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ENVIRONMENTALLY BENIGN ELECTRO-ORGANIC SYNTHESIS OF 2-PHENYL-1-PROPANOL

Chemistry

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ABSTRACT

Recently, electro-organic synthesis has progressively emerged as a foundation stone of modern synthetic organic chemistry. This method offers a green and economically viable route under mild conditions, avoiding toxic reagents while requiring minimal resources, producing negligible waste and delivering high-purity products with improved atom efficiency. In this context, an eco-friendly and facile electrochemical synthesis of 2-phenylpropan-1-ol has been investigated. The electrochemical behaviour of 2-phenylpropanal was studied using cyclic voltammetry, followed by constant-current electrolysis to obtain 2-phenylpropan-1-ol. The effects of scan rate, pH and other parameters on the reduction peaks were examined and kinetic parameters were evaluated, indicating a diffusion-controlled reduction process. The synthesized compound was characterized using IR, 1HNMR and Mass spectrometry techniques. 2-Phenyl-1-propanol is an important aromatic alcohol used as a lilac-like fragrance and reported as a potential biomarker in blackberries. The developed electrochemical protocol offers a sustainable and efficient approach for synthesizing pharmaceutically relevant aromatic alcohol derivatives, providing a promising platform for green pharmaceutical manufacturing and biomedical research applications.

KEYWORDS

electro-organic synthesis, eco-friendly, atom efficiency, economically viable, sustainable

1 INTRODUCTION

Substantial use of fossil fuels and restricted resources are pushing scientists to focus on rethinking chemical processes and creating fresh, sustainable methods. The process of creating organic molecules while using electricity is known as electro organic synthesis [1]. Traditionally, stoichiometric amounts of reducing and oxidizing chemicals are needed to comprehend redox processes. The addition of stoichiometric extents of redox reagents will inescapably result in dangerous and parlous waste, harming the environment and speeding up these operations even if these approaches allow the creation of target molecules [2-3].

In an attempt to reduce waste or even better, prevent it altogether, scientists and chemists are working hard and persistently [4]. This situation has given rise to a number of promising techniques, one of which is electro-organic synthesis, which improves organic synthesis in aqueous medium incorporating mild circumstances with no need of noxious and harmful substances [5-6]. It is also economically feasible, environmentally benign, timesaving, sustainable and energy-efficient [7]. This method reveals new approaches for the synthesis of organic molecules, including precise control, ease of workup, high reaction proficiency, intrinsic sustainability, atom-economic conversion of reactants with intermediates, high functional group tolerance and benign transformation [8-9].

This approach is significantly greener than traditional chemical techniques because it uses electrons as redox counterparts to achieve a high level of selectivity without the need for stoichiometric reagents or the creation of unwanted byproducts. The utilized electrodes operate as easily removable heterogeneous catalysts from the reaction site during these operations [10-12].

Cyclic voltammetry is a potential method for studying kinetics, redox reaction processes, and hydrogen adsorption or desorption of a wide range of compounds. Accurate electro-organic synthesis may be accomplished by utilizing cyclic voltammetric experimental data to support the reactant nature. The best reaction conditions for the electrolysis process may be selected using the signals obtained from the cyclic voltammetry research [13-14].

2-Phenyl-1-propanol also known as b-hydroxy cumene is aromatic in nature, having single monocyclic ring structure comprising of benzene. 2-Phenyl-1-propanol is a valuable lilac-scented compound used in detergents and cosmetics, and it may also act as a biomarker for the ingestion of evergreen blackberries. [15].

The current research study explores the electro organic synthesis of 2-phenylpropan-1-ol using cyclic voltametric measurements and constant current electrolysis to electro reduce 2-phenyl propanal, on the basis of the aforementioned uses.

2 Experimental

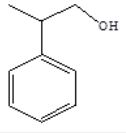
All the analytical grade chemicals have been used during investigation. Prior use distillation process has been carried out for all solvents. To make necessary solvents double distilled water has been used. ^{1}H NMR spectra have been taken by spectrophotometer-Joel, Japan (300MHZ). FT-IR spectra have been taken by spectrophotometer-Nicolet USA, FT-IR and Mass study has been done in CDRI, Lucknow, India.

2.1 Process Of Electrochemical Reduction

Employing supporting electrolyte (potassium chloride) at glassy carbon electrode, cyclic voltammograms of 2-phenyl propanal at various pH values (Fig. 2) and scan rates (Fig. 1) in aqueous DMF were recorded using the fully computer regulated fundamental Electrochemistry System design ECDA-001. Reference electrode (Ag/AgCl), auxiliary electrode (Pt), and working electrode (glassy carbon) (A = 0.1 mm2) have all been used in cyclic voltametric investigations. Each and every measurement has been made at room temperature. Before each electrochemical experiment, the working electrode was polished with 0.4 µm alumina on a polishing cloth and then degreased with dimethylformamide (DMF).

Prior to the studies, the solutions were purged with pure clean dry nitrogen for five minutes to eliminate any oxygen (which has been dissolve) from the medium, and blank cyclic voltammetric parameters were noted. After adding a 1 mM solution of 2-phenyl propanal to the blank solution, the resultant current was evaluated as a function of applied potential after the early potential, ending potential, scan rate (sr), and current sensitivity (cs) were given (Figs. 1 and 2).

Table 1 Physical Data As A Result Of Electrochemical Reduction

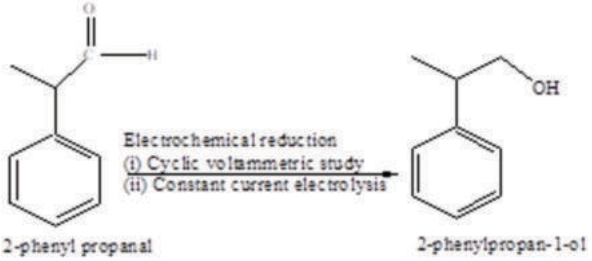
Product	Structure	Reaction Time (In hours)	Boling Point (^{0}C)	Yield (%)
2-phenylpropan-1-ol		6	110	87

2.2 Constant Current Electrolysis

2-phenyl propanal was electrolyzed at a steady current of 1.0 amp for seven hours in aqueous Dimethyl formamide (DMF). Utilizing a galvanostat of type ICVD 60/2 provided by OMEGA, the experiment was conducted. Throughout the electrolysis process, the solution has been stirred with a Remi hot plate cum magnetic stirrer (2 M LH type).

For electrolysis, a 2-section H-shaped glass cell consisting a fritz glass disk (G-4) has been employed. Stainless steel of SS-316 in rectangular shape, measuring 4 cm by 6 cm, have been utilized as both anode and cathode. In both limbs of the H-shaped glass cell, the supporting electrolyte (CH_3COONa) and the Britton Robinson buffer at the proper pH have been added. After dissolving 2-phenyl propanal in the least amount of Dimethyl formamide (DMF), it was electrolyzed at a continuous current of 1.0 amp in the cathodic compartment. Diethyl

ether was used in the extraction step following the conclusion of the reaction. Several analytical and spectral approaches have been used to characterize the resultant product (Tables 1 and 2). The following is the reaction scheme that illustrates 2-phenyl propanal's electrochemical reduction. (See Scheme-1)



Scheme-1 Electrochemical reduction of 2-phenyl propanal

Table 2 Spectral Data for Synthesized Product

Product	IR Data (cm^{-1})	^1H NMR Data (δ)	Mass Data m/z (M^+)
2-phenylpropan-1-ol	3325(OH), 2890-2950(CH-str), 3040(Ar C-H str), 1055 (C-O str due to primary alcohol), 1595, 1450(C=C ring str), 690-790 (C-H bend.vib.)	2.1; OH (s), 1.2; 3H (d), 4.72; 1H (m), 7.1; Ar-5H (m)	136 (M^+) and 137 (M+1)

3.RESULTS AND DISCUSSION

In electro organic synthesis, the organic reaction molecules are stimulated by an external stimulus, allowing for the control of the reaction and selectivity, which is a major limitation of traditional chemical methods. In this approach following electrochemical parameters are taken into consideration.

3.1 Electrochemical reduction

A single irreversible cathodic peak was seen in the cyclic voltammetric graph of 2-phenyl propanal at pH 5.0, pH 7.0, and pH 9.0. This was caused by reduction of the >C=O moiety to the respective secondary alcohol, which produced 2-phenylpropan-1-ol as the end product. Table 3 lists the kinetic parameters that were determined using cyclic voltammetric graph.

3.2 Impact of scan frequency (rate)

It was noted how scan rate affected the reduction peak potential (Epc). The impact of scan rate on reduction peak potential (Epc) is shown in Fig. 1. A shift towards a more negative direction has been observed in the value of the reduction peak potential (Epc) as the scan rate increases from 100 to 500. This has an indication of electron transfer process is to be an irreversible in nature.

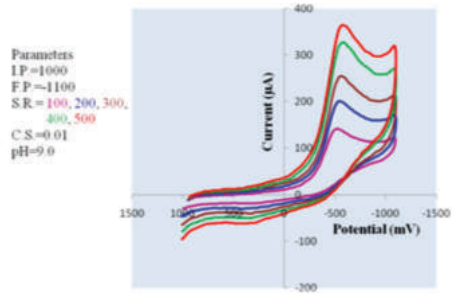


Fig. 1 Cyclic voltammetric graph of 2-phenyl propanal at pH 9.0 having different scan rates

3.3 Impact of pH

Cyclic voltammetry data collected at various pH values can be used to explain the effects of pH (Fig. 2). Based on a comparative analysis of cyclic voltammograms conducted at a scan rate of 100 mV/s across various pH levels, the basic medium is identified as the most suitable environment for the reduction process. In acidic medium, the reduction peak is entirely absent. In neutral medium, the reduction peak is present but poorly defined. In basic medium, a prominent, well-defined reduction peak is observed. Consequently, electrochemical reduction is most feasible and yields optimal results under basic condition.

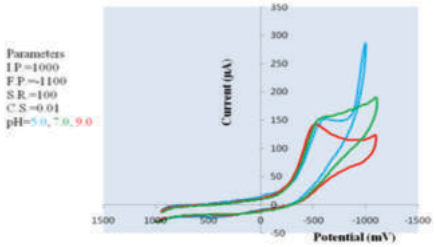


Fig.2 Cyclic voltammetric graph of 2-phenyl propanal at varying pH

Table 3. Voltammetric statistics estimated by cyclic voltammograms of 2-phenyl propanal

Product	scan rate v (mV/s)	cathodic peak potential E_{pc}(mV)	cathodic peak current I_{pc}(μA)	(Ip/√v)
2-phenyl propanal	100	- 493	144	14.4
	200	- 505	203	14.35
	300	- 532	252	14.54
	400	- 539	291	14.55
	500	- 545	323	14.44

3.4 Cathodic Peak Current (Ipc) Variation With Scan Rate Square Root ($v^{1/2}$)

At pH 9.0, the peak current (Ipc) of the wave has a linear dependency on the scan rate's square root ($v^{1/2}$), with correlation coefficients that are nearly equal to unity which indicates the current process was diffusion controlled (Fig.3). Therefore, electrochemical reduction of 2-phenyl propanal was diffusion controlled and irreversible in nature.

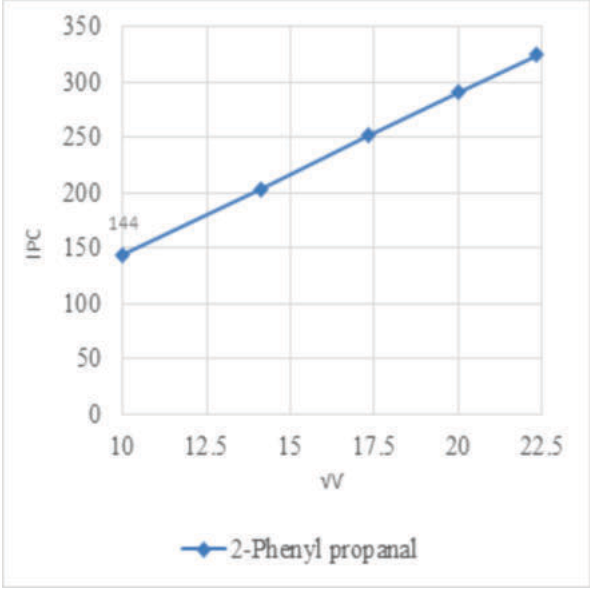


Fig. 3 The variation of the cathodic peak current (Ipc) for 2-phenyl propanal at pH 9 in respect of scan rate's square root ($v^{1/2}$)

4 CONCLUSION

Electro organic synthesis of 2-phenylpropan-1-ol has been achieved. Analytical and spectral data have been used to characterize synthesized 2-phenylpropan-1-ol. The produced 2-phenyl-1-propanol, a potential biomarker, is utilized as a lilac-scented ingredient in various products such as detergents and cosmetics. Compared to conventional chemical methods, this approach is cost-effective, energy-efficient and environmentally benign. This electrochemical protocol provides a sustainable and efficient route for synthesizing aromatic alcohol derivatives, using electrons as clean reagents and, electric current, to precisely control redox processes for advancing green chemistry in pharmaceutical research.

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