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Electro Organic Synthesis and Characterization of 1-(4-Bromophenyl) ethanol

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ABSTRACT:

Electro Organic Synthesis and characterization of 1-(4-Bromophenyl) ethanol is analyzed. Cyclic voltammetry having glassy carbon electrode (GCE) is used to study the electrochemical behavior of 4-Bromoacetophenone in acetonitrile and constant current electrolysis is used to carried out electrochemical reduction of 4-Bromoacetophenone to yield 1-(4-Bromophenyl) ethanol. Effect of pH and scan rate on the reduction reaction peaks has been calculated. The kinetic parameters have been also calculated and the process has found to be diffusion controlled. For characterization of synthesized 1-(4-Bromophenyl) ethanol IR, ^{1}HNMR and Mass spectral analysis were carried out.

KEYWORDS: - Electro Organic Synthesis, Cyclic voltammetry, Glassy carbon electrode, Constant current electrolysis, Electrochemical reduction.

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INTRODUCTION

Electro Organic Synthesis is capable of simplifying and developing novel synthetic processes as well as carrying out a few syntheses which are otherwise very difficult to achieve by usual chemical procedures[1-2]. Here the electron plays an important role and acts as a reagent and is generated during the electrochemical reaction therefore it is known as Electro organic synthesis. This avoids undesirable byproducts formation, simplifies the work up procedure, ecofriendly, cost effective and products thus obtained are in good yield with relatively purer state[3-4].

Electro-organic synthesis[5-6] may be achieved with greater certainty and precision with the help of information obtained about the behavior of the substrate from cyclic voltammetric experiments. The optimum conditions for the electrolysis reaction can be set using logical interpretation of the signals obtained from cyclic voltammetry[7].

The electrochemical reduction is one of the greener approaches because it is pollution free as electrons are regarded as one of the reagents therefore it reduces the use of at least one hazardous chemical reagent [8-10]. Electrochemical techniques are also very useful to investigate kinetics and mechanisms of the reactions hence electro organic synthesis provide novel synthetic approach[11].

Based on the above-mentioned applications in present work electrochemical reduction of 4-Bromoacetophenone to the corresponding aromatic alcohol 1-(4-Bromophenyl) ethanol has been carried out using cyclic voltammetry (to evaluate electrode reaction) and constant current electrolysis. Aromatic alcohols have also been synthesized via microbial transformation using Baker's Yeast and these synthesized products are biological active material against the pathogenic bacteria [12-13].

MATERIALS AND METHODS

All chemicals used in the present investigation were of analytical grade. All the solvents were dried and then distilled out. Doubly distilled water was used to prepare the required solutions. ^{1}H NMR spectra were recorded using Joel (Japan) 300MHZ spectrophotometer. FT-IR spectra were recorded from Nicolet (USA) FT-IR spectrophotometer. Mass spectral analysis has been done in Central Drug Research Institute (CDRI), Lucknow.

Electrochemical Reduction

The completely computer controlled Basic Electrochemistry System model ECDA-001 was used for recording cyclic voltammograms of 4-Bromoacetophenone at different pH and scan rates in aqueous acetonitrile using potassium chloride as supporting electrolyte at glassy carbon electrode.

Cyclic voltammetric studies were carried out using a glassy carbon working electrode (A = 0.1 mm^2), Ag/AgCl reference electrode and a platinum auxiliary electrode. All the measurements were carried out at room temperature. The working electrode was polished intensively with aluminium oxide (0.4 μ) on a polishing cloth and degreased in acetonitrile prior to each electrochemical measurement.

The solutions were purged with purified clean dry nitrogen for 5 min prior to the experiments in order to remove dissolved oxygen from the media and blank cyclic voltammograms were recorded. Solution of 1mM of 4-Bromoacetophenone was added to blank solution then initial potential, final potential, scan rate and current sensitivity were provided and the resulting current was measured as a function of applied potential.

Constant Current Electrolysis

4-Bromoacetophenone was subjected to constant current electrolysis at constant current at 1.0 amp for 6 hrs in aqueous acetonitrile. Galvanostat supplied by OMEGA type ICVD 60/2 was used to perform the experiment. A Remi hot plate cum magnetic stirrer (2 M LH model) was used to stir the solution throughout the electrolysis.

A two compartment H- shaped glass cell provided with a fritz glass disc (G-4) was used for electrolysis. Rectangular plates of stainless steel (SS-316) each of size (4 cm × 6 cm) was used as cathode as well as anode. The Britton Robinson buffer of appropriate pH and the supporting electrolyte (CH_3COONa) was filled in both the limbs of H- shaped glass cell. 4-Bromoacetophenone was dissolved in minimum amount of acetonitrile and placed in the cathodic compartment and electrolyzed at constant current (1.0 amp). After the completion of reaction, extraction was done with diethyl ether. The synthesized product was then characterized by combined application of boiling point measurement, chromatographic and spectral techniques (Table-2 and 3).

RESULTS AND DISCUSSION

Electrochemical reduction of 4-Bromoacetophenone has been depicted by reaction scheme as follows:-

1-(4-Bromophenyl)-ethanone

1-(4-Bromophenyl)-ethanol

In the cyclic voltammograms of 4-Bromoacetophenone at pH 5.0, pH 7.0 and pH 9.0 single irreversible cathodic peak was observed due to the reduction of >C=O moiety to the corresponding secondary alcohol yielding 1-(4-Bromophenyl) ethanol as final product. Kinetic Parameters evaluated from cyclic voltammograms are given in Table No.1.

Effect of scan rate

The effect of scan rate on cathodic peak potential (Epc) was studied. As shown in Fig. 1 on increasing the scan rate, the cathodic peak potential (Epc) is shifted towards more negative potentials indicating an irreversible electron transfer process. The dependence of the voltammetric peak current (Ipc) of the wave on the square root of scan rate ($v^{1/2}$) is linear with correlation coefficients close to unity (Fig.3) at all the pH . Under these conditions the current process was diffusion controlled. Therefore 1-(4-Bromophenyl) ethanol was reduced electrochemically in a diffusion -controlled irreversible cyclic voltammetry wave.

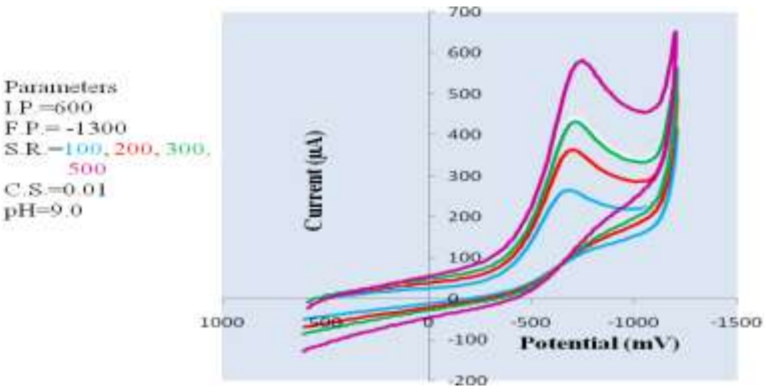


Fig. 1: Cyclic voltammograms of 4-Bromoacetophenone at various scan rates at pH 9.0

Effect of pH

The effect of pH was studied on the basis of cyclic voltammograms obtained by varying the pH from 5.0 to 9.0 as shown in Fig.2. Cyclic voltammograms show that electrochemical reduction is best carried out in basic medium because in acidic and neutral medium less defined peaks are observed while in basic medium a well-defined peak appeared.

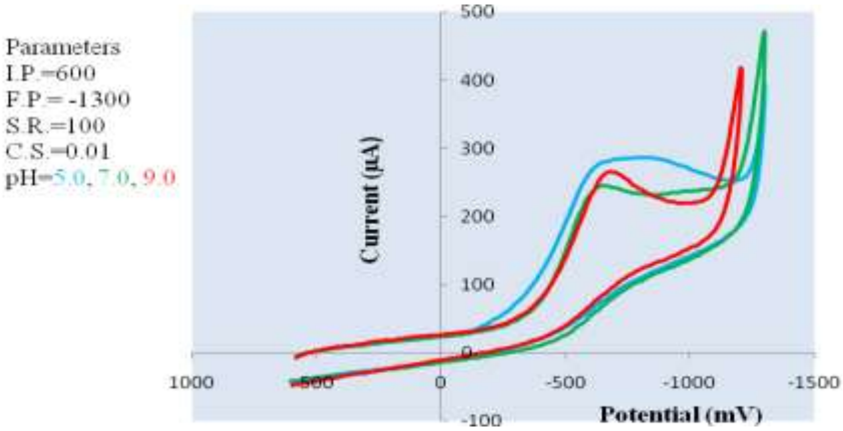


Fig. 2: Cyclic voltammograms of 4-Bromoacetophenone at different pH

Table No. 1: "Voltammetric data evaluated from cyclic voltammograms of 4-Bromoacetophenone"

S. No.	**Compound**	**Scan rate v (mV/s)**	**Cathodic peak potential E_{pc}(mV)**	**Cathodic peak current I_{pc}(µA)**	**peak current / square root of scan rate (Ip/√v)**
1.	4-Bromoacetophenone	100	-627	249	24.90
		200	-665	354	25.03
		300	-677	449	24.92
		500	-713	551	24.64

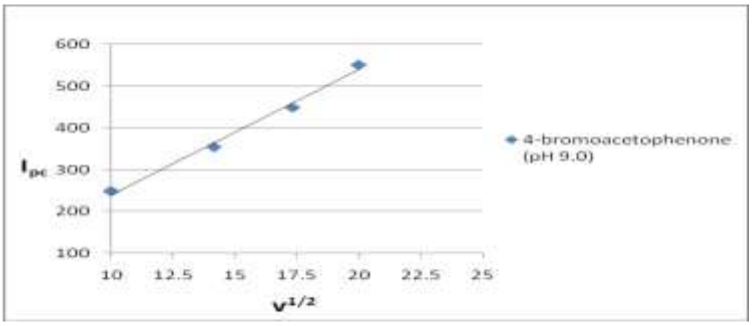
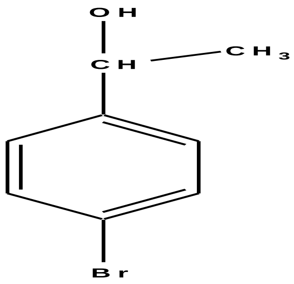


Fig. 3: Graphical representation of relation between cathodic peak current (i_{pc}) and $v^{1/2}$ for 4-Bromoacetophenone at pH 9.0

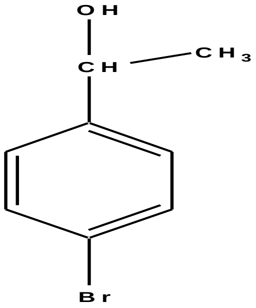
Table No. 2: "Physical data from Electrochemical Reduction"

Product		Reaction Time (in hours)	Boiling Point (^{0}C)	Yield (%)
Name	**Structure**			
1-(4-Bromophenyl) ethanol		6	120	73

CONCLUSIONS

1-(4-Bromophenyl) ethanol has been synthesized using Electro organic synthesis and characterized on the basis of analytical and spectral data. In Electro organic synthesis the electron plays an important role and acts as a reagent and is generated during the electrochemical reaction. This avoids undesirable byproducts formation, simplifies the work up procedure, ecofriendly, easy to handle, economically viable and products thus obtained are in good yield with relatively purer state.

Table No. 3: "Spectral data from Electrochemical Reduction"

Product		IR Data (cm^{-1})	^{1}H NMR Data (δ)	Mass Data m/z (M$^+$)
Name	Structure			
1-(4-Bromophenyl) ethanol		3400(OH), 3040(Ar C-H str), 2970(CH-str), 1600, 1470(C=C ring str), 1100 &1260(C-O str), 650-850 (strong Aromatic absorption as C-H out of plane bend. Vib.)	2.1; OH (s), 4.8; 1H (m), 1.48; 3H (d), 7.3; Ar-4H (m)	200 (M$^+$) and 202 (M+2)

REFERENCES:

1. Bocarsly AB. Electrochemical Techniques, Introduction. 2012. DOI: 10.1002/0471266965.
2. Clarke RE, Kulkarni A, Giddey S et al. Evaluation of solid electrolyte cells with a versatile electrochemical techniques. Ionics. 2013; 19:265-275. DOI: 10.1007/s11581-012-0749-1.
3. Shano T. Electro-organic Chemistry as a New Tool in Organic Synthesis. Springer: New York; 1984.
4. Torri S. Electro-organic Synthesis. Kodansha: Tokyo; 1985.
5. Nematollahi D and Amani A. Electrochemical synthesis of the new substituted phenylpiperazines. J. Electroanal. Chem. 2011; 651(1): 72-79.
6. Lateef S, Krishna Mohan SR, Jayarama Reddy SR. Electroorganic synthesis of benzathine. Tetrahedron Letters. 2007; 48(1): 77-88.
7. Masek A, Zaborski M, Chrzescijanska E. Electro-oxidation of flavonoids at platinum electrode studied by cyclic voltammetry. Food Chem. 2011; 127(2): 699-704.
8. Saharan R. Electrochemical Synthesis and Characterization of 2, 4-Dichlorobenzylalcohol, Ascent International Journal for Research Analysis. 2017; 2(4): 2.1–2.5.
9. Niyazymbetov ME. Electrochemical methods in organic synthesis of valuable intermediates. Electrosynthesis. 1997; 3 (2).
10. Mamatha GP, Sherigara BS, Mahadevan KM. Electrochemical reduction of 2-acetyl benzofuran and its derivatives at glassy carbon electrode. Indian Journal of Chemical Technology. 2007; 14: 566-571.

11. Jayadevappa H, Shivraj Y, Mahadevan K M et al. Electrochemical behavior of some industrially important azonaphthol derivatives at glassy carbon electrode. Indian Journal of Chemical Technology. 2006; 13(3): 269-274.
12. Saharan R, Analysis of Antibacterial Properties of 2-Methylcyclohexanol. International Journal of Scientific Research and Review. 2018; 7(6): 347-351.
13. Saharan R, Joshi J. Synthesis, characterization and antibacterial studies of 4-Hydroxy-3-methoxy-benzyl alcohol and 1-(4-Hydroxy-3-methoxyphenyl) ethanol. Journal of Biological and Chemical Research. 2016; 33(1): 529-536.