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Electroanalytical investigation of Schiff base derived from sulfaguanidine at glassy carbon electrode by cyclic voltammetry

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Abstract
A Schiff base compound of sulfa drug was prepared by the condensation reaction of 2-chlorobenzophenone with sulfaguanidine in ethanol. The synthesized compound was characterized by analytical and spectroscopic methods. The electrochemical reduction of synthesized Schiff base in buffered and acetone solvent were studied using the eco-friendly cyclic voltammetry (CV) technique on a glassy carbon electrode at different sweep rates and various pH levels (5,7, 8.2). The resulting voltammograms showed a single irreversible reduction curve in cathodic direction. The electrochemical parameters extracted from the cyclic voltammetry curve were the cathodic peak current and potential (Ipc and Epc, respectively). Therefore kinetic and electrochemical parameters were evaluated using the cathodic peak current and potential. The electrode reaction is found to be diffusion controlled.

Keywords: 2-Chlorobenzophenonesulfaguanidine, cyclic voltammetry, electrochemical parameters, spectroscopic studies, diffusion controlled

Introduction
Schiff bases are typically formed by the condensation of a primary amine and an aldehyde. They are important intermediates for the synthesis of various bioactive compounds [1].
Schiff bases derived from sulpha drugs and many of their metal complexes exhibit a wide range of biological activities, corrosion inhibition effects and analytical applications [2]. Sulpha drugs (also called sulphur drugs or sulfonamide-containing drugs) are an imprecise term that generally refers to drugs that contain a sulphonamide functional group in their chemical structure. Sulfaguanidine belongs to a class of sulfonamides or sulfa drugs extensively used as antibiotic agents for the treatment of intestinal infection, bacillary dysentery [3].
Cyclic voltammetry (CV) is a powerful and popular electrochemical technique commonly employed to investigate the reduction and oxidation processes of molecular species. Cyclic voltammetry (CV) is a versatile electrochemical technique used to analyze redox status in a wide range of industrial and research settings. The common applications include evaluation of drug quality in pharmaceuticals [4], determination of phenolics and antioxidants in food and wine [5], and label-free detection of biomolecules such as hormones [6]. In this paper, we report the synthesis, elemental and spectral analysis, electrochemical behavior of 2-chlorobenzophenone sulfaguanidine (CBPSG).

Experimental
Preparation of Schiff base (CBPSG)
All chemicals used were of analytical reagent (AR) grade and of the highest purity available. Schiff base was prepared by refluxing a solution of 2-chlorobenzophenone with sulfaguanidine in ethanol, then allowed to cool to room temperature. The product was filtered, recrystallized from ethanol and dried in desiccators over anhydrous $CaCl_2$. The purity was checked by elemental analyses. The physical data of corresponding compound are collected in Table-1.

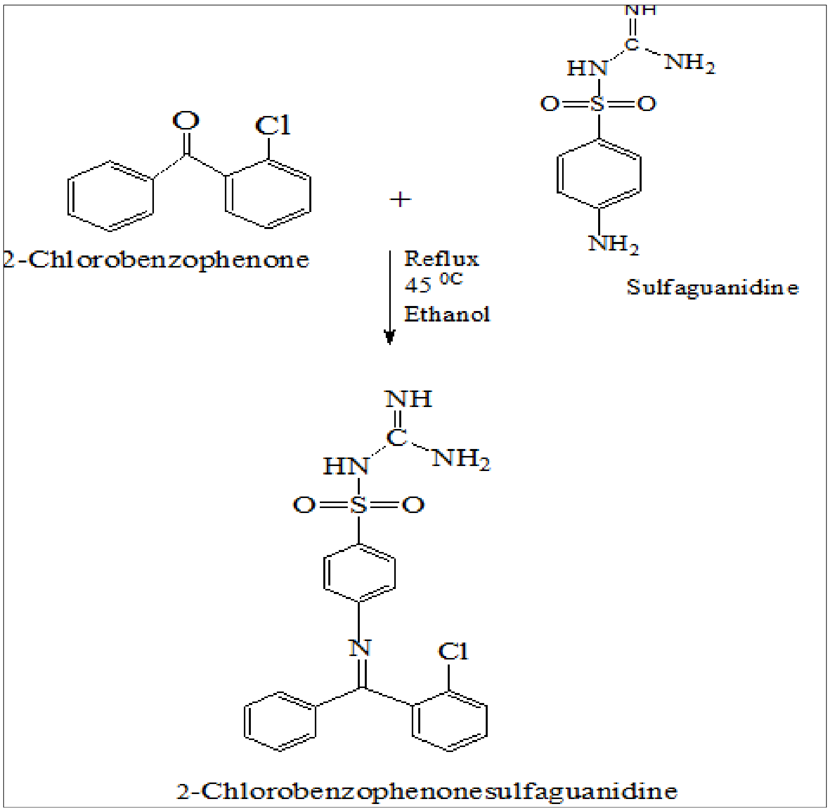


Fig 1: Synthesis of CBPSG

Table 1: Physicochemical Characterization of the Synthesized Schiff Base

Sl. No	Composition of compound	Colour	Melting Point	Yield	Elemental analysis (%):found(cal)				
					C	H	N	Cl	M.Wt
1.	$C_{21}H_{21Cl}N_4O_2S$	White	237 °C	80%	58.80 (58.95)	4.94 (4.87)	13.06 (13.72)	8.27 (8.12)	428.94 (428.11)

Table 2: IR Spectroscopic Studies of the Synthesized Schiff Base

S. No.	Sulfa drug Schiff base	ν(C=N)	ν_{asy} (SO_2)	ν_{sy}(SO_2)
1.	2-Chlorobenzophenone sulfaguanidine (CBPSG)	1611	1226	1144

Electrochemical Studies of Synthesized Schiff Base (CBPSG)

The experimental setup for recording cyclic voltammograms consisted of an electrochemical cell with three electrodes: a glassy carbon electrode with a surface area of 0.0083 cm^2 as the working electrode, a platinum wire as the counter electrode and an Ag/AgCl in a saturated KCl solution as the reference electrode. Cyclic voltammograms measurement were performed using computerized cyclic voltammetry system.

The experimental solution was prepared by mixing 2 ml of a 0.01M stock solution of the compound, (Prepared in acetone), 6ml BR buffer or phosphate buffer of desired pH and 2ml of the solvent. The final solution was made up to 10.0ml (1mM concentration) and used in voltammetric experiments at sweep rates of 25,50,75,100 mV/sec, within a potential window range of +0.8 to -1.6 V, and at a current sensitivity of 0.01mA.

The cyclic voltammograms of the examined compound exhibited a single, 2-electron, irreversible, diffusion-controlled cathodic curve within the entire pH range, attributed to the reduction of the azomethine group -CH=N- to -CH_2-NH-. Upon scanning in the reverse direction, no anodic peak was observed. Representative voltammograms are shown in Figures (2, 3). The electrochemical parameters extracted from the cyclic voltammetry curve included the cathodic peak current and potential (Ipc and Epc, respectively) [7].

The symmetry transfer coefficient (αn), the diffusion coefficient ($D_0^{1/2}$) and rate constant ($K^o f,h$) of the reactant species were determined by using the following equations(1-3) [8] and are listed in Tables 2 to 4.

$$|Ep-Ep/2| = \frac{1.857RT}{\alpha_n F} = \left[\frac{47.7}{\alpha n}\right] \text{mV} \quad (1)$$

$$Ip = 3.01\times10^5 n\,(\alpha_n)^{1/2}\ ACD_0^{1/2}\ v^{1/2} \quad (2)$$

$$Ep = -\frac{RT}{\alpha n F}\ 0.78 + \ln\left[\frac{D_0^{1/2}}{k^0 f,h}\right] + \ln\left(\frac{\alpha_n F v}{RT}\right)^{1/2} \quad (3)$$

Results and Discussion

Cyclic voltammetric studies

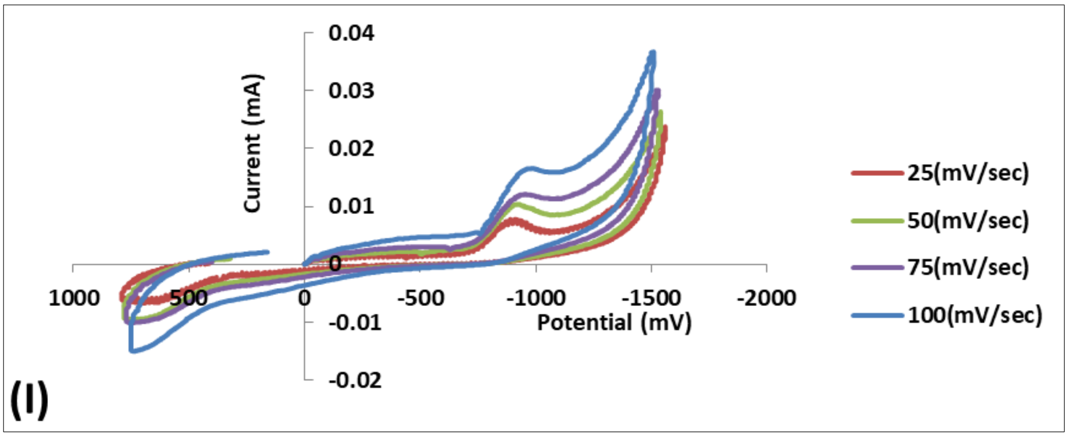
The cyclic voltammetric results for 2-chlorobenzophenone Schiff base are given in Tables 3 to 4, and its voltammograms are depicted in Figures. 2 to 3.

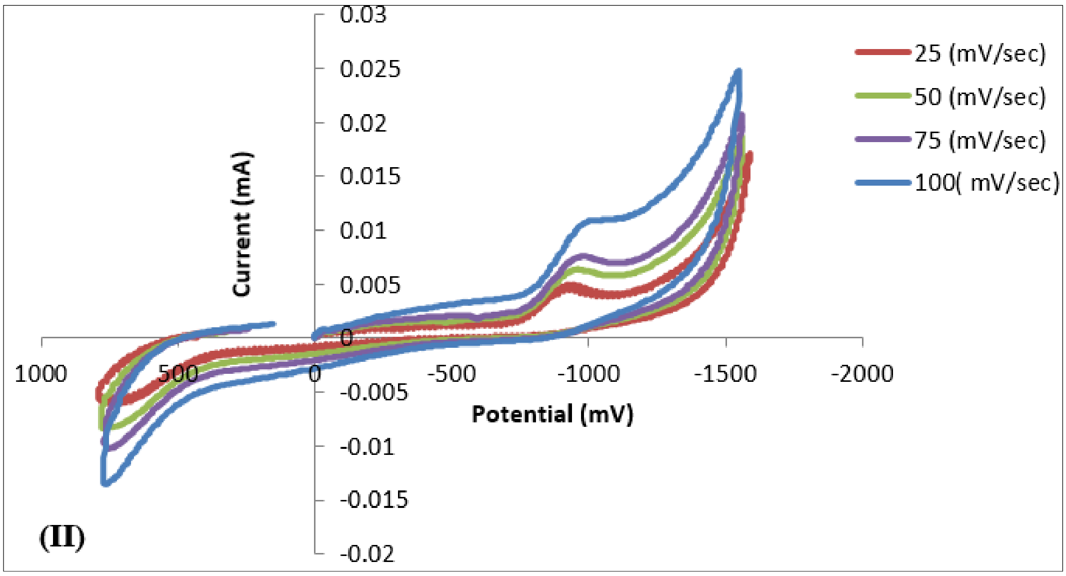
Table 3: Effects of the sweep rate and pH on the voltammetric parameters of CBPSG Schiff base in acetone-phosphate buffer

pH	*v* (mV/sec)	Epc(mV)	Ipc(µA)	Ep/2 (mV)	Ipc/v$^{1/2}$	αn	$D_0^{1/2}$ (Cm^2s^{-1})	K^0f,h ($Cm.s^{-1}$)
5	25	-900	7.1	-770	1.42	0.36692	14.7563	4.62× 10^{-8}
	50	-935	10.3	-800	1.45	0.35334	15.4261	7.21× 10^{-8}
	75	-955	12.0	-820	1.38	0.35334	14.6742	6.38× 10^{-8}
	100	-970	16.4	-840	1.64	0.36692	17.0425	4.24× 10^{-8}
7	25	-930	7.5	-800	1.5	0.36692	15.58772	3.43× 10^{-8}
	50	-970	12.6	-830	1.78	0.34071	19.2163	8.77× 10^{-8}
	75	-990	14.5	-840	1.67	0.28059	18.6896	1.86× 10^{-7}
	100	-1010	16.1	-860	1.61	0.31800	17.9717	1.61× 10^{-7}
8.2	25	-960	20.8	-820	4.16	0.34071	44.8619	6.44× 10^{-8}
	50	-1000	30.35	-850	4.29	0.31800	48.1480	3.45× 10^{-7}
	75	-1020	34.9	-860	4.02	0.34071	43.4589	1.25× 10^{-7}
	100	-1040	43.4	-890	4.34	0.31800	48.4454	2.99× 10^{-7}

Table 4: Effects of sweep rate and pH on voltammetric parameters of CBPSG Schiff base in acetone -BR buffer

PH	*v* (mV/sec)	Epc(mV)	Ipc(µA)	Ep/2 (mV)	Ipc/v$^{1/2}$	$α_n$	$D_0^{1/2}$ (Cm^2s^{-1})	K^0f,h ($Cm.s^{-1}$)
5	25	-930	6.5	-800	1.30	0.36692	13.5093	2.97× 10^{-8}
	50	-960	13.8	-820	1.95	0.34071	21.0464	1.1× 10^{-7}
	75	-990	19.4	-830	2.24	0.34071	24.1577	1.03× 10^{-7}
	100	-1010	24.0	-875	2.40	0.3533	25.4165	5.98× 10^{-8}
7	25	-950	19.5	-820	3.90	0.36692	40.5280	6.71× 10^{-8}
	50	-990	19.7	-840	2.78	0.31800	31.0988	2.6× 10^{-7}
	75	-1020	26.3	-865	3.03	0.30771	34.4618	3.5× 10^{-7}
	100	-1050	35.6	-860	3.56	0.31800	39.7387	2.17× 10^{-7}
8	25	-980	22.5	-830	4.50	0.31800	50.2315	3.26× 10^{-7}
	50	-1010	30.7	-870	4.34	0.34071	46.8207	1.26× 10^{-7}
	75	-1040	34.0	-890	3.92	0.31800	43.8239	2.34× 10^{-7}
	100	-1070	45.0	-925	4.50	0.32897	49.3868	1.35× 10^{-7}





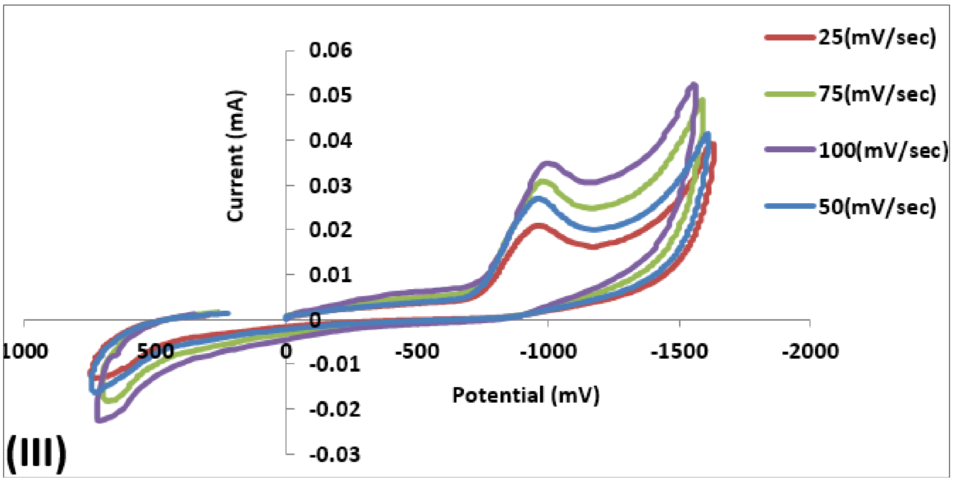


Fig 2: Cyclic voltammograms of CBPSG recorded at a glassy carbon electrode in acetone with phosphate buffer (I) 5.0 pH (II) 7 pH (III) pH 8.2

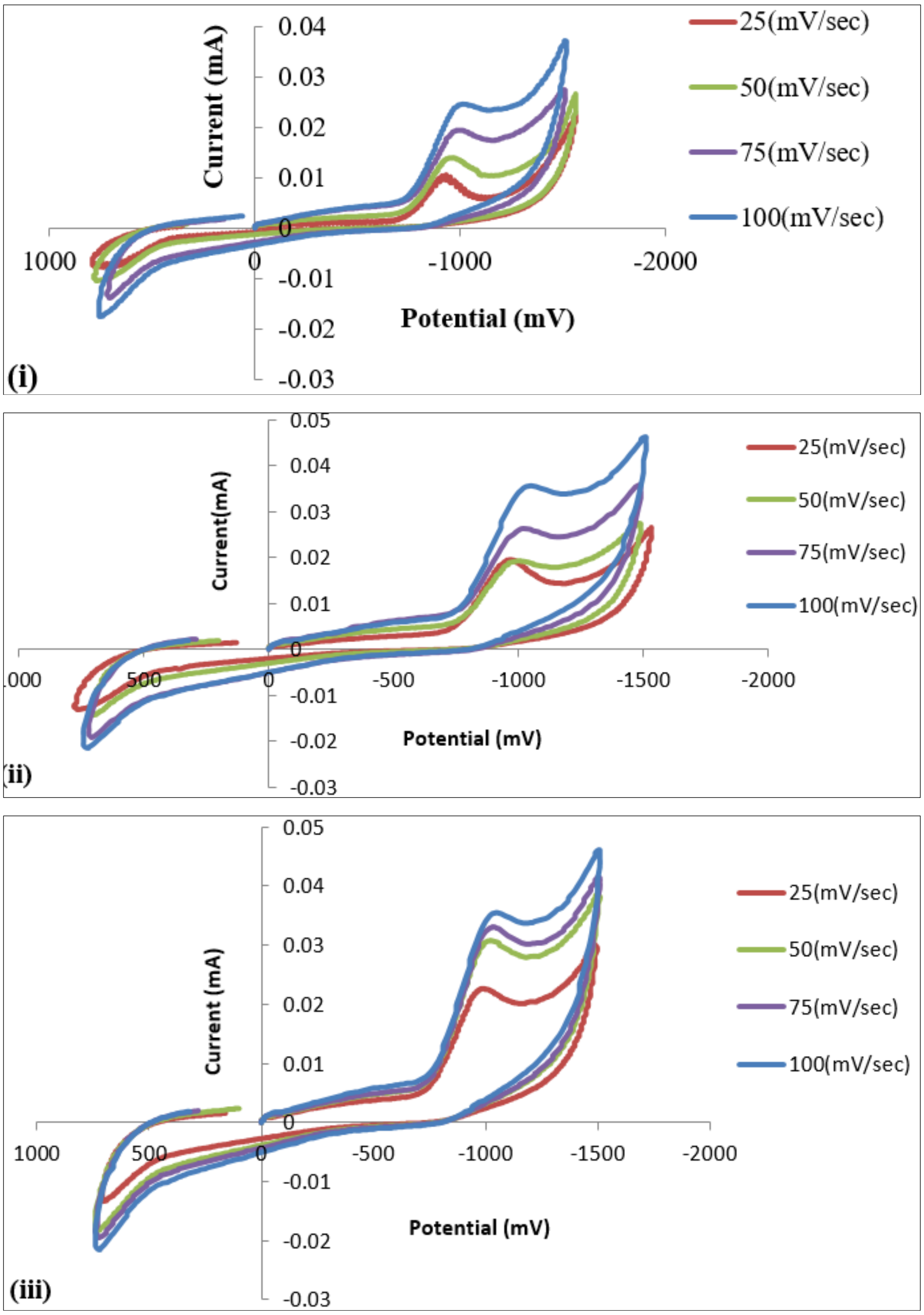


Fig 3: Cyclic voltammograms of CBPSG recorded at a glassy carbon electrode in acetone with BR buffer at (i) 5.0 pH (ii) 7 pH (iii) pH 8.2

The effect of various factors (pH, sweep rates, and buffers) on electrochemical parameters

The effect of scan rate on the reduction peak potentials for the compound was investigated (Table 3,4). The reduction peak currents increased with an increase in sweep rate values from 25 to100 mV/s. The peak potential (*Ep*) shifted to increasingly negative values with varying sweep rate (25 – 100mV/sec). These shifts confirmed the irreversible nature of the reduction process of the investigated compound at the glassy carbon electrode. The reduction peak currents were proportional to the square root of the sweep rate ($v^{1/2}$) (Figure 4), indicating that the electron transfer reaction is diffusion controlled [9].

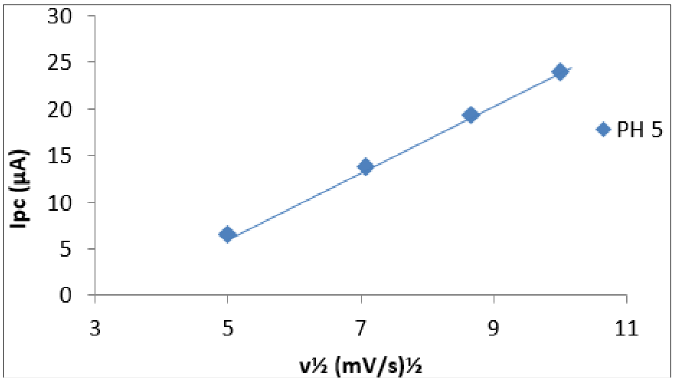


Fig 4: Peak current v/s square root of sweep rate for CBPSG in acetone-BR buffer at pH 5

The effect of pH on the electrochemical behavior of CBPSG Schiff base was analyzed at pH levels of 5.0, 7.0, and 8.2. in acetone-phosphate and BR buffer solutions, as depicted in Figures 2, 3, 5, and 6). All cyclic voltammograms indicate that the parameters of the cathodic peak change with pH. The Epc values depend on the pH of the electrolyte solution, A graph ploting Epc and ln*v* at various pH levels shows a straight line, indicating that cathodic peak potential shifts to more negative values at alkali pH compared to acidic pH. In acetone phosphate buffer, potential changed from -935 mV at pH 5.0 to -1000 mV at pH 8.2, at sweep rate of 50mVs-1. Similar results were also observed at other sweep rates (Tables 3 and 4). These shifts confirm the involvement of protons in the electrode reaction, suggesting that the reduction is easier at low pH in compared to higher pH. This confirms the formation of an easily reducible protonated intermediate during the reduction process [10].

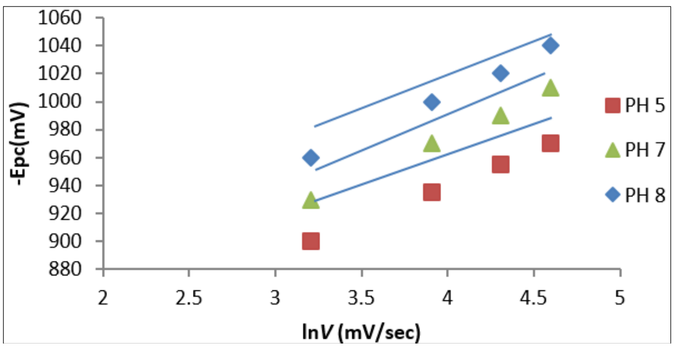


Fig 5: Reduction potential v/s ln*v* for 1mM CBPSG in acetone-phosphate buffer.

The effect of buffers (BR buffer and Phosphate buffer) on the electrochemical process of the CBPSG was also studied. To observe this effect, the cyclic voltammograms of the compound were taken in phosphate buffer and BR buffer with acetone solvent at 25-100 mV/s sweep rate and at three distinct pH values (5, 7, 8.2). This establishes that the peak potential shifts towards more negative potentials with a buffer solution of lower polarity.(Figure.6) The reduction potential value in acetone - phosphate buffer is reported to vary from -900mV to -1040 mV at 25 to 100 mVs-1 sweep rates at three distinct pH values. In acetone-BR buffer, Epc values vary from -930mV to -1070mV at 25- 100 mVs-1 sweep rates, possibly due to lower ionized components in BR buffer [11].

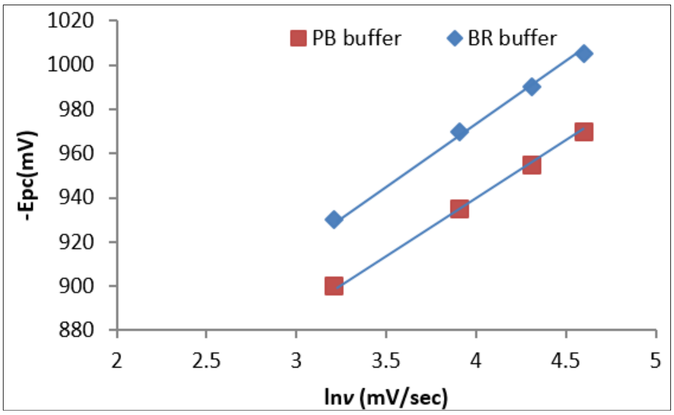


Fig 6: Epc v/s ln v for 1mM CBPSG in acetone-phosphate and acetone-BR buffer at pH 7

Conclusion

In this research paper, Schiff base synthesized from the condensation reaction between 2-chlorobenzophenone and sulfaguanidine and was characterized by spectral techniques. The voltammetric behavior of CBPSG Schiff base is significantly influenced by sweep rate, pH and buffers. The reduction potential is affected by the sweep rate, with peak potentials shifting to more negative values as the sweep rate increases, conforming the irreversible and diffusion-controlled nature of the process. Additionally, the pH of the buffer solution plays a crucial role, with higher pH levels resulting in more negative cathodic peak potentials, highlighting the involvement of protons in the reduction mechanism.

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