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Innovation The Research Concept

Electrochemical Reduction of Carbonyl Compound at Stainless Steel Electrode at different pH

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Abstract

Electrochemical reduction of propiophenone has been studied with the help of cyclic voltammetry at various scan rates and pH value in aqueous alcoholic medium. All cyclic voltammogram recorded exhibit one irreversible cathodic wave. The electrochemical reduction of propiophenone was carried out galvanostatically using economically viable stainless steel (SS-316) electrode at constant current. The product was isolated purified and characterized by combined application of chromatographic and spectroscopic techniques.

Keywords: Electrochemical Reduction, Propiophenone, Stainless Steel (SS-316) Electrode, Cyclic Valtammetry.

Introduction

Electrochemistry has been widely used in industry in effluent treatment, corrosion prevention and electroplating as well as in electrochemical synthesis. Electro-organic synthesis is now a well established technique for synthesize the desired compound by oxidation or reduction of substrates. Here electron obtained during electrochemical reaction play an important role by acting as a reagent.

The carbonyl group is an electrophoric group offering interesting synthetic possibilities. The reduction of a large no. of organic compounds including carbonyl compounds has been carried out electro chemically using stainless steel electrode in aqueous media at controlled potential and controlled current.

In the present work the electrochemical reduction of propiophenone is described. The reduction potential of the reactant was recorded by polarographic techniques. cyclic voltammetry was used to decide the reversibility of the process. Different natures of cyclic voltagrams were obtained in different medium (Acidic, Basic and Neutral) This indicates that in different media different electrolysis products were obtained.

Aim of the Study

The present investigation is specific to only basic medium because the corresponding alcohol is obtained only in higher PH. secondly the SS-316 electrode a economically viable and ecofriendly electrode can be used under these conditions.

Experiment

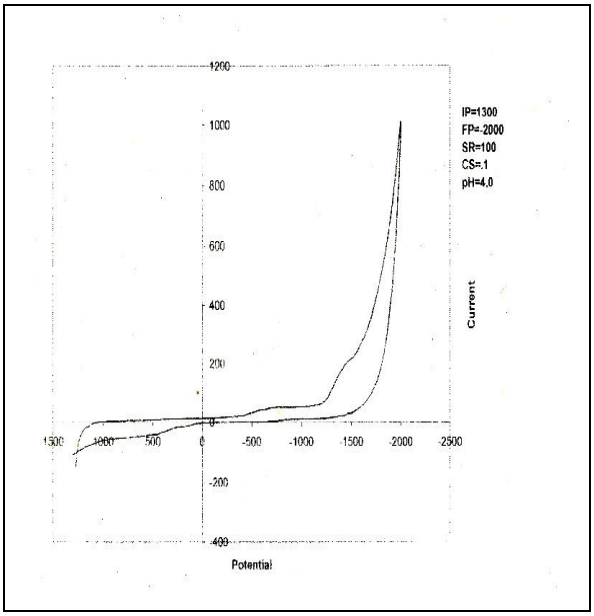
All the used reagents NaoH, CH_3COONa, KCl, propiophenone etc. were of AR grade. The solutions were prepared in double distilled water.

Cyclic voltammograms were obtained on a fully computer controlled basic electrochemistry system ECDA 001, using 3 electrode cell assembly with 1 mm diameter glassy carbon as warking electrode, AglAgcl as reference electrode and pt wire a counter electrode. Cyclic voltammetric studies of propiophenone was carried out in alcoholic media using 1 M KCl as supporting electrolyte , .001 M reactant and BR buffer at different PH (4.0,7.0,9.0) at platinum electrode. The optimum conditions for bulk electrolysis were decided by cyclic voltammetry results and the same were applied for reduction. Using galvanostat at stainless steel electrode (SS-316).

The conventional H-Type cell with two limbs separated by G-4 Disk was used for electrolysis. The supporting electrolyte (1M) sodium acetate was filled in both the limbs. The reactant (.001 M) was dissolved in minimum amounts of alcohol and placed in cathodic chamber and the PH of cathodic solution was maintained at 9.0. The stainless steel electrode was used cathode as well anode. Although in this reaction electrode gets corroded but since the electrodes are economically viable and they were used as sacrificial cathode. The constant potential electrolysis was done for

6 hours with the help of CDPE (Centre for development of physical education, University of Rajasthan, India) make galvanostate.

There after the working up of the reaction mixture involved extracting the aqueous solution with diethyl ether (3 x 25 Ml) The ether layer was then separated and washed with aqueous saturated NaCl solution. The organic extracted were dried over anhydrous Na_2So_4 and then characterized.



Result and Discursion

The voltammographic carves of 0.1 M propiophenone in aqueous medium, 1M Kcl as supporting electrolyte and BR buffer (PH=4, 7 and 9) at glassy carbon electrode using AglAgcl as reference electrode are recorded.

Cyclic voltammograms were recorded with an initial potential Ei 1300 Mv and final (Switching) potential Es of – 2000 Mv at different PH and at same scan rate I.E. 100 Mv in aqueous medium at different PH cyclic voltammograms recorded are shown below.

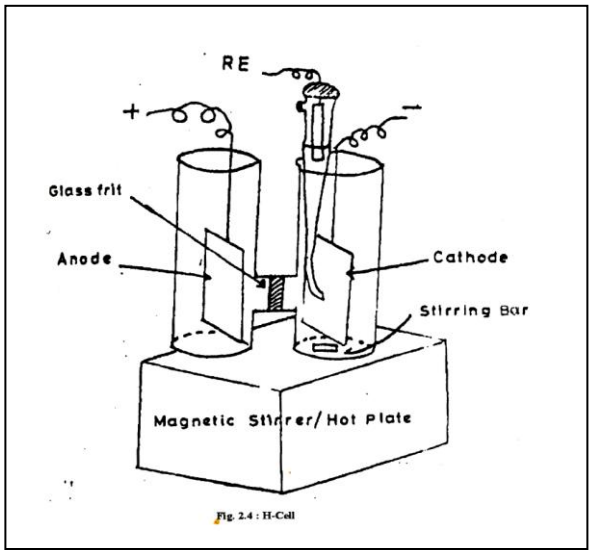
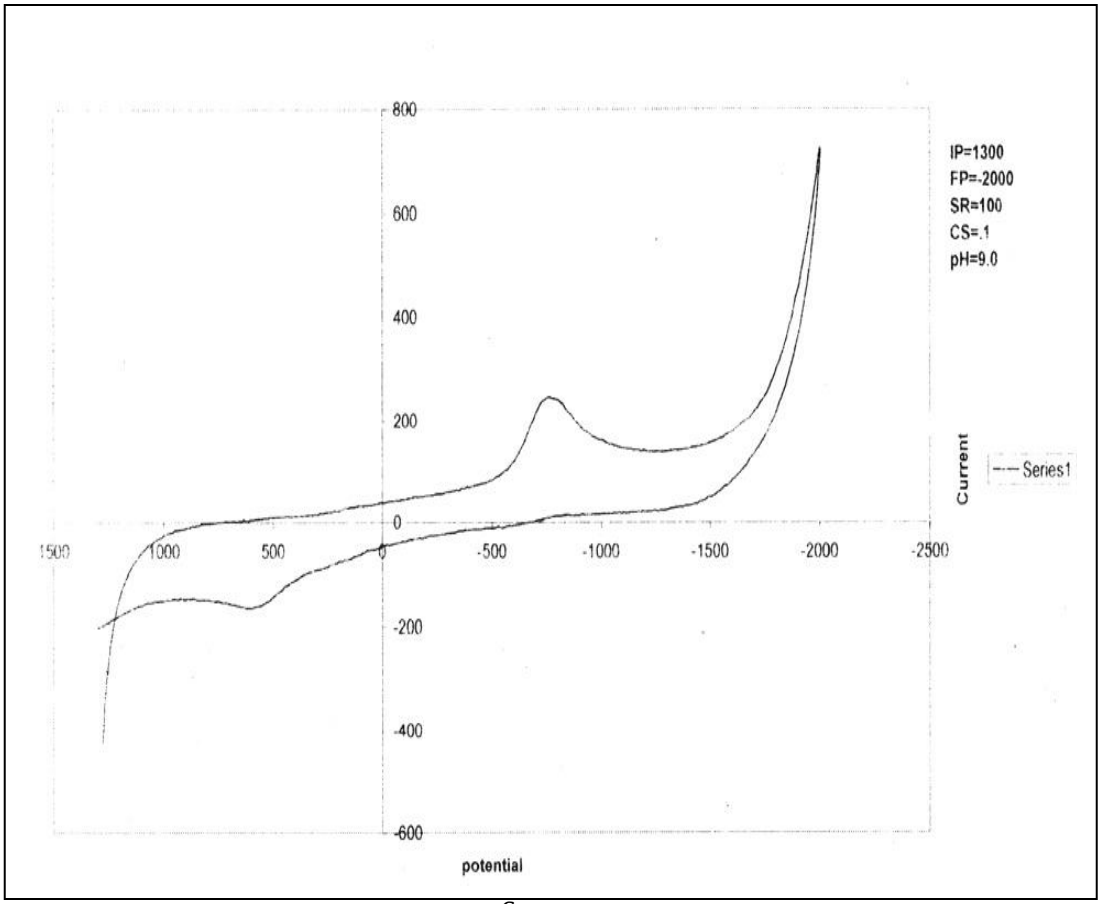
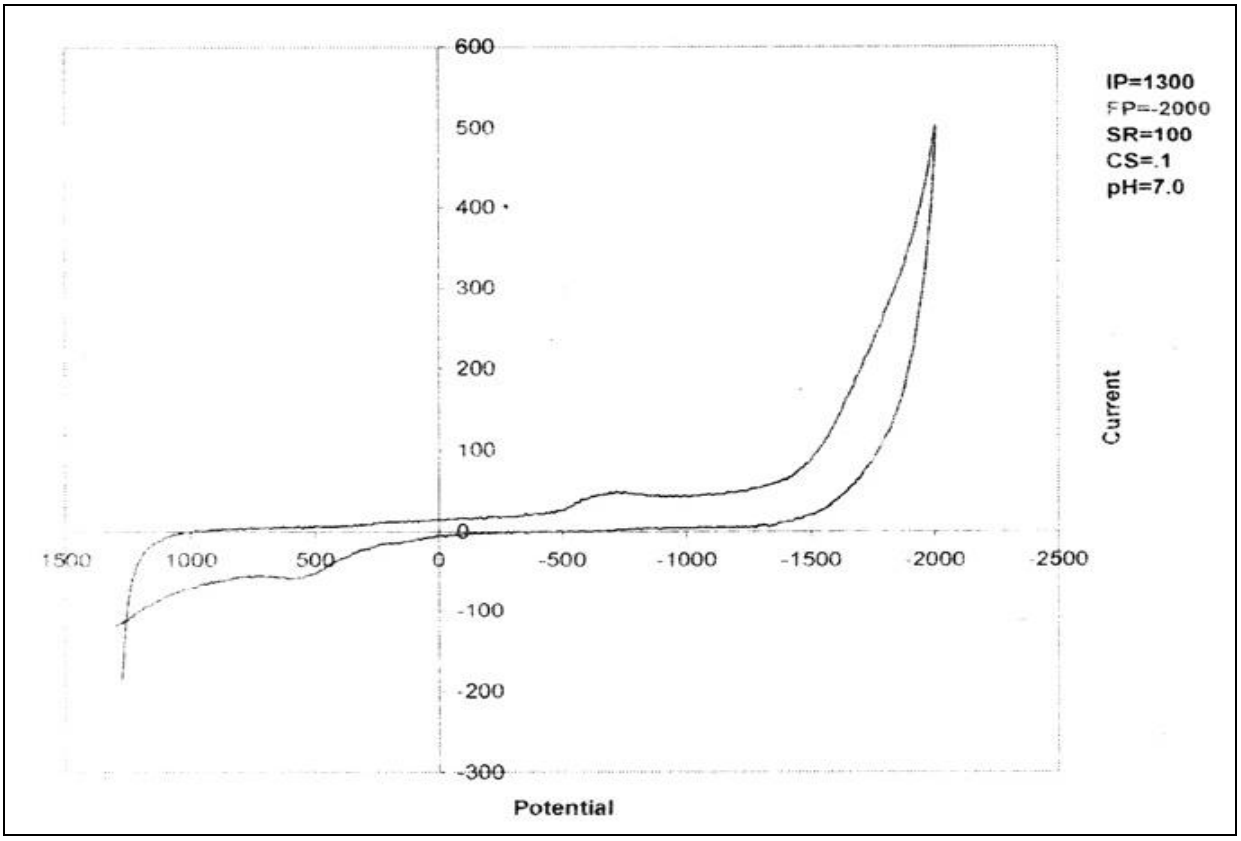


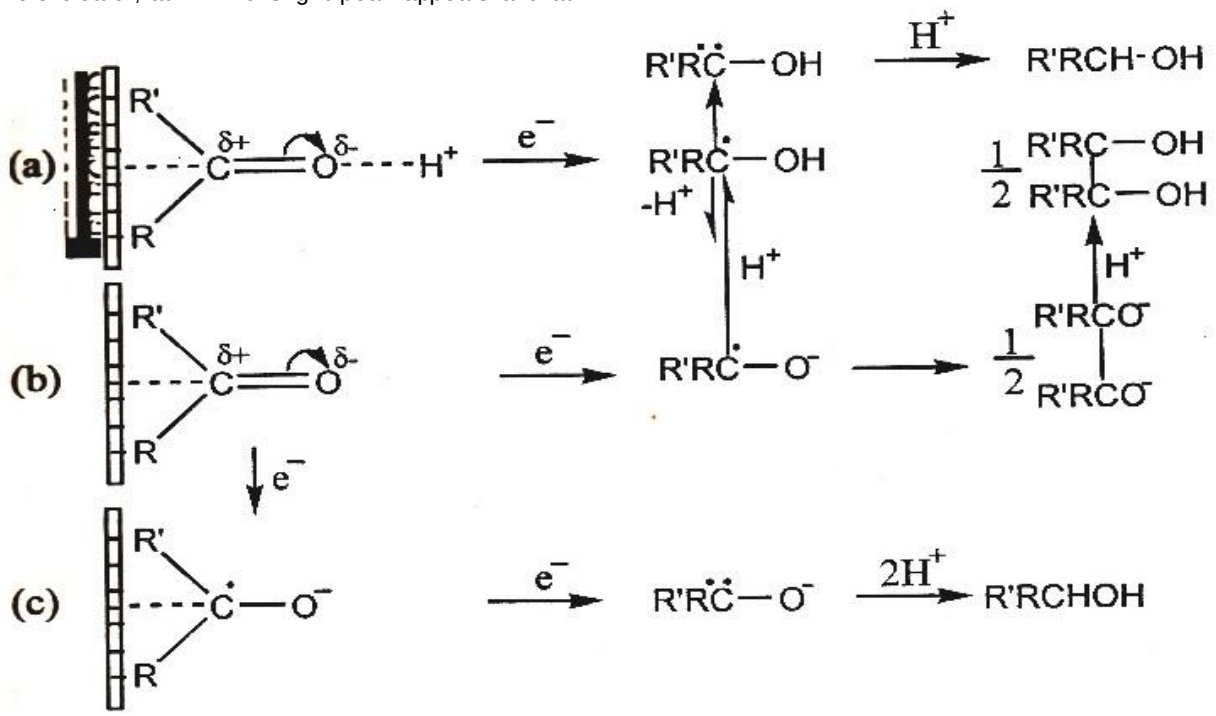
Fig. 2.4 : H-Cell





From above cyclic voltammograms we can concluded that at lower PH i.e. at PH 4.0 there is less appreareance of peak in cyclic voltammogram.

As PH increases a cathodic peak begins to appear & with increasing PH appreance becomes more clearer, at PH 7.0 Slight peak appears and at PH 9.0 peak shows prominent appearance. From above cyclic voltammetric studies it can be concluded that the process of reduction is easier in basic media as compare to acidic and neutral media. The possible mechanism [7] of the reaction as follow:-



Electrochemical reduction of propiophenone yielded under the above stated condition product 1-Phenyl 1- Propanol was obtained in good yield. The single spot TLC checked the purity of compounds and further confirmed by spectroscopic analysis. The results are tabulated as below –

Product Name	B.P (^{o}C)	IR value (cm^{-1})	Mass (m/z)	NMR (svalue)
1- Phenyl-1-propanol	219^{0}C	3410cm^{-1} 1619cm^{-1} 1610cm^{-1} 1035cm^{-1} 3100cm^{-1} 2840cm^{-1}	136 107 79 77 51 29	6.80-7.30 (m,5H) 3.5(t,1H) 4.5(s,1H) 1.7(m,1H) 1.5(m,1H) 1.2(t,3H)

Conclusion

The electrochemical reduction of carbonyl compounds at constant current provides an alternative synthetic route for synthesis of hydroxyl compounds. the product used in industry as a heat transfer medium in the manufacture of perfumes and medicines.

Reference

1. *T. Shano, "Electro-organic Chemistry as a new Tool in Organic Synthesis", Springer, New York (1984).*
2. *S. Tori, "Electro-Organlanic Synthesis", Kodansha, Tokyo (1985).*
3. *W.Barik, J.L. Han, K.C. Lee, N.H. Lee, B.H. Kim and J.T. Hahn, Tetrahedron Lett., 1994, 35, 3965.*
4. *W. Baik, J.U. Rhee, S. H. Lee, N.H. Lee, B.H. Kim and K.S. Tetrahedron Lett., 1995, 36, 2793.*
5. *W. Baik and T.H. Park, J. Org. Chem., 1995, 60, 5683.*
6. *Malik R., Sharma I.K. and Verma P.S. (1999) : Electrochemical reduction of o-nitrotoluene at Stainless Steel (SS-316) cathode in Basic Aqueous Methanol Medium.Bulletin of Electrochemistry 15(12), 529-30.*
7. *Yadav S.R., Yadav R., Sharma, A., Sharma I.K. and Verma P.S.(2002a) : Electrochemical reduction of o-Aminoacetophenone and acetophenone at stainless and cathode in basic aqueous ethanol medium. Bull. Electrochem. 18(2), 87-90.*
8. *Yadav S.R., Goyal, P., Sharma A., Sharma I.K and Verma P.S. (2002b): Electrochemical reduction of Benzil and Salicylaldehyde at Stainless Steel Cathode in Basic Aqueous Ethanol Medium. J. Ind. Chem. Soc. 79, 695-97.*
9. *Zuman P. and Rupp E. (1995) : Polarographic and Electrochemical Studies of Some Aromatic Nitro Compounds. Electroanalysis, 7, 132-137.*