International Journal of Scientific Research in Science and Technology
Print ISSN: 2395-6011 | Online ISSN: 2395-602X (www.ijsrst.com)
doi : https://doi.org/10.32628/IJSRST

Cyclic Voltammetric Behaviour of 4-Nitroacetanilide at Glassy Carbon Electrode at different pH

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Article Info
Volume 9, Issue 1
Page Number : 499-503

Publication Issue
January-February-2022

Article History
Accepted : 01 Jan 2022
Published : 20 Jan 2022

ABSTRACT

The cyclic voltammetric behaviour of 4- nitro acetanilide has been studied with the help of cyclic voltammetry technique at various scan rates and pH values in aqueous methanol solvent. The voltammograms of studied compound exhibits the one reduction peak. The effect of scan rate on cathodic peak potential indicates that these reactions are irreversible. The constant current electrolysis of 4-nitroacetanilide in acidic media was carried out galvanostatically using economically viable stainless steel (SS-316). The product was isolated purified and characterized by combined application of chromatographic and spectroscopic techniques.

Keywords : Cyclic voltammetry, 4- nitro acetanilide, Irreversible, Stainless steel (SS-316) electrode

I. 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.

Numerous investigations have been made on the reduction of aromatic nitro compounds and number of different products has been obtained under various conditions. This is caused more by differences in the chemical follow-up reactions rather than by the electrochemical reactions. It is well known that the reduction of the nitro compounds has played a vital role in the development of organic-electrochemistry.

The present work deals with the electrochemical studies including cyclic voltammetry and constant current electrolysis at stainless steel (SS-316) electrode of 4-nitroacetanilide in various media. Cyclic voltammetry was used to decide the reversibility of the process. Different natures of cyclic voltagramms were obtained in different medium (Acidic, Basic and Neutral) this indicates that in different media different electrolysis products were obtained. Constant current electrolysis at (SS-316) electrode, 4-nitroacetanilide gave amino product in acidic medium. The SS-316 electrode an economically viable and ecofriendly electrode has been used successfully in our lab.

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II. MATERIAL AND METHODS

Experiment:- All the used reagents NaOH, CH3COONa, 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 working electrode, Ag/AgCl as reference electrode and Pt wire a counter electrode. Cyclic voltammetric studies of 4- nitro acetanilide was carried out in alcoholic media using 1 M KCl as supporting electrolyte , .001 M reactant and BR buffer at different PH (5.0,7.0,9.0) at platinum electrode. The optimum conditions for bulk electrolysis were decided by cyclic voltammetry results and the same were applied for reduction by using galvanostate 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 5.0. 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 Na2So4 and then characterized by spectroscopic techniques.

Result and Discussion-

The voltammographic curves of 0.1 M 4- nitro acetanilide in aqueous medium, 1M KCl as supporting electrolyte and BR buffer (PH=5, 7 and 9) at glassy carbon electrode using Ag/AgCl as reference electrode are recorded. Cyclic voltammograms were recorded with an initial potential Ei +1100 mV and final (Switching) potential Es of – 1300 mV at different pH and at different scan rate viz 100,200,300,400 and 500mV/s (fig. 1,2,3) in aqueous medium at different pH.

Typical cyclic voltammograms of 4- nitro acetanilide are given in Figs.1 to 3, The shape of cyclic voltammograms indicates the irreversible nature of reduction.

III. RESULTS AND DISCUSSION

The voltammographic curves of 0.1 M 4- nitro acetanilide in aqueous medium, 1M KCl as supporting electrolyte and BR buffer (PH=5, 7 and 9) at glassy carbon electrode using Ag/AgCl as reference electrode are recorded. Cyclic voltammograms were recorded with an initial potential Ei +1100 mV and final (Switching) potential Es of – 1300 mV at different pH and at different scan rate viz 100,200,300,400 and 500mV/s (fig. 1,2,3) in aqueous medium at different pH.

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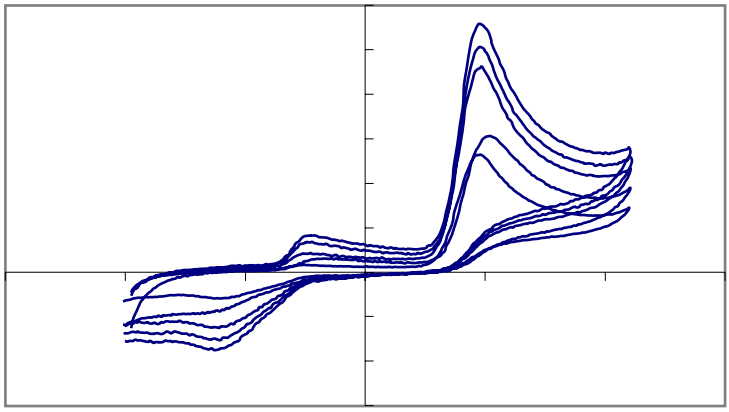


Fig.-1: Cyclic Voltammograms of 4- nitro acetanilide at pH 5 with Different Scan Rates

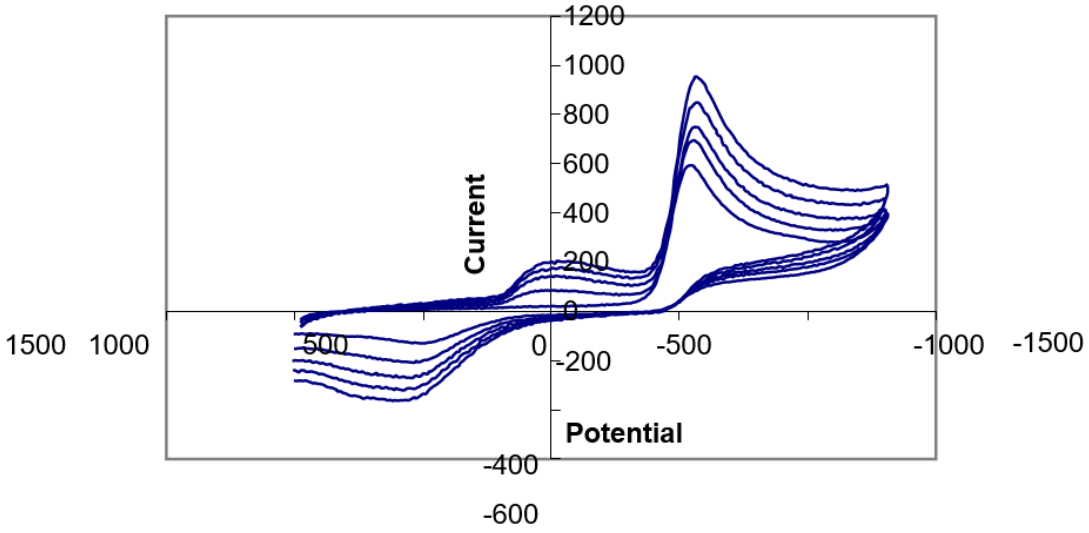


Fig.-2: Cyclic Voltammograms of 4- nitro acetanilide at pH 7 with Different Scan Rates

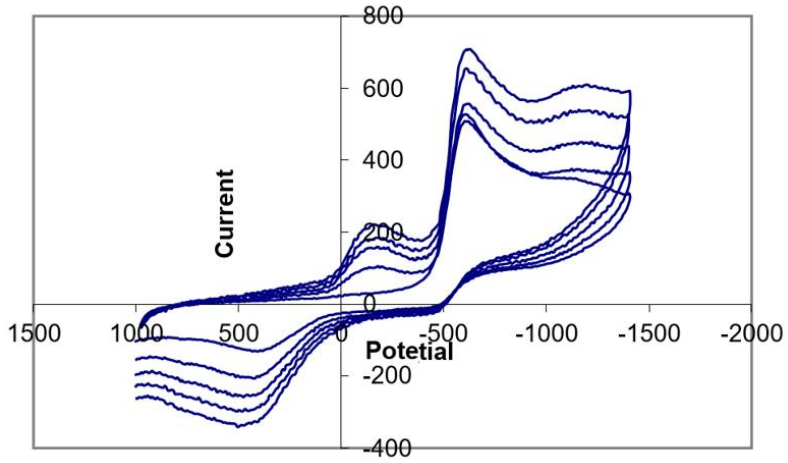
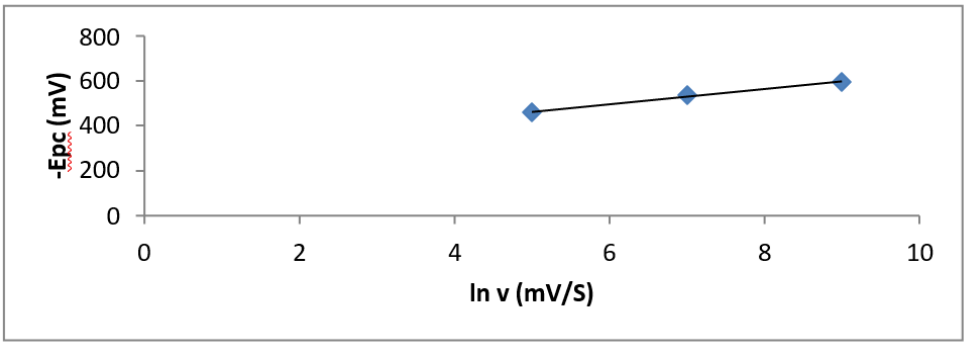


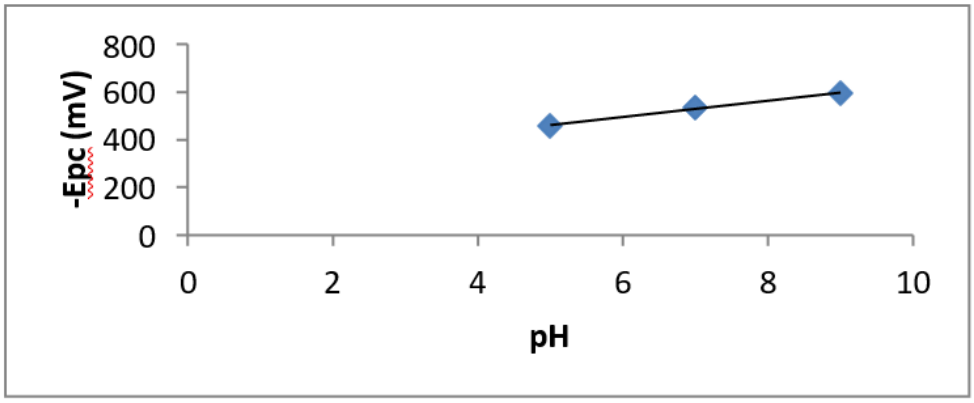
Fig.-3: Cyclic Voltammograms of 4- nitro acetanilide at pH 9 with Different Scan Rate

From above cyclic voltammograms we can report, the effect of scan rate and effect of pH on cyclic parameters. Effect of scan rate on cyclic parameters:- From the above cyclic voltammograms it is clear that as the sweep rate was gradually increased to 100mV/s to 500mv/s ,peak gradually shifted towards higher values as is expected for an irreversible electron transfer processes.



Dependence of peak potential on scan rates at pH7.0

Effect of pH on cyclic parameters:- All the cyclic voltammograms showed that the cathodic peak potential shifting towards more negative potential direction with increasing pH values This indicates participation of proton during electrode process.



Above mentioned electrolytically reduced product (4- amino acetanilide) in acidic medium was obtained in reasonably good yield .Single spot TLC checked the purity of compounds. The identity of products was further confirmed on the basis of melting point spectroscopic analysis.

Product Name	**B.P (°C)**	**IR value (cm^{-1})**	**NMR (δ value)**
4-amino acetanilide	165°C	3510-3600 cm^{-1} (N-H str.),3030 S (Ar-H str.), 2950 (C-H str.),1650 (C=O str.)1677cm^{-1} (N-H ben.), 1190 cm (C-O str.)	9.30 (s 1H),6.40 (m, 2H,Ar-H), 7.32(m,2H, Ar-H) 4.65(s,2H) 1.5(s,3H)

IV. CONCLUSION

The cyclic voltammetric studies of studied compound reveal irreversible electrochemical behaviour. The electrochemical reduction of 4-nitro acetanilide compound at constant current provides an alternative synthetic route for synthesis of 4-amino acetanilide compounds.

Conflicts of interest.

The authors declare that they have no conflicts of interest.

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Cite this Article

Ishwar Chand Balaee, Kamal Kishor Verma , "Cyclic Voltammetric Behaviour of 4-Nitroacetanilide at Glassy Carbon Electrode at different pH", International Journal of Scientific Research in Science and Technology (IJSRST), Online ISSN : 2395-602X, Print ISSN : 2395-6011, Volume 9 Issue 1, pp. 499-503, January-February 2022.

Journal URL : https://ijsrst.com/IJSRST2215415