

Cyclic Voltammetric Studies of 3,5-Dichloro-2-Hydroxyacetophenone Sulfamethazine -Based Azomethine Compound and its Cu (II) Complex at Glassy Carbon Electrode

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ABSTRACT

Cyclic voltammetry has been used as an electrochemical method to study the electrochemical redox behavior of 3,5-dichloro-2-hydroxyacetophenone sulfamethazine (DCHASMA) and its Cu(II) complex. Cyclic voltammograms of DCHASMA were recorded at glassy carbon electrode at different pH values using Britton–Robinson buffer and aqueous phosphate buffer. Under the examined experimental conditions, the complex exhibits a two-electron quasi-reversible reduction wave in the cathodic area, whereas the cyclic voltammograms of DCHASMA consist of an irreversible cathodic wave that follows a one-step, two-electron transfer electrochemical process. The diffusion controlled mechanism was proven by the linear relationship between the peak current and the square root of the scan rate. On cyclic voltammetric parameters, the effects of solvent, buffer medium, scan rates, and pH were examined. The various kinetic parameters such as charge-transfer coefficient (α_n), diffusion coefficient ($D_0^{1/2}$), and rate constant ($K_{f,h}^0$) were also calculated.

Key word: 3,5-dichloro-2-hydroxyacetophenone sulfamethazine (DCHASMA), Charge-transfer coefficient (α_n), Cyclic voltammetry, Diffusion coefficient ($D_0^{1/2}$) and Rate constant ($K_{f,h}^0$).

1. INTRODUCTION

Cyclic voltammetry is a powerful and popular electrochemical technique commonly used for obtaining qualitative and quantitative information about electrochemical reactions. The technique is significant because of its convenience of simplicity, sensitivity, speed, and low costs which results in versatile applications in a number of areas involving the electron transfer process, both directly and indirectly. It was first theoretically described in 1948 by Randles [1]. Cyclic voltammetry makes possible the elucidation of the kinetics of electrochemical reactions taking place at electrode surfaces. The electrochemical behavior of sulfa drugs derived from Schiff base ligands has also drawn the attention of many workers. They have been a subject of interest in recent decades due to their biological activity, excellent complexing nature, and analytical application [2-5]. A systematic perusal of the review literature reveals that despite the variegated importance associated with Schiff base ligands, relatively insufficient information's are available in the literature regarding the electrochemical behavior of the Schiff bases of sulfa drugs and their metal complexes. Hence, efforts have been laid down to undertake cyclic voltammetric studies on the Schiff base of sulfa drug 3,5-dichloro-2-hydroxyacetophenone sulfamethazine (DCHASMA) and its Cu (II) complex. The present study reports the electrochemical behavior of DCHASMA in various pH levels using phosphate and Britton-Robinson (BR) buffers at glassy carbon electrode (GCE), and the redox behavior of Cu (II) complex in DMF with NaClO₄ as a supporting electrolyte.

2. EXPERIMENTAL

2.1. Materials

All chemicals were analytical grade and of the highest available purity, and used without further purification. For the preparation of DCHASMA,

3,5-dichloro-2-hydroxy acetophenone and sulfamethazine were obtained from Sigma-Aldrich. For the preparation of phosphate buffer and BR buffer, disodium hydrogen phosphate (Na₂HPO₄), sodium dihydrogen phosphate (NaH₂PO₄), acetic acid, phosphoric acid, and boric acid were obtained from Sigma- Aldrich. Double distilled water (DDW) and purified solvent were used for cyclic voltammetric studies.

2.2. Equipment's

Cyclic voltammetric measurements were performed using a computerized constant current source (Cyclic Voltammetry System) provided by retrace electronics which is fully digitally controlled. A three-electrode system consisting of GCE as the working electrode, Ag/AgCl as the reference electrode, and platinum wire as the counter electrode was used. The digital pH meter (Elico) was used to measure the pH of solutions.

2.3. Methods

To prepare phosphate buffer, 0.2 M Na₂HPO₄, and 0.2 M NaH₂PO₄ were prepared separately in two volumetric flasks, and then buffer

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solutions of pH 5.0, 7.0 and 8.2 were prepared by mixing the requisite volume of the solution, individually. Similarly, three BR buffer solutions of pH 5.0, 7.0, and 8.2 were also prepared separately by mixing the requisite volume of acetic acid, phosphoric acid and boric acid and pH maintained by 0.2M NaOH solution using pH meter [6,7].

In the typical cyclic voltammetric experiment, the experimental solution consisted of the compound solution, methanol or acetone, and buffer solution. Before recording the current-voltage curve, the solution was deoxygenated by purging a stream of nitrogen gas for 10–15 min prior to the experiments in order to remove dissolved oxygen from the media. The three electrodes were connected to the electrochemical cell. Required potential, scan rates, current sensitivity, initial potential, and final potential were entered into the menu. After putting the cell on the software was given the command to execute the experiment within the predetermined parameters. The data so obtained were stored in MS Excel programs. The pH of experimental solutions varied from 5.0 to 8.2 and the scan rate was varied from 50 mVs⁻¹ to 250 mVs⁻¹.

For the cyclic voltammetric study of Cu (II) complex, the stock solution of the complex was prepared in DMF solvent. For this purpose, appropriate amount of complex was dissolved in DMF solvent so as to yield 0.01 M solution. 1 M sodium perchlorate solution was prepared in DDW and used as a supporting electrolyte. 1 mL of stock solution (0.01 M) and 1 mL of supporting electrolyte were taken in the electrochemical cell and this solution was made up to 10 mL with DMF solvent, then applied aforementioned method to obtain cyclic voltammogram.

3. RESULTS AND DISCUSSION

The electrochemical reduction behavior of 1 mM DCHASMA in acetone and methanol solvents, using phosphate buffer and BR buffer of pH values 5.0, 7.0, and 8.2 was investigated at GCE within the potential range of +700 mV–1600 mV. The cyclic voltammetric data for DCHASMA are summarized in Tables 1-3 and Figures 1-3 showed the cyclic voltammograms of DCHASMA in acetone-phosphate buffer, acetone BR buffer, and methanol phosphate buffer respectively. The shape of the reduction wave in all cyclic voltammograms of DCHASMA shows the irreversible nature of the electrode process.

Thus, kinetic parameters such as charge transfer coefficient (α_n), diffusion coefficient ($D_0^{1/2}$), and rate constant ($K_{f,h}^0$) have been calculated [8-10] and detailed in Tables 1,2,4.

3.1. Effect of Scan Rate on Cyclic Voltammetric Parameters

A series of cyclic voltammograms of 1 mM DCHASMA were recorded at scan rates 50 mV/s, 100 mV/s, 150 mV/s, 200 mV/s, and 250 mV/s, as shown in Figures 1-3. It is found that the cathodic peak potential and peak current increase linearly with the increasing scan rate. The cathodic peak potential shifted to a more negative value with an increasing scan rate which indicates the irreversibility of the electrochemical process. In all the cyclic voltammograms the current function ($I_{pc}/v^{1/2}$) has been found to be fairly constant with respect to scan rate [Figure 4] showing that the electrode process is diffusion controlled [11,12].

3.2. Effect of Solvent on Cyclic Voltammetric Parameters

Cyclic voltammograms of DCHASMA were recorded in acetone and methanol solvents with phosphate buffer at pH 5.0, 7.0, and 8.2. It showed that the negative peak potential value is maximum in methanol solvent and minimum in acetone solvent [Figure 5] indicates the electrochemical reduction is more facial in more polar solvent. This trend is similar to the trend in viscosity, dielectric constant, and polarity of this solvent [13,14].

3.3. Effect of pH on Cyclic Voltammetric Parameters

The pH effect on the electrochemical behavior of DCHASMA was also investigated at pH 5, 7, and 8.2. The cyclic voltammograms shows that the cathodic peak shifts toward a more negative potential direction with increasing pH value [Figure 6]. This indicates the participation of protons during the electrode process. The reduction is easier at low pH in comparison to higher pH [15]. It confirmed the formation of an easily reducible protonated intermediate during the reduction [Tables 1,2,4].

3.4. Effect of Buffer on Cyclic Voltammetric Parameters

To observe the effect of buffer medium, the electrochemical behavior of DCHASMA was recorded in phosphate buffer and BR buffer with acetone solvent at sweep rates ranging from 50 mV/s to 250 mV/s

Table 1: Scan rate and voltammetric parameters of 1 mM DCHASMA in acetone - BR buffer at pH 5.0, 7.0, and 8.2 [Figure 2a–c, respectively].

pH	v (mVs ⁻¹)	E_{pc}	I_{pc}	$E_{p/2}$	$I_{pc}/v^{1/2}$	α_n	$D_0^{1/2}$ (cm ² s ⁻¹)	$k_{f,h}^0$ (cm.s ⁻¹)
5	50	-910	33.2	-755	4.6951	0.30774	53.2766	1.65×10^{-06}
	100	-940	37.4	-767	3.7400	0.27572	44.8345	4.19×10^{-06}
	150	-970	40.4	-774	3.2986	0.24336	42.0902	1.11×10^{-05}
	200	-983	58.1	-785	4.1082	0.24090	52.6879	1.56×10^{-05}
	250	-991	65.7	-789	4.1552	0.23613	53.8256	1.96×10^{-05}
7	50	-945	42.9	-750	6.0669	0.24461	77.2160	1.43×10^{-05}
	100	-960	40.7	-772	4.0700	0.25372	50.8617	8.37×10^{-05}
	150	-975	43.8	-810	3.5762	0.28909	41.8685	2.03×10^{-06}
	200	-992	48.8	-820	3.4570	0.27732	41.3225	2.95×10^{-06}
	250	-1005	52.5	-830	3.3222	0.27257	40.0566	3.31×10^{-06}
8.2	50	-960	47.3	-760	6.6963	0.23850	86.3113	1.72×10^{-05}
	100	-988	62.3	-770	6.2300	0.21880	83.8367	3.73×10^{-05}
	150	-965	66.3	-790	5.4133	0.27257	65.2686	6.39×10^{-06}
	200	-1010	69.3	-800	4.9030	0.22714	64.7584	2.48×10^{-05}
	250	-1025	71.6	-820	4.5315	0.23268	59.1345	1.80×10^{-05}

Table 2: Scan rate and voltammetry parameters of 1 mM DCHASMA in methanol phosphate buffer at pH 5.0, 7.0, and 8.2 [Figure 3a–c, respectively].

pH	ν (mVs ⁻¹)	E_{pc}	I_{pc}	$E_{p/2}$	$I_{pc}/\nu^{1/2}$	α_n	$D_o^{1/2}$ (cm ² s ⁻¹)	$k_{f,h}^o$ (cm.s ⁻¹)
5	50	-880	37.3	-710	5.2750	0.2805	62.6853	6.74×10 ⁻⁰⁶
	100	-930	44.2	-730	4.4200	0.2385	56.9711	2.12×10 ⁻⁰⁵
	150	-953	53.7	-760	4.3886	0.2471	55.5686	1.51×10 ⁻⁰⁵
	200	-962	60.2	-775	4.2567	0.2550	53.0542	1.15×10 ⁻⁰⁵
	250	-973	67.3	-790	4.2564	0.2606	52.4793	9.37×10 ⁻⁰⁶
7	50	-920	43.5	-740	6.1518	0.2650	75.2243	8.87×10 ⁻⁰⁶
	100	-955	49.8	-755	4.9800	0.2385	64.1892	1.90×10 ⁻⁰⁵
	150	-970	57.4	-769	4.6866	0.2373	60.5594	1.99×10 ⁻⁰⁵
	200	-982	64.7	-778	4.5806	0.2338	59.6292	2.30×10 ⁻⁰⁵
	250	-1003	68.1	-792	4.3063	0.2260	57.0128	2.70×10 ⁻⁰⁵
8.2	50	-950	53.3	-745	7.5377	0.2326	98.3642	2.64×10 ⁻⁰⁵
	100	-975	61.2	-780	6.1200	0.2446	77.8908	1.53×10 ⁻⁰⁵
	150	-993	70.0	-795	5.7203	0.2409	73.3626	1.71×10 ⁻⁰⁵
	200	-1015	68.0	-815	4.8118	0.2385	62.0220	1.48×10 ⁻⁰⁵
	250	-1045	80.6	-840	5.1007	0.2326	66.5624	1.69×10 ⁻⁰⁵

Table 3: Cyclic voltammetric response of 1 mM and 5 mM Cu (II) complex of DCHASMA in DMF solvent containing NaClO₄ as supporting electrolyte.

Conc.	ν (mVs ⁻¹)	E_{pc} (mV)		E_{pa} (mV)	I_{pc} (mA)		I_{pa} (mA)	I_{pa}/I_{pc2}	$E_{1/2}$	ΔE_p	$I_{pc}/\nu^{1/2}$
		E_{pc1}	E_{pc2}		I_{pc1}	I_{pc2}					
1 mM	50	-400	-760	100	0.0038	0.0145	0.0090	0.6206	-330	860	0.00053
	100	-460	-780	130	0.0097	0.0178	0.0126	0.7078	-325	910	0.00097
	150	-490	-785	150	0.0106	0.0194	0.0141	0.7268	-317	935	0.00086
	200	-500	-790	160	0.0137	0.0235	0.0172	0.7319	-315	950	0.00009
	250	-520	-800	170	0.0156	0.0267	0.0190	0.7116	-315	970	0.00098
	300	-540	-810	190	0.0183	0.0324	0.0230	0.7098	-310	1000	0.00105
5 mM	50	-480	-790	190	0.0130	0.0294	0.0221	0.7517	-300	980	0.00183
	100	-500	-800	230	0.0149	0.0338	0.0265	0.7840	-285	1030	0.00149
	150	-510	-805	220	0.0181	0.0380	0.0292	0.7684	-292	1025	0.00147
	200	-540	-810	260	0.0208	0.0389	0.0321	0.8251	-275	1070	0.00147
	250	-550	-812	300	0.0231	0.0438	0.0353	0.8059	-256	1112	0.00146

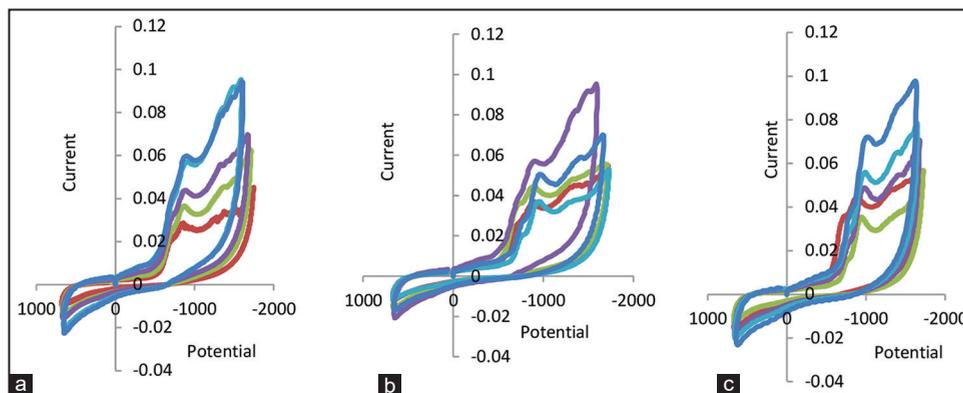


Figure 1: Cyclic voltammograms of 1 mM 3,5-dichloro-2-hydroxyacetophenone sulfamethazine in acetone-phosphate buffer at pH 5.0 (a), 7.0 (b) and 8.2 (c).

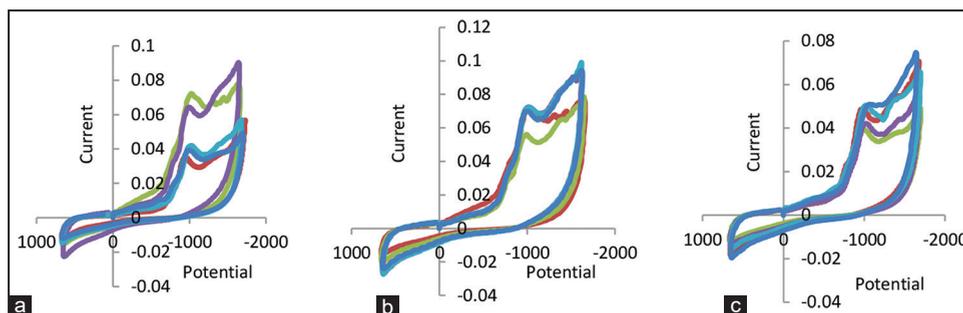


Figure 2: Cyclic voltammograms of 1 mM 3,5-dichloro-2-hydroxyacetophenone sulfamethazine in acetone - BR buffer at PH 5.0 (a), 7.0 (b) and 8.2 (c).

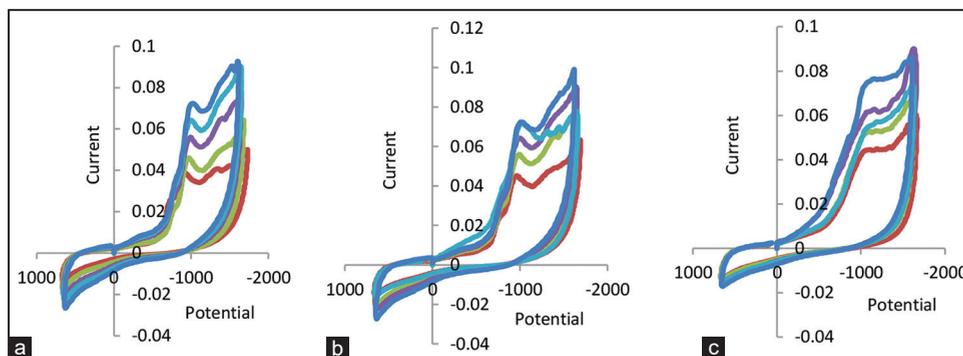


Figure 3: Cyclic voltammograms of 1 mM 3,5-dichloro-2-hydroxyacetophenone sulfamethazine in methanol - phosphate buffer at pH 5.0 (a), 7.0 (b) and 8.2 (c).

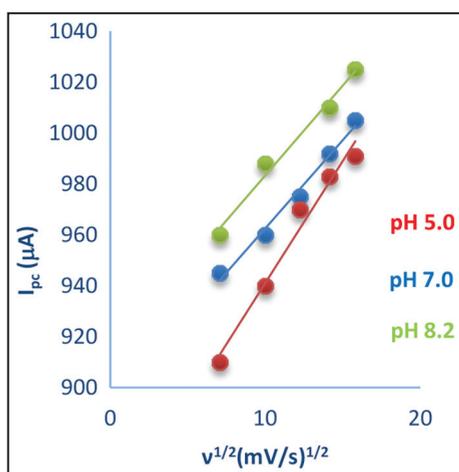


Figure 4: A plot between peak current with the square root of scan rate.

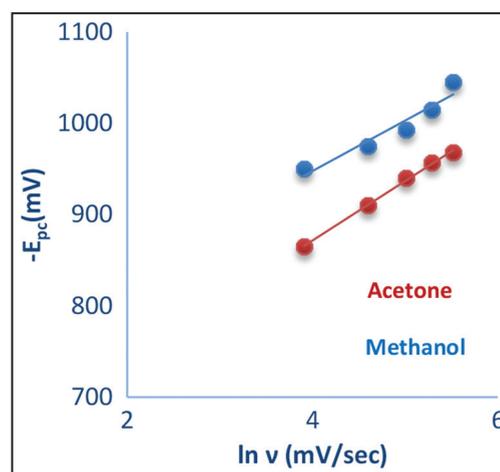


Figure 5: Reduction potential versus $\ln v$ for 1 mM 3,5-dichloro-2-hydroxyacetophenone sulfamethazine in acetone and methanol solvents at pH 8.5 using phosphate buffer.

and at different pH 5, 7, and 8.2 [Tables 1 and 4]. These cyclic voltammograms show that cathodic peak potential swiftness toward a high negative value in BR buffer solution [16]. The E_{pc} values in acetone-phosphate buffer at 50 mV/s to 250 mV/s are -820 mV– -869 mV whereas it is -910 mV– -991 mV at the same scan rate and pH values in acetone - BR buffer medium indicates that the polarity of buffer solution affected electrochemical behavior of redox species [Figure 7].

3.5. Cyclic Voltammetric Response of Cu (II) Complex of DCHASMA

The electrochemical response of Cu(II) complex was studied by recording the cyclic voltammogram at GCE within the potential range from $+700$ mV to -1100 mV at 50 mV s^{-1} – 300 mV s^{-1} scan rates. The cyclic voltammograms and its parameters are shown in Table 3, and in

Figures 8 and 9, respectively.

Using cyclic voltammetric methods, it is possible to examine the electrochemical characteristics of the Cu (II) complex throughout a potential range of $+600$ mV– -750 mV in DMF solvent containing 0.1 M sodium perchlorate. The scan rates were varied from 50 mV s^{-1} to 300 mV s^{-1} . A site observed in Figures 8 and 9, the 1.0 mM Cu(II) complex of DCHASMA exhibits two cathodic peaks E_{pc1} and E_{pc2} in the forward scan at potentials ranging from -400 mV to -540 mV and -760 mV to -800 mV, respectively, and in reverse scan an intense corresponding anodic wave occurs at potentials ranging from $+100$ mV to $+190$ mV. Two cathodic peaks are indicative of the reduction of Cu

Table 4: Scan rate and voltammetric parameters of 1 mM DCHASMA in acetone phosphate buffer at pH 5.0, 7.0, and 8.2 [Figure 1a-c respectively].

pH	ν (mVs ⁻¹)	E_{pc}	I_{pc}	$E_{p/2}$	$I_{pc}/\nu^{1/2}$	α_n	$D_o^{1/2}$ (cm ² s ⁻¹)	$k_{f,h}^{\circ}$ (cm.s ⁻¹)
5	50	-820	27.3	-642	3.8608	0.26797	46.9467	1.42×10^{-05}
	100	-842	35.4	-649	3.5400	0.24715	44.8228	2.90×10^{-05}
	150	-851	41.5	-657	3.3884	0.24587	43.0151	3.25×10^{-05}
	200	-863	53.8	-666	3.8042	0.24213	48.6651	4.26×10^{-05}
	250	-869	57.2	-672	3.6176	0.24213	46.2782	4.28×10^{-05}
7	50	-850	35.02	-645	4.9525	0.23268	64.6287	4.29×10^{-05}
	100	-862	54.2	-654	5.4200	0.22932	71.2441	6.66×10^{-05}
	150	-872	59.06	-665	4.8222	0.23043	63.2340	6.39×10^{-05}
	200	-905	35.05	-695	2.4784	0.22714	32.7341	3.17×10^{-05}
	250	-930	48.2	-730	3.0484	0.23850	39.2925	2.32×10^{-05}
8.2	50	-865	42.7	-675	6.0386	0.25105	75.8643	2.46×10^{-05}
	100	-910	39.9	-727	3.9900	0.26065	49.1944	1.05×10^{-05}
	150	-940	46.78	-739	3.8195	0.23731	49.3549	2.14×10^{-05}
	200	-957	53.8	-742	3.8042	0.22186	50.8398	3.74×10^{-05}
	250	-968	69.5	-755	4.3955	0.22394	58.4685	4.06×10^{-05}

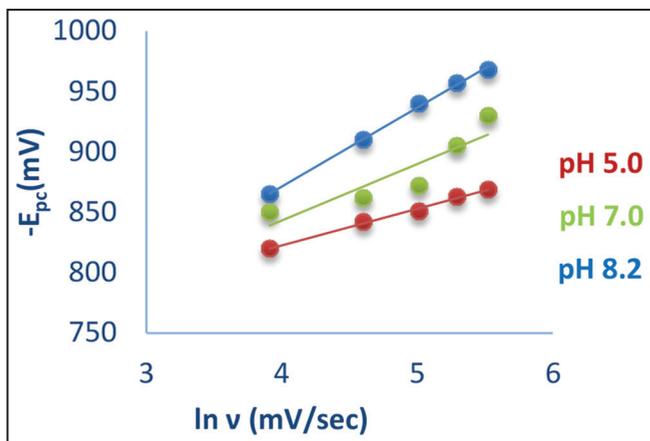


Figure 6: Reduction potential versus $\ln \nu$ for 1 mM 3,5-dichloro-2-hydroxyacetophenone sulfamethazine in acetone - phosphate buffer at 5, 7, and 8.2.

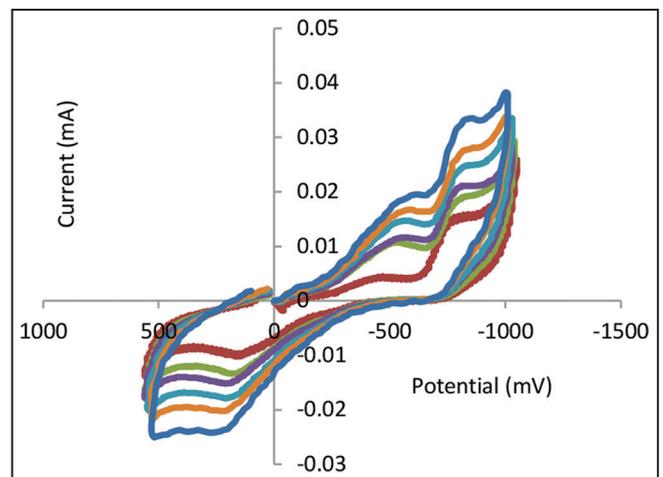


Figure 8: Current potential curves of 1.0 mM Cu (II) complex at 50 mVs⁻¹ to 300 mVs⁻¹ scan rates.

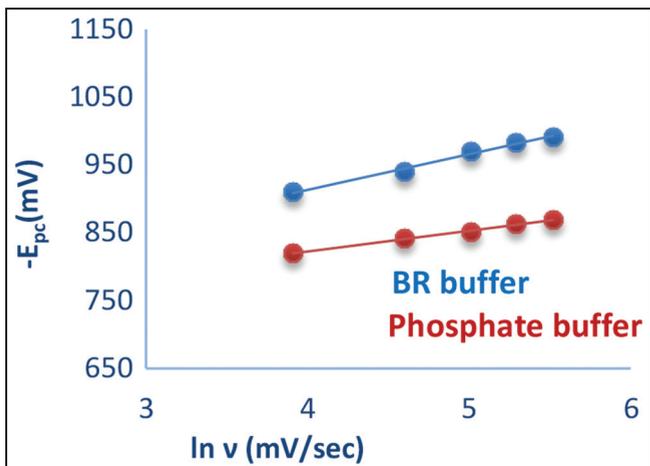


Figure 7: Reduction potential versus $\ln \nu$ for 1 mM 3,5-dichloro-2-hydroxyacetophenone sulfamethazine in acetone solvent at pH 5 with phosphate and BR buffer.

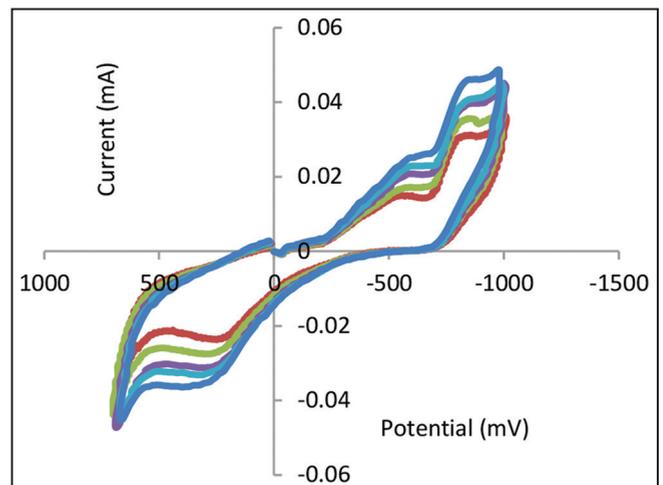


Figure 9: Current potential curves of 5.0 mM Cu (II) complex at 50 mVs⁻¹ to 250 mVs⁻¹ scan rates.

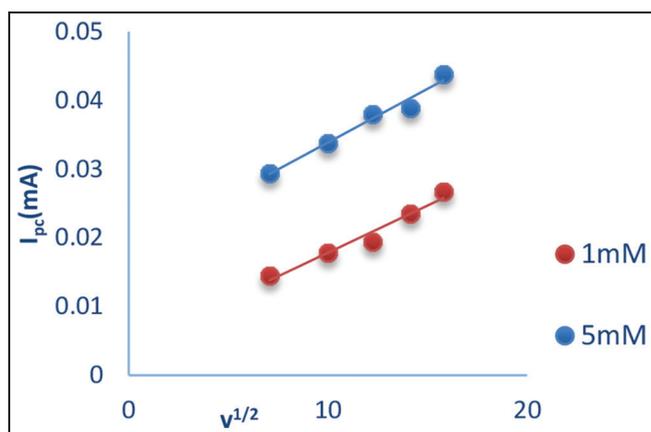


Figure 10: Graphical representation of cathodic peak current (I_{pc}) versus square root of scan rate ($v^{1/2}$) for Cu (II) complex of 3,5-dichloro-2-hydroxyacetophenone sulfamethazine.

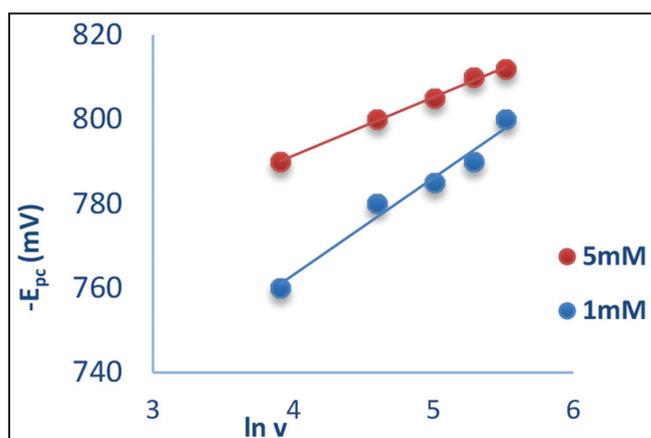


Figure 11: Graphical representation of cathodic peak potential (E_{pc}) versus $\ln v$ for Cu(II) complex of 3,5-dichloro-2-hydroxyacetophenone sulfamethazine.

(II) to Cu (I) and the deposition of Cu (I) to metallic Cu, respectively. The oxidation of Cu (0) to Cu (II) results in one anodic peak E_{pa} being seen in the reverse sweep [17,18]. The above result suggests that Cu (II) complex undergoes two electron transfer redox reactions.

The redox process is attributed to the copper center only because the DCHASMA is bound to the copper ion and it additions not engaged in redox reaction under the applied potential range in experimental conditions. The ΔE_p values listed in Table 3 are considerably greater (860 mV–1000 mV) than the theoretical Nernstian values (59 mV) for an electrochemically reversible one-electron process.

Our results clearly demonstrate that the Cu (II) to Cu (0) reduction in the above-mentioned complexes at GCE is a quasi-reversible, diffusion-controlled process. The fact that the ratio of anodic peak current to cathodic peak current is <1 ($I_{pa}/I_{pc}<1$) confirms the redox couple's quasi reversibility [19].

3.6. Effect of Scan Rate on Voltammogram of Cu (II) Complex

The variation of I_{pc} values with the square root of scan rate is represented in the Figure 10. The current scan responses have linear connection between cathodic peak current (I_{pc}) and the square root of the scan rate ($v^{1/2}$) was seen in all situations. This mean that these electrochemical processes are mostly regulated by diffusion [19]. It is also found that both cathodic and anodic peak current linearly increase

with the increasing of scan rate. The E_{pc} and E_{pa} values slightly shifted toward negative and positive direction respectively with the scan rate shown in figure 11.

3.7. Effect of Concentrations on Voltammogram of Cu(II) Complex

Cyclic voltammetry studies of the Cu (II) complex at 1.0 mM and 5.0 mM concentrations unveiled several key electrochemical properties. The analysis revealed a concentration-dependent behavior, characterized by a linear increase in peak currents for both oxidation and reduction reactions, accompanied by enhanced diffusion intensity toward the electrode surface as concentration increased. Electrochemical characteristics included a progressive rise in peak current with increasing Cu (II) concentration, suggesting the presence of multiple electro-active species on the electrode surface. Mechanistic insights were gained, confirming a diffusion-controlled process and a quasi-reversible character of the electrode couple, supported by the linear correlation between cathodic peak potential (E_{pc}) and $\log v$. These findings provide a comprehensive understanding of the complex's electrochemical properties, highlighting its potential for various applications in fields such as electrochemical sensing, catalysis and materials science.

4. CONCLUSION

In this study, the electrochemical properties of DCHASMA were investigated, revealing a two-electron irreversible reduction and a diffusion-controlled process. The cyclic voltammetric parameters of DCHASMA were found to be sensitive to various experimental conditions, including scan rates, pH values, and the choice of solvent and buffer mediums. In addition, the Cu (II) complex of DCHASMA exhibited a quasi-reversible, diffusion-controlled process in cyclic voltammetry. The electrochemical data confirmed that the cyclic voltammetric parameters of the complex were influenced by both sweep rate and complex concentration. These findings provide valuable insights into the electrochemical behavior of DCHASMA and its copper complex under different experimental conditions.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

The authors declare no conflicts of interest.

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