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Physico-chemical studies of a biologically active semicarbazone from 4-methylbenzophenone

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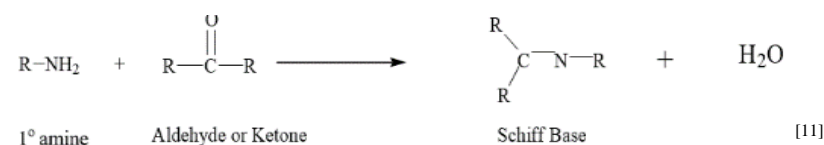
Abstract

The cyclic voltammetric behavior of a biologically active 4-methylbenzophenone semicarbazone (MBSC) was investigated at a glassy carbon electrode in ethanol and DMF solvents using phosphate and Britton-Robinson (BR) buffers. The peak potential and peak current were computed in relation to solvents, buffers, sweep speeds, and pH. The compound is characterized using infrared spectra method. Within the whole pH range, the cyclic voltammograms of MBSC showed irreversible and diffusion-controlled single cathodic peaks. Cyclic voltammetric measurements are used to calculate the kinetic parameters.

Keywords: Cyclic voltammetry, electrochemical studies, kinetic parameters, 4-methylbenzophenone semicarbazone

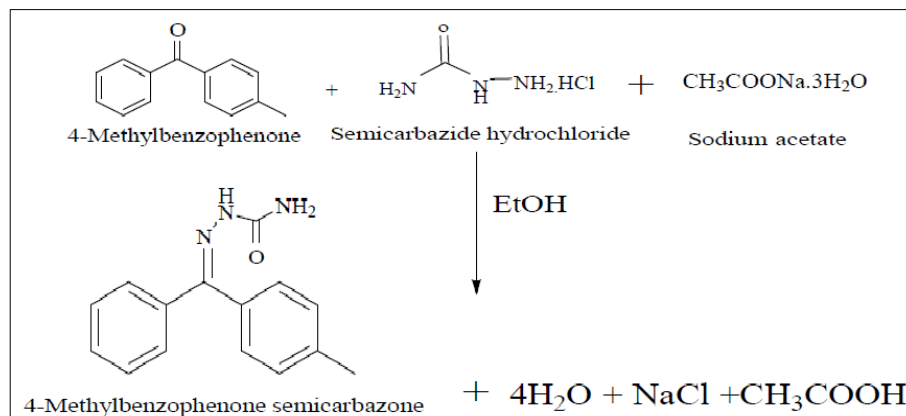
Introduction

Schiff bases are typically formed from the condensation reaction between primary amine with carbonyl compounds either ketone or aldehyde. Schiff bases are an important class of ligands for coordination chemistry, and they coordinate to metal ions via the imine or azomethine nitrogen. Schiff bases have been comprehensively studied in the field of coordination chemistry mainly due to their facile syntheses, easy availability, and interesting electronic properties [1-4]. Moreover, the coordination chemistry of Schiff base has drawn remarkable attention because of their significance in catalysis, analytical chemistry, dye industry and metallurgy [3, 5, 6]. Additionally, the Schiff base's azomethine linkage (-C=N) is an essential structural requirement for biological activities. Several Schiff bases and their transition metal complexes are reported to possess remarkable antibacterial, antifungal, and anticancer activities [7-10].



Various SBs have a wide range of reported uses in a variety of domains, such as analytical, catalysis, organic and inorganic chemistry, pharmaceutical, biological [12], food and pharmacological activities [13]. Such as anti-inflammatory [14], antitumor [15], anticonvulsant [16], antifungal [17], analgesic [18], antibacterial [19], antimalarial [20], antiviral [21], antihelmintic [22], antioxidant [23] and so forth. Semicarbazones are also among Schiff bases. The compound under investigation was synthesized by condensation of 4-methylbenzophenone and semicarbazide hydrochloride and characterized by IR spectra.

Experimental: All chemicals obtained were of analytical grade and were used as received. Ethanol was freshly distilled prior to use. Synthesis: Equimolar concentration (1mM) of semicarbazide and 4-methylbenzophenone was taken in the experimental mixture with sodium acetate in ethanolic medium. The mixture was refluxed for 5 hours and the product obtained was recrystallized using a minimum amount of alcohol. White crystals were obtained with yield 77%.

**Fig 1:** Synthesis of MBSC

A three electrode configuration was used for current-voltage studies, incorporating glassy-carbon (working electrode), Ag/AgCl (reference electrode) and a Pt wire (auxiliary electrode). Glassy carbon electrode was cleaned prior to every voltammetric reading with alumina and doubly-distilled water. A stock solution of MBSC and buffers *viz.*, BR and phosphate buffer were prepared for reaction mixture. The experimental solution was prepared keeping overall volume 10 mL. The mixture was degassed with nitrogen gas to

remove dissolved oxygen. Electrodes were connected to the cell and potential was applied in order to measure current.

Results and Discussion

Elemental and spectral analysis were used to characterize the synthesized compound. The presence of peak at 1625-1633 cm suggests the condensation between ketonic and NH₂ group of semicarbazide and the formation of C=N group.

Sr. No.	Specification	Colour & state	Yield (%)	Elemental Analysis Found (Calculated)		
				C	H	N
1.	4-Methylbenzophenone semicarbazone (MBSC)	White Solid	77	71.13 (71.07)	5.97 (5.94)	16.59 (16.62)

The CV studies were recorded with an initial potential of +1000 mV and final potential of -1600 mV. MBSC exhibited a single cathodic peak in the range -970 mV to -1090 mV in BR buffer ethanolic medium (Table 1). This cathodic peak shifts to -950mV to -1020mV range in BR buffer acetone media (Table 3). This shows more negative peak potential

values in ethanolic media as compared to acetone which can be supported by their similar viscosity trends as viscosity of ethanol at 250C (1.04mPa.s) is higher than that of acetone (0.30mPa.s) as reduction is comparatively tougher in a more viscous medium than a lesser viscous medium.

Table 1: Cyclic Voltammetric parameters of 1mM 4-methylbenzophenonesemicarbazone at different pH levels in ethanol BR-buffer solution

pH	N mVs ⁻¹	E _{pc} mV	I _{pc} μA	Ep/2 mV	I _{pc} /v ^{1/2}	αn	D01/2x10 ³ cm ² s ⁻¹	k ^o f, h cm.s ⁻¹
5	25	-910	8.1	-800	1.14	0.4336	7.7431	3.28E-09
	50	-950	10.4	-820	1.04	0.3669	7.6422	2.53E-08
	75	-990	11.0	-850	0.89	0.3407	6.8489	4.15E-08
	100	-1000	11.3	-860	0.80	0.3407	6.0931	2.64E-08
	125	-1010	15.3	-870	0.96	0.3512	7.2678	2.07E-08
7	25	-890	20.2	-810	2.85	0.5962	16.4676	2.89E-11
	50	-950	22.4	-830	2.24	0.3975	15.8140	1.24E-08
	75	-960	24.5	-860	2.00	0.4771	12.8907	5.93E-10
	100	-1010	27.2	-880	1.92	0.4336	13.0008	2.02E-09
	125	-1010	29.1	-890	1.84	0.3975	12.9932	6.37E-09
8.0	25	-970	10.2	-820	1.44	0.3180	11.3858	8.36E-08
	50	-1000	14.2	-840	1.42	0.2981	11.5762	1.74E-07
	75	-1030	16.7	-860	1.36	0.2805	11.9476	2.63E-07
	100	-1080	21.9	-880	1.54	0.2385	14.1139	5.44E-07
	125	-1090	23.5	-900	1.49	0.2650	12.8510	3.28E-08

Table 2: Cyclic Voltammetric parameters of 1mM 4-methylbenzophenonesemicarbazone at varying pH in ethanol phosphate buffer solution

pH	N mVs ⁻¹	E _{pc} mV	I _{pc} μA	Ep/2 mV	I _{pc} /v ^{1/2}	αn	D01/2x10 ³ cm ² s ⁻¹	k ^o f, h cm.s ⁻¹
5	25	-910	11.5	-840	2.3	0.6814	8.7648	5.05E-13
	50	-940	17.3	-850	2.44	0.5300	10.4788	8.68E-11
	75	-960	22.7	-870	2.62	0.5400	11.2265	5.23E-11
	100	-970	29.1	-870	2.91	0.4770	13.2614	5.88E-10
	125	-975	36.1	-885	3.32	0.4147	15.7809	4.73E-09
7	25	-910	14.8	-820	2.96	0.5300	22.2214	2.42E-10

	50	-940	25.7	-840	3.63	0.4770	25.5210	1.40E-09
	75	-960	39.6	-860	4.57	0.4705	22.0939	1.29E-09
	100	-970	41.7	-870	4.17	0.4770	22.3297	1.23E-09
	125	-980	49.0	-890	4.38	0.5300	13.9592	8.00E-11
8.0	25	-920	28.8	-830	5.76	0.5300	24.9018	2.20E-10
	50	-950	36.0	-840	5.09	0.4336	24.3343	5.25E-09
	75	-960	40.2	-860	4.64	0.4770	21.1535	9.77E-10
	100	-970	48.2	-875	4.82	0.5021	21.4091	3.77E-10
	125	-980	54.8	-900	4.90	0.5962	19.9791	9.70E-12

Table 3: Cyclic Voltammetric parameters of 1mM 4-methylbenzophenonesemicarbazone in acetone BR-buffer solution of pH 8 level

pH	N mVs ⁻¹	E _{pc} mV	I _{pc} μA	E _{p/2} mV	I _{pc} /v ^{1/2}	αn	D ₀₁ /2x10 ³ cm ² s ⁻¹	k ⁰ f, h cm.s ⁻¹
8.0	25	-950	17.4	-820	3.48	0.5300	15.0448	8.53E-10
	50	-970	33.5	-840	4.73	0.3669	24.6168	4.33E-08
	75	-980	34.2	-860	3.94	0.3975	19.7139	1.19E-08
	100	-990	36.0	-870	3.60	0.3975	17.9713	1.07E-08
	125	-1020	49.3	-890	4.41	0.4336	21.0763	3.09E-09

Cyclic voltammograms

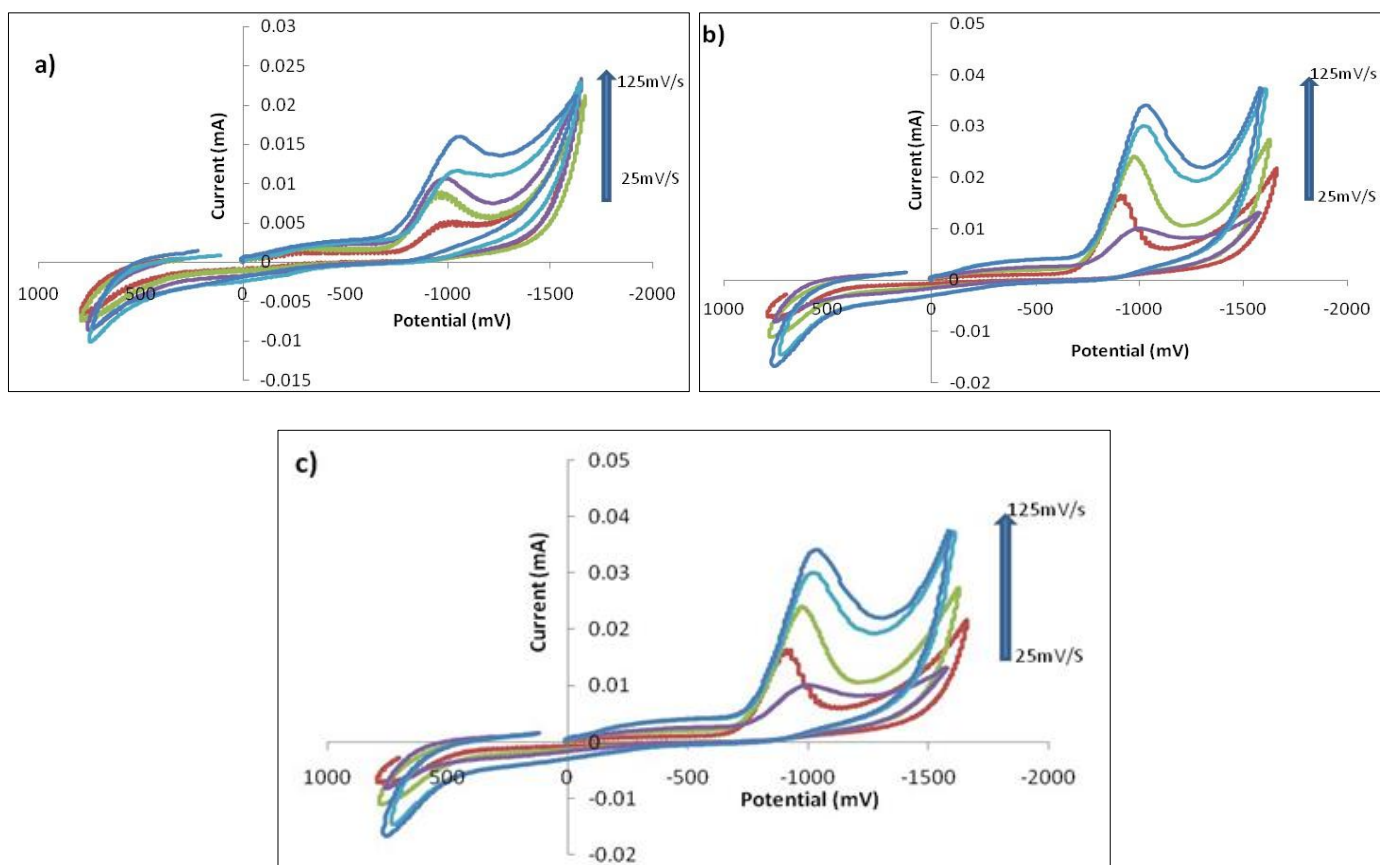
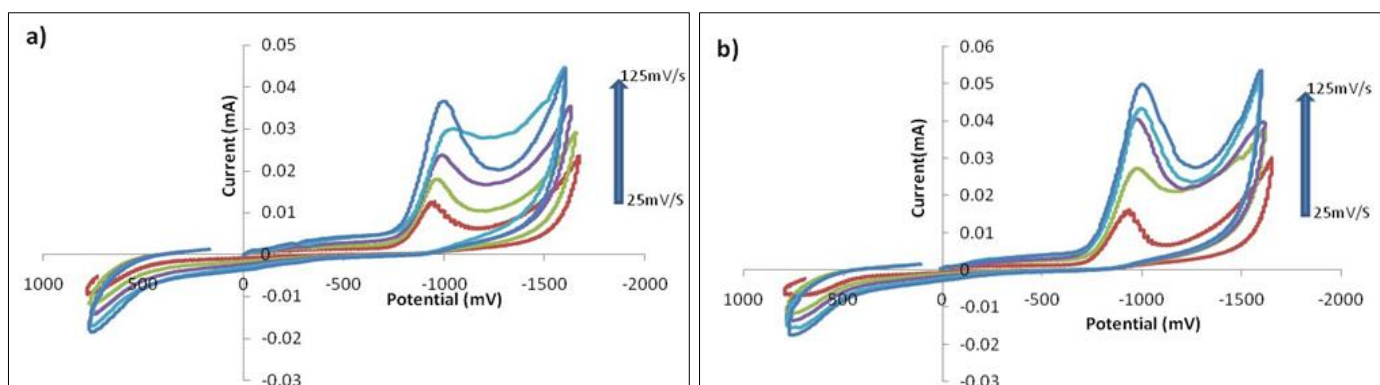


Fig 2: Cyclic voltammograms of MBSC in Ethanol- BR buffer, (a) pH5, (b) pH7 (c) pH8



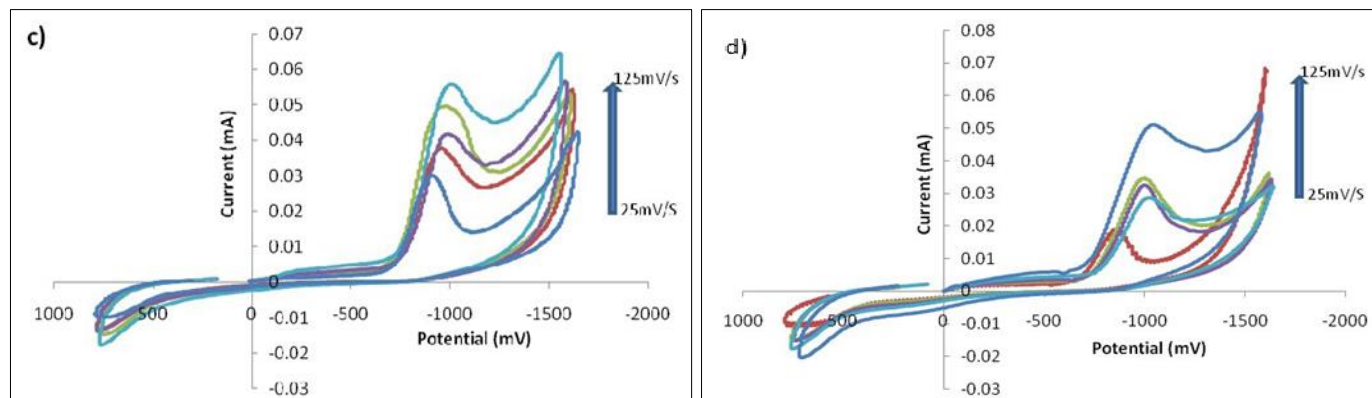


Fig 3: Cyclic voltammogram of MBSC in Acetone - BR buffer at pH8

The effect of pH can be observed when cyclic voltammetric studies were done in various pH medium *viz.*, acidic (pH5), neutral (pH7) and basic (pH8) since on increasing pH, peak potential shifted to more negative side, as observed in tables 1 and 2. The CV study was executed at different pH ranges and sweep potentials. As seen in table 1, at a constant sweep rate say 100mV/s, peak potentials were -1000, -1010 and -1080mV at a pH of 5(acidic), 7(neutral) and 8(basic) respectively. The peak potential dependence on pH indicates a involvement of proton transfer during reduction of compound. It was found that reduction was easier in acidic medium than in neutral or basic media which suggests that a high concentration of protons favours reduction process. This might be inferred due to easy reducible protonated intermediate. The similar results can be observed in table 2 in different buffer media.

An effect of buffer media can also be seen in tables 1 and 2 as the peak potential was more negative in BR buffer as compared to phosphate buffer. From table1 (BR buffer) the peak potentials at a constant pH 8 are -970,-1000,-1030,-1080,-1090mV at different scan rates, which were more negative as compared to table2 (Phosphate buffer) values at similar scan rates -920,- 950,-960,-970,-980 mV. The variation can also be seen in figure-1(c) and figure-3. This can be concluded due to high polarity of phosphate buffer medium, which leads to an easy reduction in this buffer media. Similar results were observed at varying pH in the reaction mixture.

From tables 1-3, an effect of scan rate on peak potential can also be seen, as in table-2, at pH5, Epc values increases negatively from -910mV to -975mV as the scan rate increases from 25mVs-1 to 125mVs-1. A similar trend was also found at all pH levels and in different media. An irreversible reduction is concluded from the observed data and irreversibility increases as scanrate increases which tends to follow Nicholson theory^[24].

Conclusion

The cyclic voltammetric study of 4-methylbenzophenone semicarbazone (MBSC) at a glassy carbon electrode demonstrated significant insights into its electrochemical behavior across various solvents and buffer systems. The analysis revealed that MBSC exhibits an irreversible and diffusion-controlled single cathodic peak, indicating a straightforward reduction process.

The peak potential values shifted more negatively with increasing pH, suggesting that proton transfer plays a crucial

role in the reduction mechanism. This behavior is consistent across both Britton-Robinson and phosphate buffer systems, with the BR buffer facilitating more negative peak potentials, likely due to its higher polarity. Additionally, the variation in peak currents and potentials with differing scan rates and pH levels highlighted the kinetic parameters, providing a comprehensive understanding of the electrochemical properties of MBSC.

The synthesis and characterization of MBSC were successfully confirmed through infrared spectroscopy, supporting the presence of the key azomethine (-C=N-) functional group. The findings underscore the potential applications of MBSC in coordination chemistry and its relevance in biological systems, paving the way for further studies into its pharmacological activities and interactions with metal ions.

Overall, this research enhances the understanding of the electrochemical characteristics of Schiff base derivatives, particularly in their role as biologically active compounds, and sets a foundation for future explorations in electrochemical sensors and catalysis.

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References

1. Kumawat GL, Choudhary P, Varshney AK, Varshney S. Cyclic voltammetric studies of biologically active azomethine 2'-hydroxyacetophenone sulfamethoxazole. *Orient J Chem.* 2019;35(3):1117.
2. El-Ghamry MA, Elzawawi FM, Aziz AA, Nassir KM, Abu-El-Wafa SM. New Schiff base ligand and its novel Cr (III), Mn (II), Co (II), Ni (II), Cu (II), Zn (II) complexes: Spectral investigation, biological applications, and semiconducting properties. *Sci Rep.* 2022;12(1):17942.
3. Bharathi M, Indira S, Vinoth G, Shanmuga Bharathi K. Implanted mixed ligand Ni complex of phenolic Schiff base and 2,2'-bipyridine on MCM-41 as an efficient catalyst for Suzuki-Miyaura cross-coupling reactions: A greener approach. *Res Chem Intermed.* 2022;48(9):3701-3719.
4. Nantapon T, Naweephattana P, Surawatanawong P, Saetear P, Chantarojsiri T, Ruangsapapichat N, *et al.*

- Amino-coumarin-based colorimetric and fluorescent chemosensors capable of discriminating Co^{2+} , Ni^{2+} , and Cu^{2+} ions in solution and potential utilization as a paper-based device. *Spectrochim Acta A Mol Biomol Spectrosc.* 2022;282:121662.
- Eltaweil AS, Hashem OA, Abdel-Hamid H, El-Monaem EMA, Ayoup MS. Synthesis of a new magnetic sulfacetamide-ethylacetoacetate hydrazone-chitosan Schiff base for Cr(VI) removal. *Int. J Biol. Macromol.* 2022;222:1465-1475.
 - Akl MA, Mahdy NAE, Sayed E, Gharkawy RHE. Design, structural, spectral, DFT and analytical studies of novel nano-palladium Schiff base complex. *Sci Rep.* 2022;12:01-15.
 - Abd-Elzaher MM, Labib AA, Mousa HA, Moustafa SA, Ali SA, El-Rashedy AA. Synthesis, anticancer activity and molecular docking study of Schiff base complexes containing thiazole moiety. *Beni-Suef Univ J Basic Appl Sci.* 2016;5:85-96.
 - Chaudhary NK, Mishra P. Metal complexes of a novel Schiff base based on penicillin: Characterization, molecular modeling, and antibacterial activity study. *Bioinorg Chem.* 2017;1-13:142.
 - Al-Zaidi BH, Hasson MM, Ismail AH. New complexes of chelating Schiff base: Synthesis, spectral investigation, antimicrobial, and thermal behavior studies. *J Appl Pharm Sci.* 2019;9(4):45-57.
 - Ferretti V, Matos CP, Canelas C, Tomaz I, Correia I, Le IE, *et al.* New ternary Fe(III)-8-hydroxyquinoline-reduced Schiff base complexes as selective anticancer drug candidates. *J Inorg Biochem.* 2022;236:111961.
 - Al Zoubi W, Al-Hamdani AAS, Kaseem M. Synthesis and antioxidant activities of Schiff bases and their complexes: A review. *Appl Organomet Chem.* 2016;30:810-817.
 - Parvarinezhad S, Ramezanipoor S, Kubicki M, Salehi M. Zn(II), Mn(III), and Co(III) complexes of Schiff base derived from 2-hydroxy-1-naphthaldehyde: synthesis, spectral surveys, single crystal structure studies, Hirshfeld surface analysis, density functional theory calculation, molecular electrostatic potential, and molecular docking evaluations. *Appl Organomet Chem.* 2024, 38(6).
 - Ejidike IP, Ajibade PA. Transition metal complexes of symmetrical and asymmetrical Schiff bases as antibacterial, antifungal, antioxidant, and anticancer agents: Progress and prospects. *Rev. Inorg. Chem.* 2015;35:191-224.
 - Hanif M, Hassan M, Rafiq M, Abbas Q, Ishaq A, Shahzadi S, *et al.* Microwave-assisted synthesis, *in vivo* anti-inflammatory and *in vitro* anti-oxidant activities, and molecular docking study of new substituted Schiff base derivatives. *Pharm Chem J.* 2015;52:424-437.
 - Wilkinson SM, Sheedy TM, New EJ. Synthesis and characterization of metal complexes with Schiff base ligands. *J Chem Educ.* 2016;93(2):351-354.
 - Ali SM, Azad MA, Jesmin M, Ahsan S, Rahman MM, Khanam JA, *et al.* *In vivo* anticancer activity of vanillin semicarbazone. *Asian Pac J Trop Biomed.* 2012;2(6):438-442.
 - Biswas A, Das LK, Drew MG, Aromí G, Gamez P, Ghosh A, *et al.* Synthesis, crystal structures, magnetic properties and catecholase activity of double phenoxido-bridged penta-coordinated dinuclear nickel (II) complexes derived from reduced Schiff-base ligands: Mechanistic inference of catecholase activity. *Inorg Chem.* 2012;51(15):7993-8001.
 - Fareed G, Rizwani GH, Ahmed M, Versiani MA, Fareed N. Schiff bases derived from 1-aminoanthraquinone: A new class of analgesic compounds. *Pak J Sci Ind Res.* 2012;60(3):122-127.
 - Jana S, Dalapati S, Guchhait N. Proton transfer assisted charge transfer phenomena in photochromic Schiff bases and effect of $-\text{NEt}_2$ groups to the Anil Schiff bases. *J Phys Chem.* 2012;116:10948-10958.
 - Facchinetti V, da R Reis R, Gomes CRB, Vasconcelos TRA. Chemistry and biological activities of 1,3-benzothiazoles. *Mini Rev Org Chem.* 2012;9(1):44-53.
 - Chow MJ, Licon C, Yuan Qiang Wong D, Pastorin G, Gaidon C, Ang WH, *et al.* Discovery and investigation of anticancer ruthenium-arene Schiff-base complexes via water-promoted combinatorial three component assembly. *J Med Chem.* 2014;57:6043-6059.
 - Khan SA, Asiri AM, Al-Amry K, Malik MA. Synthesis, characterization, electrochemical studies, and *in vitro* antibacterial activity of novel thiosemicarbazone and its Cu(II), Ni(II), and Co(II) complexes. *Sci World J.* 2014;2014:1-9.
 - Kalaiarasi G, Dharani S, Puschmann H, Prabhakaran R. Synthesis, structural characterization, DNA/protein binding and antioxidant activities of binuclear Ni(II) complexes containing ONS chelating ligands bridged by 1,3-bis(diphenylphosphino)propane. *Inorg Chem Commun.* 2018;97:34-38.
 - Nicholson RS. Theory and application of cyclic voltammetry for measurement of electrode reaction kinetics. *Anal Chem.* 1965;37(11):1351-1355.