

# Synthesis and Characterization of 2-Chloro-6-fluorobenzylalcohol using Electrochemical Approach

Ritu Saharan

Department of Chemistry, University of Rajasthan, Jaipur, 302004, India

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**Abstract**—Electrochemical Synthesis and characterization of 2-Chloro-6-fluorobenzylalcohol is investigated. The electrochemical behavior and electrochemical reduction of 2-Chloro-6-fluorobenzaldehyde has been carried out using cyclic voltammetry having glassy carbon electrode (GCE) and constant current electrolysis respectively. Reduction reaction peaks at various scan rate and pH has been reported. In kinetic parameters study, the process has been found to be diffusion controlled. The synthesized 2-Chloro-6-fluorobenzylalcohol has been characterized using IR, <sup>1</sup>H NMR and Mass spectral analysis.

**Keywords**—Cyclic voltammetry, Electrochemical Synthesis, Constant current electrolysis, Glassy carbon electrode, Electrochemical reduction

## I. INTRODUCTION

The electrochemical technique is a methodology in which either oxidation or reduction of substrate yields the desired product [1-2]. In electrochemical synthesis the main role is of electron. It is produced during the electrochemical reaction and participated as a reagent in reaction. It is pollution free therefore it eliminates the use of harmful reagent [3]. This makes simple work up procedure, avoids formation of undesirable byproducts, economically viable, ecofriendly in nature, products are pure and in good yield [4-5].

The kinetics and mechanisms of redox reactions is being carried using cyclic voltammetry. The signals from cyclic voltammetry are helpful to predict optimum conditions for electrolysis process [6-8]. Cyclic voltammograms are also useful to know the mechanism of organic reaction [9-10].

In agricultural derivatives and pharmaceutical drugs, 2-Chloro-6-fluorobenzylalcohol is utilized as intermediates synthesis.

In keeping view of all mentioned above applications, presented work describes the electrochemical reduction of 2-Chloro-6-fluorobenzaldehyde to the corresponding aromatic alcohol 2-Chloro-6-fluorobenzyl alcohol using cyclic voltammetry (to estimate electrode reaction) and constant current electrolysis. Biotransformation via Baker's Yeast has also been employed to synthesize aromatic alcohols and

these synthesized products possess potential antibacterial properties against the pathogenic bacteria [11-12].

Organization of the paper (Rest of the paper is organized as follows, Section I contains the introduction of Electrochemical Synthesis, Section II contain the materials and methods adopted, Section III describes results and discussion, and Section IV concludes research work).

## II. MATERIALS AND METHODS

All the used chemicals in present study are of AR (analytical reagent) grade. All the solvents have been dried and then distilled out. The required solutions have been prepared using doubly distilled water. Joel (Japan) 300MHZ spectrophotometer has been used to record <sup>1</sup>H NMR spectra. Nicolet (USA) FT-IR spectrophotometer has been used to record FT-IR spectra. Mass spectra were recorded from Central Drug Research Institute (CDRI), Lucknow.

### Electrochemical Reduction

The was used for recording .Cyclic voltammograms of 2-Chloro-6-fluorobenzaldehyde have been recorded on completely computer controlled Basic Electrochemistry System model ECDA-001 at various scan rates and pH in aqueous methanol. As a supporting electrolyte, Potassium chloride is used. Cyclic voltammograms were obtained using a working electrode of glassy carbon ( $A = 0.1 \text{ mm}^2$ ), reference electrode of Ag/AgCl and an auxiliary electrode of

platinum. All the experiments have been performed at room temperature. Before each electrochemical measurement glassy carbon working electrode has been polished with aluminium oxide (0.4  $\mu$ ) with the help of polishing cloth.

For removal of dissolved oxygen from the media, the test solutions have been purged with clean purified and dry nitrogen for 5 min before the experiments and then blank cyclic voltammograms have been recorded. 1mM Solution of 2-Chloro-6-fluorobenzaldehyde has been added to blank solution followed by setting up the initial potential (IP), final potential (FP), scan rate (SR) and current sensitivity (CS). Finally cyclic voltammograms were obtained.

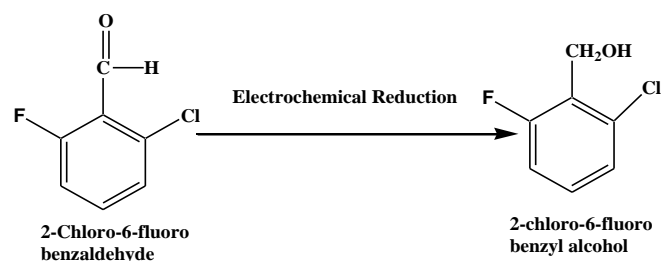
### Constant Current Electrolysis

Electrochemical reduction of 2-Chloro-6-fluorobenzaldehyde has been carried out using constant current electrolysis keeping constant current of 1.0 amp for duration of 06 hrs in aqueous methanolic solution. Experiment was performed using Galvanostat (supplied by OMEGA type ICVD 60/2). In electrolysis for stirring the test solution, a Remi hot plate cum magnetic stirrer (2 M LH model) has been used.

For electrolysis H- shaped glass cell (consisting two compartment) having a fritz glass disc (G-4) has been employed. Stainless steel (SS-316) electrode (4 cm  $\times$  6 cm) has been used as cathode and anode. The Britton Robinson (BR) buffer of pH 9.0 and CH<sub>3</sub>COONa as supporting electrolyte has been used to fill both the compartments of H-shaped glass cell. By using minimum amount of methanol, 2-Chloro-6-fluorobenzaldehyde has been dissolved and poured in the cathodic chamber. Then electrolysis was done at constant current at 1.0 amp. Diethyl ether has been used for extraction after the successfully completion of reaction. The synthesized product has been then characterized by melting point measurement, chromatographic and spectral techniques (Table-2 and 3).

## III. RESULTS AND DISCUSSION

Electrochemical reduction of 2-Chloro-6-fluorobenzaldehyde has been illustrated by reaction as below:-



In the cyclic voltammograms of 2-Chloro-6-fluorobenzaldehyde at different pH viz. pH 5.0, pH 7.0 and pH 9.0 single irreversible reduction peak has been observed because of reduction of  $>\text{C}=\text{O}$  group to the respective secondary alcohol to yield final product as 2-Chloro-6-fluorobenzylalcohol. Table 1 shows the kinetic Parameters calculated from cyclic voltammograms.

### Scan rate effect

The scan rate effect on Epc (cathodic peak potential) has been investigated. As shown in Figure 1, an increase in scan rate from 100 mV/s to 500 mV/s, shifts the cathodic peak potential in direction of more negative potentials. This indicates that process of electron transfer is irreversible. The graph between cathodic peak current (I<sub>pc</sub>) and square root of scan rate ( $v^{1/2}$ ) is in line with correlation coefficients near equal to unity (Figure 3).

The current process has been found to be diffusion controlled under these scenario.

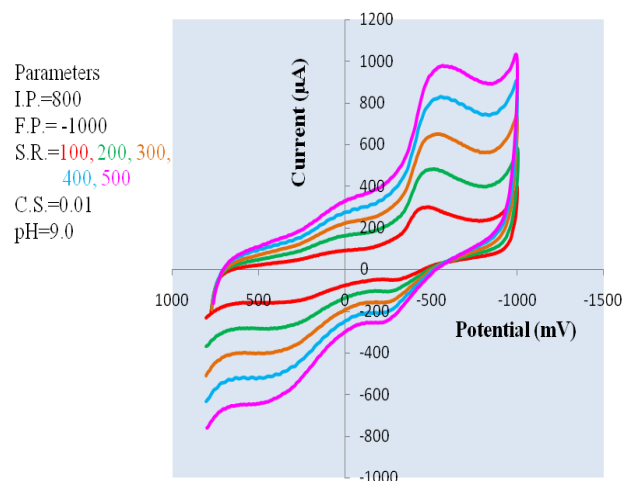


Figure 1. Cyclic voltammograms of 2-Chloro-6-fluorobenzaldehyde at different scan rates keeping pH 9.0

### Effect of pH

As shown in Figure 2, cyclic voltammograms have been obtained at various pH viz. pH 5.0, 7.0 and pH 9.0 and effect of pH on reduction peak has been observed. From cyclic voltammograms it has been found that a prominent peak has been seen in alkaline medium. So electrochemical reduction is performed extremely good in basic medium. In acidic medium peak remains absent and in neutral medium very weak peak is observed.

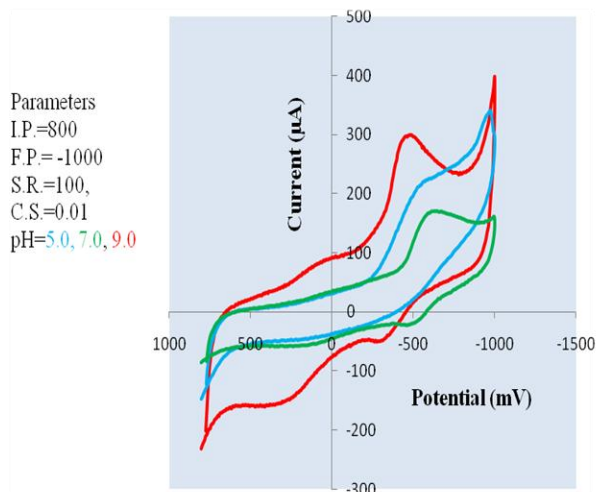


Figure 2. Cyclic voltammograms of 2-Chloro-6-fluorobenzaldehyde at different pH

Table 1. Voltammetric data evaluated from cyclic voltammograms of 2-Chloro-6-fluorobenzaldehyde

Scan rate $\nu$ (mV/s)	Cathodic peak potential $E_{pc}$ (mV)	Cathodic peak current $I_{pc}$ ( $\mu A$ )	peak current / square root of scan rate ( $I_p/\sqrt{\nu}$ )
100	-417	352	35.2
200	-428	448	35.57
300	-459	616	35.65
400	-486	789	35.5
500	-509	949	35.79

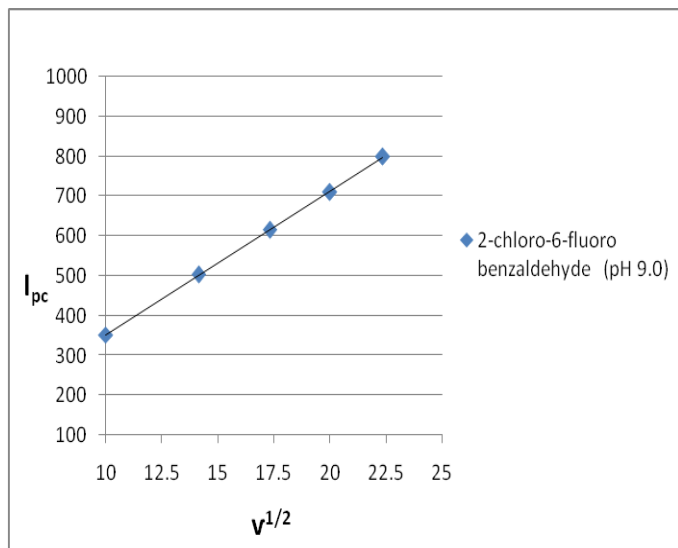
Figure 3. Graphical showing relationship between cathodic peak current ( $i_{pc}$ ) and  $\nu^{1/2}$  for 2-Chloro-6-fluorobenzaldehyde at pH 9.0

Table 2. Physical data of 2-Chloro-6-fluorobenzyl alcohol from Electrochemical Reduction

IR Data ( $\text{cm}^{-1}$ )	$^1\text{H}$ NMR Data ( $\delta$ )	Mass Data $m/z$ ( $M^+$ )
3365(OH), 3020(Ar C-H str), 2880-2960(CH-str), 1500,1450(C=C ring str), 1040(C-O str primary alcohol, 650-800 (strong Aromatic absorption as C-H out of plane bend. Vib.)	2.1; OH (s), 4.74; 2H (s), 7.2; Ar-3H (m)	160 ( $M^+$ ) and 162 ( $M+2$ )

Table 3. Spectral data of 2-Chloro-6-fluorobenzyl alcohol from Electrochemical Reduction

Product Structure	Reaction Time (in hours)	Melting Point ( $^{\circ}\text{C}$ )	Yield (%)
	6	44	86

#### IV. CONCLUSION

2-Chloro-6-fluorobenzyl alcohol has been synthesized using electrochemical technique and characterization has been done based on analytical as well as spectral data. In electrochemical synthesis the electron is participated in form of reagent and produced in course of the electrochemical process. This overcomes the formation of adverse byproducts, make work up procedure simpler, ecofriendly, easy to handle, cost effective. Hence obtained products are in pure state and good yield.

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#### AUTHORS PROFILE

Dr. Ritu Saharan pursued B. Sc., M.Sc., and Ph.D. Chemistry from MNIT Jaipur in 2005, 2007 & 2013. She is by profession as Assistant Professor in Department of Chemistry from University of Rajasthan, Jaipur since 2014. She has publication of more than 20 research papers in reputed international journals and conferences. Her main research work focuses on Electro-organic Synthesis and Bio-transformation, Organic Chemistry and Green Chemistry. She has 4 years of teaching and research experience.