

Electrochemical Investigation of 2-Hydroxy-2-Methyl Propiophenone Sulphamethizole Azomethine Compound Derived from Sulpha Drug Employing Cyclic Voltammetry Technique

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Abstract

The cyclic voltammetric behavior of newly synthesized Schiff base 2-Hydroxy-2-methyl Propiophenone sulphamethizole (HMPSMO) was investigated at glassy carbon electrode (GCE) in acetone and methanol solvents using phosphate buffer and Britton-Robinson (BR) buffer within various pH values. Under the experimental conditions the cyclic voltammograms of HMPSMO consist of an irreversible cathodic wave at different sweep rate, that follows a one-step, two-electron transfer, diffusion-controlled electrochemical process which are ascribed to the azomethine group ($-\text{CH}=\text{N}-$) being reduced to the amine group ($-\text{CH}_2-\text{NH}-$). The effects of solvent, buffer, sweep rate and pH are also studied on CV variables. Additionally computed were the kinetic parameters, including the rate constant ($K^0_{f,h}$), diffusion coefficient ($D_0^{1/2}$), and charge-transfer coefficient (α_n).

Keywords: 2-Hydroxy-2-methyl Propiophenone sulphamethizole, Cyclic voltammetry, Glassy carbon electrode, Irreversible, Diffusion-controlled.

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Introduction

The condensation of an aldehydes or ketones with a primary amine, initially discovery by Hugo Schiff in 1864, [1] is the characteristic of Schiff base reactions. One these reactions usually include a nucleophilic addition reaction in which a $\text{C}=\text{N}-\text{R}$ group—where R can be either an aryl or an alkyl group—replaces the carbonyl compound's $\text{C}=\text{O}$ group [2]. Schiff bases are crucial for organic synthesis and have a wide range of uses in material science and health, among other domains [3]. The number of Schiff bases that are prepared from sulpha drugs and their metal complex shows a wide range of biological activities such as antibacterial, antifungal, anticonvulsant, antimalarial, and anticancer [4]. The reported HMPSMO compound also shows biologically effective against the fungi (*Aspergillus niger* & *Penicillium chrysogenum*) as well as the bacteria (*Bacillus subtilis* & *Escherichia coli*), which has been studied earlier [5]. Many workers have also been interested in Schiff bases' electrochemical activity. Nevertheless, the literature has insufficient details about the electrochemical activity of the Schiff bases of sulfa drugs [6]. The advantage of cyclic voltammetry is that it may be used to find out about the kinetics of heterogeneous electron-transfer reactions and coupled chemical interactions along with to the thermodynamics of redox mechanisms [7]. It is a versatile technique involving highly affordable equipment and provides a wide linear dynamic range along with good sensitivity, accuracy, and precision. Quantitative and qualitative methods of cyclic voltammetry can be applied to collect data on non-redox chemical processes that take place either before or after a redox reaction [8]. This study focused on the electrochemical behavior of HMPSMO in order to examine several key factors, including the charge-transfer coefficient (α_n), diffusion coefficient ($D_0^{1/2}$) and rate constant ($K^0_{f,h}$), by adjusting the scan rate, solvent, buffer solution, and pH of the electrochemical solution [9].

Experimental Methods

Chemicals

All chemicals were analytical grade and of the highest available purity, and used without further purification. For the preparation of HMPSMO, 2-Hydroxy-2-methyl Propiophenone and sulphamethizole were obtained from Sigma-Aldrich. For the preparation of phosphate buffer and BR buffer, disodiumhydrogenphosphate (Na_2HPO_4), sodium dihydrogen phosphate (NaH_2PO_4), 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.

Electrochemical Equipment's and Voltammetric Parameters

Cyclic voltammetric measurements were performed using computerized 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. For the purpose of performing 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 Pt wire as an auxiliary electrode were used for electrochemical research. The digital pH meter (Elico) was used to measure the pH of solutions. There are some kinds of significant variables that characterize the CV. The diagnostics invented by Nicholson and Shain for examining the electrochemical response is centered on four of these observables, especially the two peak currents (Epc, Epa) and the two peak potentials (Ipc, Ipa).

Sample Preparation

To prepare the experimental solution with the necessary concentration (1×10^{-3} M), one ml of stock solution (.01 m) of the relevant synthesized Schiff base component was added to nine ml of phosphate buffer or BR buffer of the desired pH solution. The whole solution's volume was maintained at 10 ml. In an electrochemical cell, prepared solutions were placed and deoxygenated with dry nitrogen gas for 15 minutes before being used to record potential current curves and thereafter, used for recording CV curves. Electrochemical experiments of Schiff base compound were recorded at pH ranges of 5.0, 7.0 and 8.2 in phosphate buffer and Britton Robinson (BR) buffer,[10].

Result and Discussions

Cyclic Voltammetric Studies of HMPSMO

In anaerobic conditions, CV observations of synthesized HMPSMO were carried out using a GCE in the potential window -0.7 to +1.7 V at varied scan rates (50,100,150,200,250 mVs⁻¹) and various pH ranges (5.0,7.0,8.2). The cyclic voltammetric data for Schiff base are summarized in Table 1-3 and Fig. 1-3 showed the cyclic voltammograms of HMPSMO in acetone phosphate buffer, methanol phosphate buffer and acetone BR buffer respectively. The potential current curves of the investigated Schiff base showed the irreversible behavior of electrode process [11]. The kinetic parameters have been calculated [12] and detailed in tables 1 to 3.

Table 1: Cyclic voltammetric measurements for 1mM HMPSMO in propanone-phosphate buffer at three distinct pH levels (5, 7, and 8.2).

pH	v(mV/s)	Epc (mV)	Ipc (μA)	Ep/2 (mV)	Ipc/v ^{1/2}	α_n	Do ^{1/2} (cm ² /s)	k ^o f,h (cm/s)
5.0	50	-900	24.5	-810	3.4648	0.5300	29.9585	5.68×10^{-10}
	100	-917	34.8	-820	3.4800	0.4917	31.2379	2.23×10^{-9}
	150	-927	38.1	-833	3.1108	0.5074	27.4891	1.14×10^{-9}
	200	-940	40.5	-850	2.8637	0.5300	24.7616	4.11×10^{-10}
	250	-955	47.7	-853	3.0174	0.4676	27.7752	3.62×10^{-9}
7.0	50	-915	29.4	-820	4.1577	0.5021	36.9353	1.35×10^{-9}
	100	-924	34.2	-835	3.4200	0.5359	29.4062	3.90×10^{-10}
	150	-945	44.7	-849	3.6497	0.4968	32.5923	1.39×10^{-9}
	200	-953	54.1	-862	3.8254	0.5241	33.2599	5.22×10^{-10}
	250	-975	57.1	-879	3.6113	0.4968	32.2492	9.91×10^{-10}
8.2	50	-930	32.8	-841	4.6386	0.5359	39.8842	3.30×10^{-10}
	100	-942	36.1	-845	3.6100	0.4917	32.4049	1.43×10^{-9}
	150	-960	47.1	-870	3.8456	0.5300	33.2517	3.17×10^{-10}
	200	-969	54.8	-877	3.8813	0.5184	33.9304	4.73×10^{-10}
	250	-987	63.7	-889	4.0300	0.4867	36.3610	1.29×10^{-9}

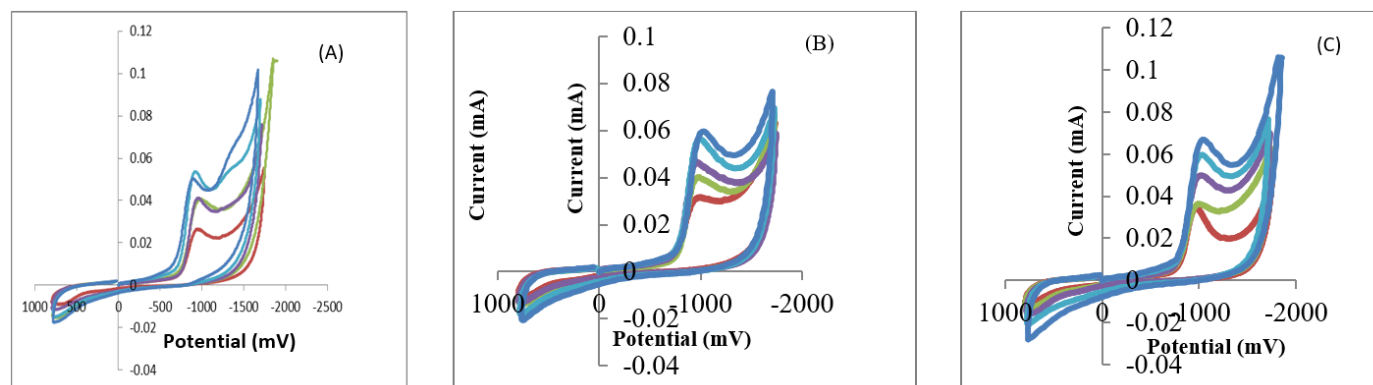


Figure 1: CV of HMPSMO in propanone -phosphate buffer at various pH values 5.0 (A), 7.0 (B) and 8.2(C).

Table 2 : Cyclic voltammetric measurements for 1mM HMPSMO in CH₃OH-phosphate buffer at three distinct pH levels (5.0, 7.0, and 8.2).

pH	ν (mV/s)	E_{pc} (mV)	I_{pc} (μ A)	$E_{p/2}$ (mV)	$I_{pc}/\nu^{1/2}$	α_n	$D_o^{1/2}$ (cm ² /s)	$k_{f,h}^o$ (cm/s)
5.0	50	-910	32.8	-819	4.6386	0.5241	40.3299	7.61×10^{-10}
	100	-924	37.8	-837	3.7800	0.5482	32.1343	2.77×10^{-10}
	150	-936	40.5	-848	3.3141	0.5420	28.3356	2.89×10^{-10}
	200	-942	44.7	-851	3.1607	0.5241	27.4809	5.40×10^{-10}
	250	-962	55.1	-861	3.4848	0.4722	31.9198	3.09×10^{-9}
7.0	50	-923	36.5	-836	5.1675	0.5482	43.9299	2.73×10^{-10}
	100	-940	41.5	-853	4.1570	0.5482	35.3392	2.16×10^{-10}
	150	-949	47.1	-850	3.8456	0.4818	34.8747	2.36×10^{-9}
	200	-957	54.1	-865	3.8311	0.5184	33.4915	5.95×10^{-10}
	250	-967	57.9	-870	3.6631	0.4917	32.8823	1.42×10^{-9}
8.2	50	-937	42.1	-848	5.9538	0.5359	51.1929	3.66×10^{-10}
	100	-946	52.1	-859	5.2100	0.5482	44.2909	2.39×10^{-10}
	150	-965	58.6	-873	4.7846	0.5184	41.8276	5.48×10^{-10}
	200	-976	60.2	-882	4.2603	0.5074	37.6464	6.86×10^{-10}
	250	-1000	61.7	-898	3.9028	0.4676	35.9255	2.06×10^{-9}

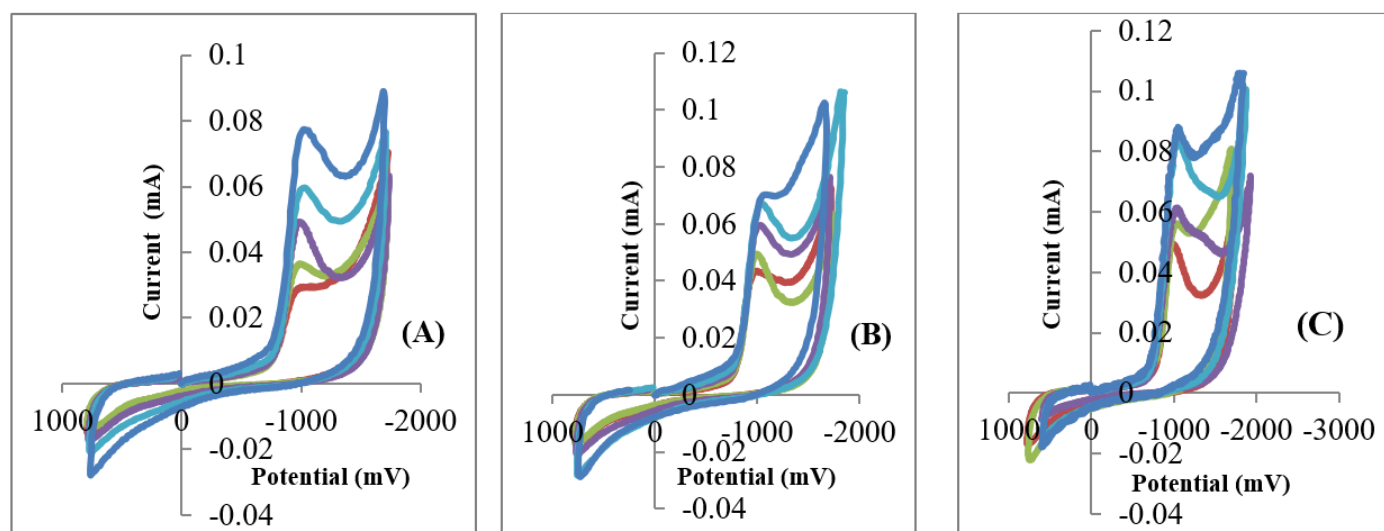


Figure 2 : Cyclic voltammograms of HMPSMO in CH₃OH -phosphate buffer at three various pH values 5.0 (A), 7.0 (B) and 8.2(C).

Figure 1 represents potential current curves at a scan rate 50-250 mVs⁻¹ in acetone phosphate buffer at PH 5.0, 7.0, 8.2 shown in Fig.1A, 1B, 1C respectively. The obtained cyclic voltammograms show one irreversible cathodic peak at all scan rate and at all taken pH medium. The peak potential value swept from -900mV to -987mV and peak current value rises from 24.5 μ A to 63.7 μ A in acidic to basic medium.

Figure 2 represents cyclic voltammograms in methanol phosphate buffer at aforementioned sweep rate and at pH 5.0, 7.0, 8.2 shown in Fig.2A, 2B, 2C respectively. The peak potential value swept from -910mV to -1000 mV and peak current value rises from 32.8 μ A to 61.7 μ A in acidic to basic medium.

Cyclic voltammograms of 1mM HMPSMO in acetone BR buffer at PH 5.0, 7.0, 8.2 are shown in Fig. 3 (A, B and C) respectively. The peak potential value swept from -928 mV to -1031 mV and peak current value rises from 27.3 μ A to 85.2 μ A in aforementioned conditions.

All CV curves shows one electron reduction peak at different scan rates and Epc value shifted to more negative cathodic direction and Ipc value also increases with acidic to basic media at 50-250 mV/s scan rates in different solvent and buffers indicating that the electrode process is irreversible.

Good linear plots of Ipc vs $v^{1/2}$ are obtained that show the reduction of HMPSMO in this medium is diffusion controlled [13] with employed scan rate in propanone-BR buffer at three distinct pH levels (5, 7, and 8.2) shown in Fig. 4.

Table 3: Cyclic voltammetric measurements for 1mM HMPSMO in propanone-BR buffer at three distinct pH levels (5, 7, and 8.2).

pH	v (mV/s)	E_{pc} (mV)	I_{pc} (μ A)	$E_{p/2}$ (mV)	$I_{pc}/v^{1/2}$	α_n	$D_o^{1/2}$ (cm ² /s)	$k_{f,h}^o$ (cm/s)
5.0	50	-928	27.3	-840	3.8608	0.5420	33.0093	2.30×10^{-10}
	100	-950	35.4	-847	3.5400	0.4631	32.7446	3.48×10^{-9}
	150	-965	48.7	-853	3.9771	0.4258	38.3618	1.48×10^{-8}
	200	-987	60.2	-866	4.2567	0.3942	42.6768	4.28×10^{-8}
	250	-993	73.1	-878	4.6245	0.4147	45.1994	2.14×10^{-8}
7.0	50	-947	40.3	-848	5.7091	0.4818	51.7737	2.10×10^{-9}
	100	-967	47.5	-859	4.7570	0.4416	45.0570	7.71×10^{-9}
	150	-986	57.7	-878	4.7128	0.4416	44.6385	6.74×10^{-9}
	200	-1010	63.7	-891	4.5042	0.4008	44.7832	2.45×10^{-8}
	250	-1025	66.9	-900	4.2323	0.3816	43.1279	4.40×10^{-8}
8.2	50	-961	43.7	-861	6.1801	0.4770	56.3267	2.09×10^{-9}
	100	-975	58.9	-868	5.8920	0.4457	55.5485	7.11×10^{-9}
	150	-1009	64.1	-865	4.9087	0.3312	53.6874	3.62×10^{-7}
	200	-1020	78.9	-872	5.5790	0.3222	61.8601	5.89×10^{-7}
	250	-1031	85.2	-880	5.3885	0.3158	60.3498	7.16×10^{-7}

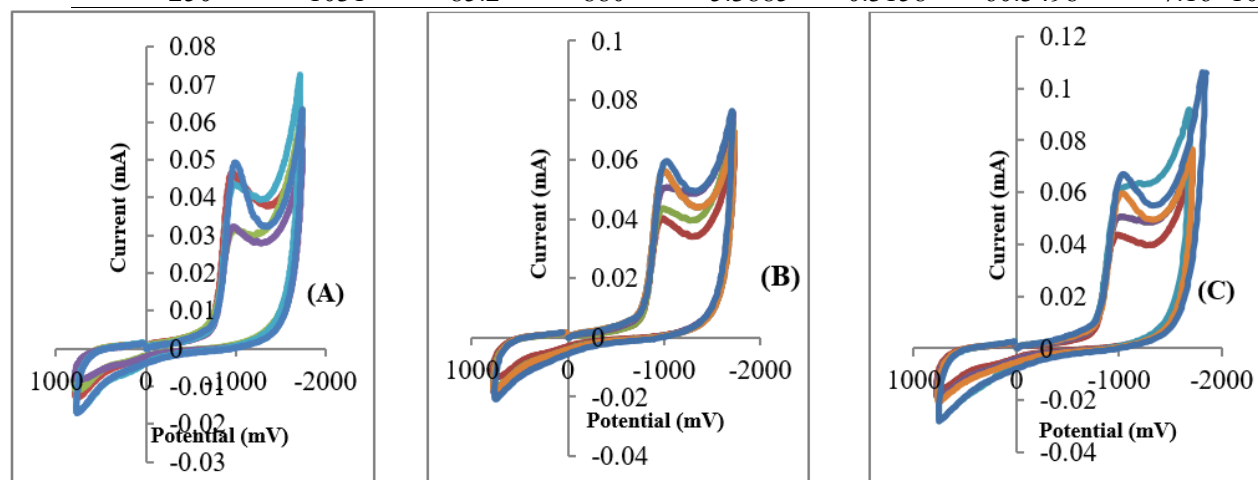


Figure 3: Cyclic voltammograms of HMPSMO in propanone -BR buffer at three various pH values 5.0 (A), 7.0 (B) and 8.2(C).

The shift of cathodic peak potential towards more negative values with the increase in scan rate indicates irreversible nature of the system shown in Fig. 5 [14]. The peak potential of irreversible reaction is given by the following equation.

$$E_p = E_0 - (RT/\alpha n F)[0.78 - \ln(D^{1/2}/K_0) + \ln(\alpha n F v / RT)^{1/2}] \quad (1)$$

Where α is the cathodic charge transfer coefficient, n is the number of electrons involved in the charge transfer step, $D^{1/2}$ the diffusion coefficient K_0 is the rate constant

Peak current for irreversible system is given by Randle Sevcik equation,

$$I_{pc} = (2.99 \times 10^5) n (\alpha n a)^{1/2} A C D_0^{1/2} v^{1/2} \quad (2)$$

Where A is the area of electrode in cm^2 , $D_0^{1/2}$ the diffusion coefficient in cm^2s^{-1} , C the concentration in molL^{-1} and v is in mVs^{-1}

Effect of solvent on cyclic voltammetric parameters

HMPSMO cyclic voltammograms have been performed at pH 5.0, 7.0, and 8.2 in acetone and methanol solvents with phosphate buffer. The electrochemical reduction is more apparent in more polar solvents, as seen by the negative peak potential value, which is highest in methanol solvent and lowest in acetone solvent shown in fig. 6. This pattern is comparable to the solvent's polarity, dielectric constant, and viscosity [15–16].

Effect of buffer on cyclic voltammetric parameters

The electrochemical behavior of HMPSMO was measured in the presence of phosphate buffer and Britton-Robinson buffer (BR) with acetone solvent at sweep speeds ranging from 50 mV/s to 250 mV/s and at various pH values of 5, 7, and 8.2 in order to study the impact of the buffer medium (table 1 and 3). These cyclic voltammetry measurements demonstrate that the cathodic peak potential rapidly approaches a high negative value in the BR buffer solution [17], suggesting that the polarity of the buffer solution influenced the redox species' electrochemical behavior shown in fig.7.

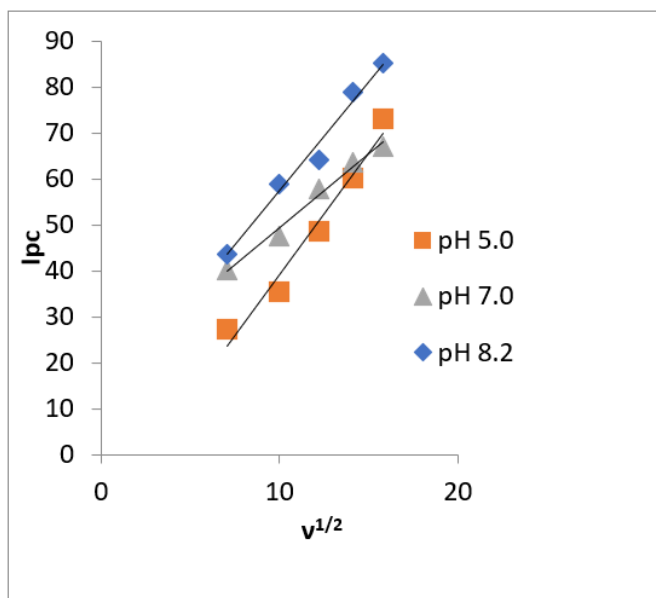


Fig.4 Plot of peak current (I_{pc}) vs square root of the sweep rate ($v^{1/2}$) for 1mM HMPSMO in propanone-BR buffer at three distinct pH levels (5, 7, and 8.2).

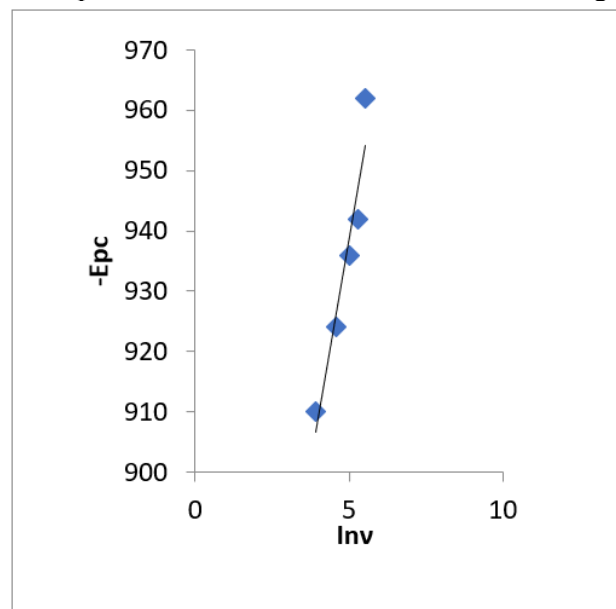


Fig. 5 Plot of E_{pc} vs $\ln v$ for 1mM HMPSMO in CH_3OH -phosphate buffer at 5.0 pH

Conclusion

Electrochemical studies revealed that HMPSMO compound is electro active as it undergoes reduction processes at a glassy carbon electrode. All the cyclic voltammograms are revealing a two-electron irreversible reduction and a diffusion-controlled process. The cyclic voltammetric parameters of HMPSMO were affected by various experimental conditions, including scan rates, pH values, and solvent and buffer mediums. The solvent effect study confirms that the electrochemical reduction is more facial in more polar solvent. Additionally, the cyclic voltammograms shows the polarity of buffer solution affected electrochemical behavior of redox species. These findings provide valuable insights into the electrochemical behavior of HMPSMO compound under different experimental conditions.

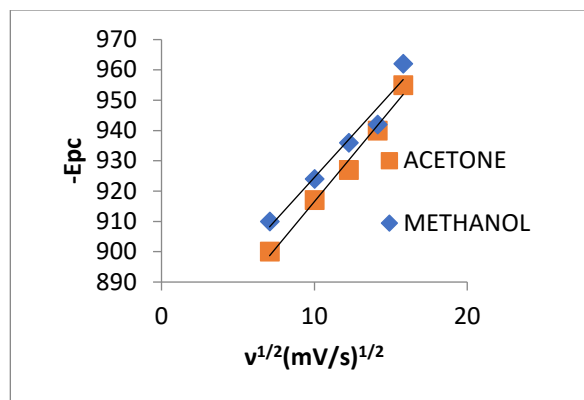


Fig.6: Reduction potential versus $v^{1/2}$ for 1 mM HMPSMO in acetone and methanol solvents at pH 5.0 using phosphate Buffer.

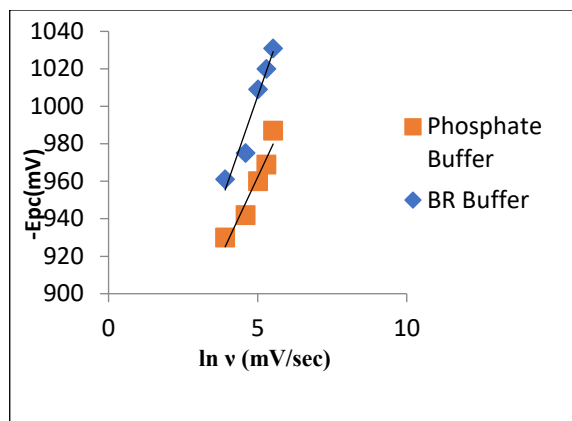


Fig.7 Reduction potential versus $\ln v$ for 1 mM HMPSMO in acetone solvent at pH 8.2 with phosphate and BR buffer

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Conflicts of interest-The author declare no conflicts of interest

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