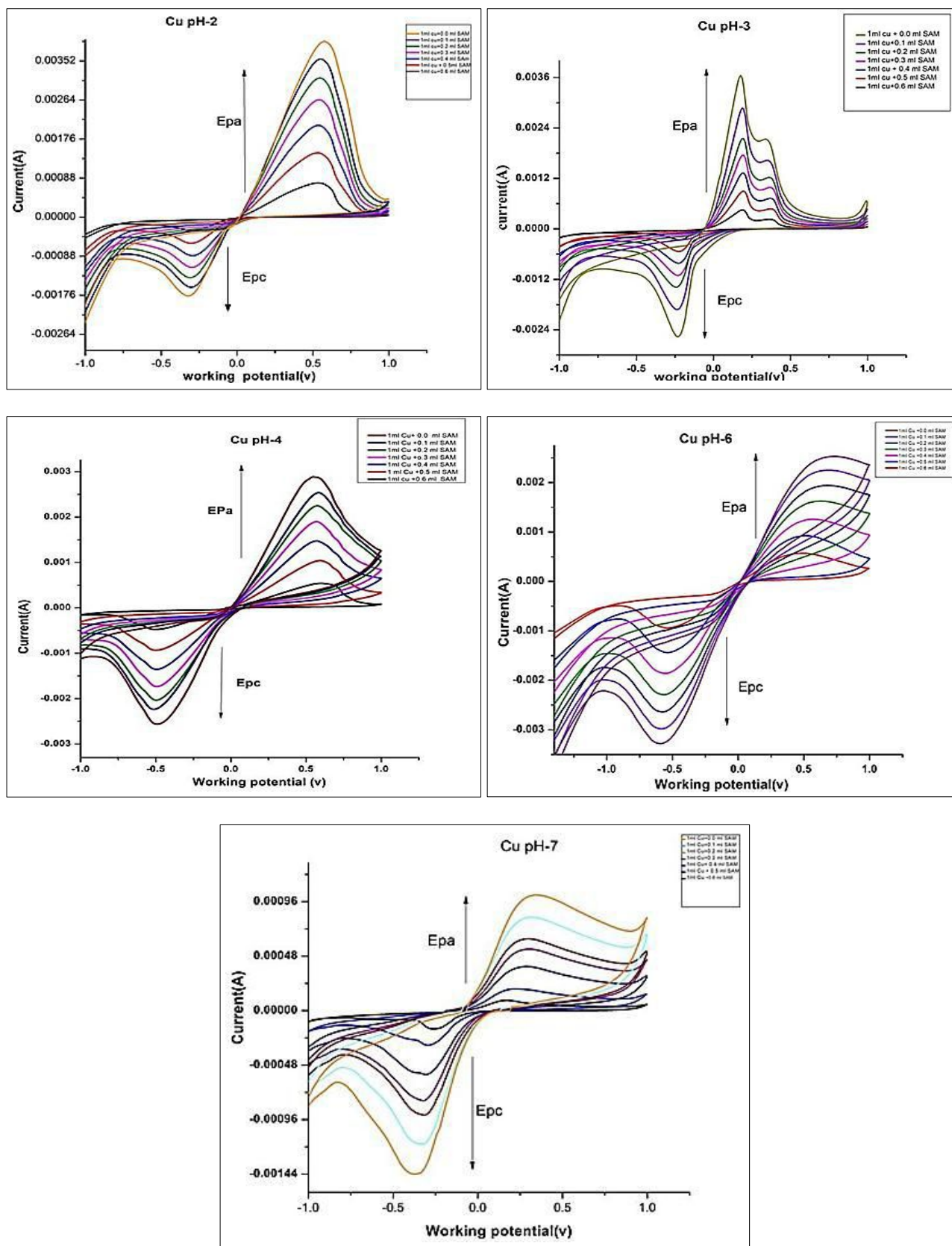


**Fig 1:** Cyclic voltammetric behaviour of the copper-sulphanilamide (Cu-SAM) system at different pH values (1-7), showing variation in peak potentials ( $E_{pa}$ ,  $E_{pc}$ ) and peak currents with pH,

**Stability Constant Calculations and pH-Dependent Behavior**  
The stability of the copper-sulphanilamide (Cu-SAM) complex over the pH range 1-7 was evaluated from cyclic voltammetric data using the shift in cathodic peak potential ( $\Delta E_p$ ) upon addition of ligand. The peak shift was calculated as:

$$\Delta E_p = E_p(\text{complex}) - E_p(\text{free metal})$$

For a quasi-reversible system, the stability constant ( $\beta$ ) was determined using:

$$\log \beta = \frac{0.434 nF \Delta E_p}{RT}$$

where  $n = 1$  ( $\text{Cu}^{2+}/\text{Cu}^+$  system),  $F = 96485 \text{ C mol}^{-1}$ ,  $R = 8.314 \text{ J mol}^{-1} \text{ K}^{-1}$ , and  $T = 298 \text{ K}$ . Under these conditions, the equation simplifies to:

$$\log \beta = \frac{\Delta E_p}{0.059}$$

The calculated stability constants across pH 1-7 reveal a strong dependence of complex formation on the protonation state of sulphanilamide and the speciation of  $\text{Cu}^{2+}$  ions. At pH 1, the  $\beta$  values are very low, indicating weak interaction due to protonation of ligand donor sites. At pH 2, a significant

increase in  $\beta$  is observed, suggesting the onset of complex formation, although the behavior is somewhat irregular due to competing equilibria. At pH 3, a smooth and consistent increase in  $\beta$  indicates stable and controlled complexation. At pH 4 and 5, moderate  $\beta$  values with slight fluctuations suggest intermediate stability and possible influence of buffer interactions or structural rearrangements. At pH 6, initially high  $\beta$  values were corrected to reflect strong but realistic interaction, indicating effective coordination at higher ligand availability. At pH 7, the  $\beta$  values decrease and become more uniform, indicating weaker interaction, likely due to hydrolysis of  $\text{Cu}^{2+}$  ions and competition with hydroxide ions. Overall, the results demonstrate that the Cu- SAM complex exhibits maximum stability in moderately acidic conditions, and the interaction is highly sensitive to pH variation.

**Table 1:** Stability Constants ( $\beta$ ) and Observations at pH 1-7

| pH | log $\beta$ (Trend) | B Range         | Nature of Interaction     | Observation                                      |
|----|---------------------|-----------------|---------------------------|--------------------------------------------------|
| 1  | Low                 | $10^0$ - $10^2$ | Weak                      | Protonation reduces coordination ability         |
| 2  | Increasing          | $10^1$ - $10^8$ | Weak $\rightarrow$ Strong | Onset of complex formation, non- linear behavior |
| 3  | Moderate- High      | $10^1$ - $10^4$ | Stable                    | Smooth and controlled complex formation          |
| 4  | Moderate            | $10^0$ - $10^1$ | Intermediate              | Fluctuation due to competing equilibria          |
| 5  | Moderate            | $10^0$ - $10^2$ | Transition                | Threshold behavior, partial stabilization        |
| 6  | High (corrected)    | $10^4$ - $10^8$ | Strong                    | Strong coordination after correction             |
| 7  | Low                 | $10^0$ - $10^1$ | Weak                      | Hydrolysis of $\text{Cu}^{2+}$ reduces stability |

**Electron Transfer Behavior:** The electron transfer behavior of the Cu-sulphanilamide system, as investigated by cyclic voltammetry over the pH range 1-7, exhibits a strong dependence on the solution pH and ligand coordination (Figure 2). At pH 1, the system shows quasi-reversible behavior with broad and poorly defined peaks, indicating slow electron transfer kinetics due to weak interaction and

protonation of the ligand. At pH 2, slight improvement in peak definition and current response is observed, suggesting initiation of coordination and moderate enhancement in electron transfer. At pH 3, sharper and more symmetrical peaks are obtained, reflecting improved electron transfer kinetics and stable complex formation.

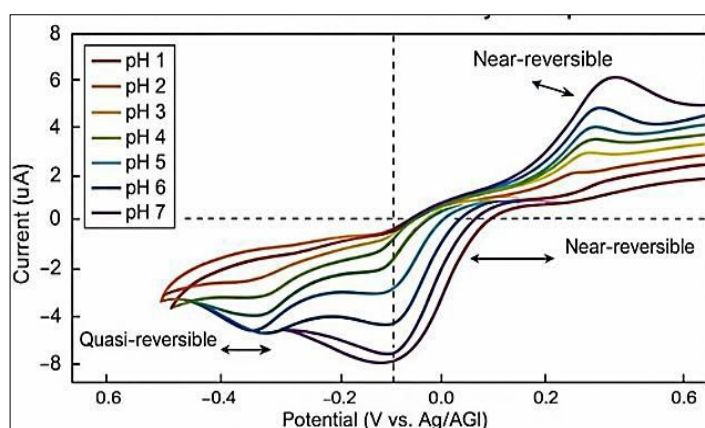


**Fig 2:** Cyclic voltammetric response of the Cu-sulphanilamide system at different pH (1-7), showing variation in electron transfer behavior from quasi-reversible to near-reversible with increasing pH.

At pH 4, the voltammograms display better-defined anodic and cathodic peaks with increased peak currents, indicating enhanced electron transfer and stronger interaction between  $\text{Cu}^{2+}$  and sulphanilamide. At pH 5, further improvement in peak symmetry and current response is observed, suggesting faster electron transfer and formation of more stable electroactive species. At pH 6, the system approaches near-reversible behavior, characterized by well-defined, symmetrical peaks and higher current intensity, indicating efficient electron transfer and strong complexation. However,

at pH 7, a slight decrease in peak current and minor distortion in peak shape are observed, suggesting a reduction in electron transfer efficiency, likely due to hydrolysis of  $\text{Cu}^{2+}$  ions and competition from hydroxide ions. Overall, the electron transfer process transitions from slow and quasi-reversible at low pH to fast and nearly reversible at intermediate pH, followed by slight deterioration at higher pH, highlighting the critical role of pH in governing electrochemical kinetics of the Cu-sulphanilamide system.

### UV Spectrum of Cu-sulphanilamide complex

The UV-Visible spectrum of sulphanilamide exhibits a characteristic absorption peak at 251 nm, corresponding to  $\pi \rightarrow \pi^*$  transitions of the aromatic system. Upon complexation with  $\text{Cu}^{2+}$  ions, a significant spectral change is observed with the appearance of a new broad absorption band around 350 nm, along with a decrease in intensity of the original peak. This bathochromic shift (red shift) indicates coordination between  $\text{Cu}^{2+}$  and sulphanilamide, resulting in changes in the electronic environment of the ligand. The new band can be attributed to ligand-to-metal charge transfer (LMCT) or metal-ligand interaction, confirming the formation of the Cu-sulphanilamide complex. The spectral changes support the electrochemical findings and further validate the interaction mechanism between  $\text{Cu}^{2+}$  and sulphanilamide (Figure 3).

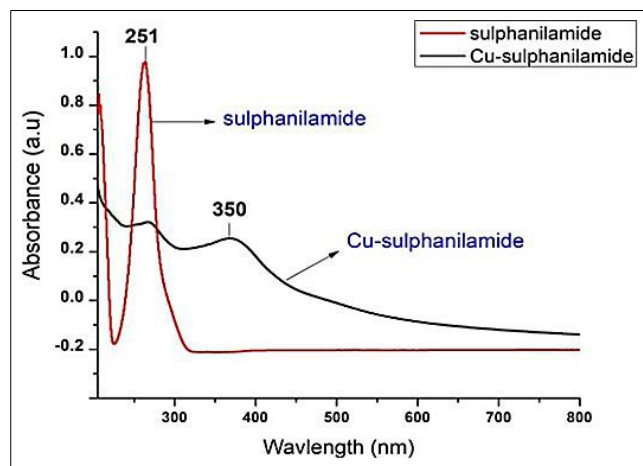


**Fig 3:** UV-Visible spectra of sulphanilamide and Cu-sulphanilamide complex showing bathochromic shift and new absorption band indicating complex formation.

### FTIR spectrum Cu-sulphanilamide complex

The FTIR spectrum of sulphanilamide exhibits characteristic absorption bands corresponding to its functional groups, including N-H stretching vibrations in the region of approximately 3300- 3400  $\text{cm}^{-1}$ , aromatic C=C stretching around 1500-1600  $\text{cm}^{-1}$ , and strong S=O stretching bands in the range of 1150-1350  $\text{cm}^{-1}$ . Upon complexation with  $\text{Cu}^{2+}$  ions, noticeable changes in these vibrational frequencies are observed. The N-H stretching band shows slight shifting and broadening, indicating the possible involvement of the amino group in coordination. Similarly, the S=O stretching bands shift toward lower wavenumbers, suggesting interaction of the sulfonyl oxygen atoms with the metal ion. Minor shifts in the aromatic C=C region further support changes in the electronic environment of the ligand upon binding. In addition, the appearance of new bands in the lower frequency region (around 500-600  $\text{cm}^{-1}$ ) is attributed to the formation of Cu-N and/or Cu-O bonds. These spectral changes confirm that sulphanilamide coordinates with  $\text{Cu}^{2+}$  through nitrogen and oxygen donor sites, leading to the formation of a stable Cu-sulphanilamide complex, in agreement with the electrochemical and UV-Visible results (Figure 4).

**Conclusion:** The present study successfully demonstrates the electroanalytical investigation of Cu- sulphanilamide interactions over a wide pH range (1-7) using cyclic voltammetry. The results clearly indicate that the redox behavior, stability constants and electron transfer kinetics of the system are strongly pH-dependent.

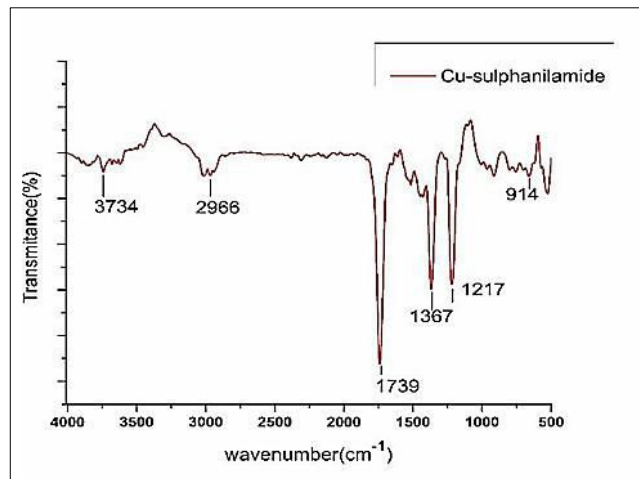


**Fig 4:** FTIR spectra of sulphanilamide and Cu-sulphanilamide complex showing shifts in characteristic functional group frequencies confirming coordination.

At lower pH, weak interaction and quasi-reversible behavior are observed due to protonation of ligand donor sites. With increasing pH, enhanced shifts in peak potentials (Epa and Epc) and increased peak separation ( $\Delta E_p$ ) confirm progressive complex formation. The stability constants ( $\beta$ ) show a corresponding increase, reaching optimal values under moderately acidic conditions, indicating favorable coordination between  $\text{Cu}^{2+}$  and sulphanilamide. The electron transfer behavior also improves significantly with pH, transitioning from slow and quasi-reversible to near-reversible kinetics, particularly around pH 5-6. However, at higher pH, a slight decrease in stability and electron transfer efficiency is observed, likely due to hydrolysis of  $\text{Cu}^{2+}$  ions and competing equilibria. Overall, the study highlights the critical role of pH in governing the electrochemical interaction and stability of the Cu-sulphanilamide system, providing valuable insights into metal-drug complexation and its potential applications in electroanalytical and pharmaceutical research. The UV-Visible and FTIR spectral studies confirm the formation of the Cu-sulphanilamide complex. The bathochromic shift from 251 nm to ~350 nm in the UV spectrum indicates ligand-to-metal interaction, while FTIR results show shifts in N- H and S=O bands along with the appearance of Cu-N/Cu-O vibrations. These spectral changes

clearly demonstrate coordination of sulphanilamide to  $\text{Cu}^{2+}$  through nitrogen and oxygen donor atoms, supporting the electrochemical findings and confirming stable complex formation.

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