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Research Article

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Mediated Biphasic electrolysis on anodic oxidation of *p*-methylbenzyl alcohol in acid medium

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ABSTRACT

The anodic oxidation of p-methylbenzyl alcohol has been studied in the biphasic system containing NaCl as a redox mediator, ClO^-/Cl^- *by hydrolysis in HCl acid medium. Since several parallel reactions are possible, we have investigated, in an undivided cell equipped with a Platinum/Platinum electrode using chloroform as a solvent in presence of the 0.36M Hcl as a supporting electrolyte,.The following parameters influencing the productionof p-methylbenzaldehyde: nature of the In-Cell and Ex-Cell method, nature of the supporting electrolyte, nature of the anode, initial p-methylbenzylalcohol concentration, medium, mediator concentrationand reusability of spent mediator. For the optimum conditions, the conversion of p-methylbenzaldehyde yields maximum at 96%. The reaction mechanism and the features distressing the current efficiency of p-methylbenzaldehyde production were explored.*

Key words: Biphasic electrolysis, anodic oxidation, *p-methylbenzyl alcohol*, Ex-cell method,Sodium Chloride.

INTRODUCTION

Aromatic aldehydes are very useful as perfume components or key synthones for the production of a variety of fine and specialty chemicals such as pharmaceuticals, dyestuffs and pesticides.Selective catalytic oxidation of alcohols to corresponding aldehydes or acids is of great importance for both laboratory and synthetic industrial applications[1].In particular, aldehydes represent an important class of products and versatile intermediates in the field of fine chemicals such as fragrances or food additives[2].In this methodthe nucleophile (Nu^-) is transferred from the aqueous phase into the organic phase as a p-methylbenzylalcohol where it undergoes reaction with the newly electrogenerated species ClO^-/Cl^-.This method presents many advantages in electrochemistry: cheap nucleophile source (sodium salts), high conductivity and in general higher product yields in comparison to anodic substitution in heterogenous organic solutions. Many of the chemical reactions are not very selective. On the other hand, electrochemical methods omit the production of inorganic wastes from the oxidant, and appear to be a means to increase the selectivity of benzaldehyde formation. Prior reported on electrochemical oxidation by chemical method using transition metal compounds such as chromium (VI) oxide, permanganates, ruthenium (VIII) oxide and dichromates[3–5].Using toxic metal salts and transition metals for the conversion of alcohol oxidation to aldehydes [6–13].

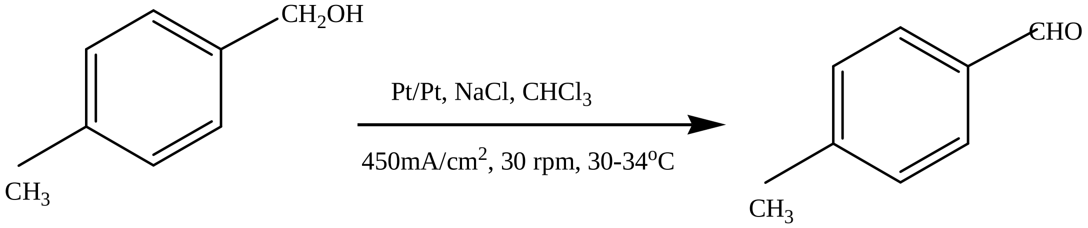
Allylic alcohols are selectively oxidized to aldehydes under the conditions of anodic oxidation of alcohols at the hydroxide nickel anode in the two-phase system K_2CO_3 (aq) petroleum ether. In the case of primary alcohols, the production of benzaldehyde had been carried out in non- polar organic phase thus preventing further oxidation to carboxylic acids [14]**.**Silica-supportedreagents [15**]** and carbon-supported platinum catalysts [16**]**Quaternary ammonium salt [17]was used to oxidize alcohols with electro-generated hypobromite as emulsion electrolysis.

Biphasic electrolysis has a distinct advantage over conventional homogeneous electrolysis with respect to easy handling and mediator recycling [18].In homogeneous systems, lower selectivity is observed due to over-oxidation of the substrate on the surface of the electrode leading to a mixture of products. In biphasic electrolysis systems, the reactive species formed by electrolytic oxidation of a halide ion in the aqueous phase, can be taken continuously into the organic phase, and then reacted with the substrate selectively to give the products. After completion of the electrolysis, separation and evaporation of the organic layer affords the product.

A biphase electrolytic system can be used readily to convert alkyl aromatic compounds to monobromo derivatives in quantitative yields [19]**.** Presently, only a few reports are available on the syntheses of fine chemicals by biphasic electrolysis method [20-21]**.** 4-methoxybenzyl alcohol was oxidized by photochemical reaction using TiO_2 as electrode [22]**.**Electrochemical oxidations have been carried out using various electrodes [23-25] as reported in the literature.

Ceric ammonium sulphate and Sodium nitrate mediators [26-27]are reported in our previous work in two phase electrolysis**.** Herein we further developed ainnovative method for the controlled oxidation of*p*-methylbenzyl alcohol using *in-situ* generated hypochlorous reactive species to the corresponding*p*-methylbenzaldehyde.

The Present work is to investigatetheof indirect electrochemical anodic oxidation of *p*-methylbenzyl alcohol by biphasic electrolysis in order to get an insight into an optimum operation conditions such as In-Cell and Ex-Cell method, supporting electrolyte, anodic materials,initial *p*-methylbenzylalcohol concentration, medium, mediator concentration and reusability of spent mediator. The maximum yield of 96% *p*-methylbenzaldehyde production at room temperature was achieved. The Scheme 1 globally representing *p*-methylbenzyl alcohol oxidation is as follows:



Scheme1: The anodic oxidation of *p*-methylbenzyl alcohol by biphasic electrolysis.

EXPERIMENTAL SECTION

2.1 Electrochemical oxidation of p-methylbenzyl alcohol.

The optimization was carried out in a 120 mL undivided cell equipped with a magnetic stirrer, and Platinum/Platinum electrodes were used for the biphasic electrolysis. Room Temperature was maintained constant throughout the electrolysis process. The volume of electrolyte40mL, the concentration of supporting electrolyte (0.36 M) and the stir rate were constant.

Aplab power source was used as a direct current source for the electrolysis. All the chemicals were of reagent grade quality.The electrolysis was monitored by HPLC (Shimadzu, Japan, Model. no. CLASS. VP-10) using (250 mm x 4.6 mm) as the stationary phase. The eluent consisted of acetonitrile/water (80:20) at a flow rate of 1 mL min^{-1}. Samples were analysed at a wavelength of 254 nm with a UV detector (Shimadzu UV–vis detector) coupled to a printer. Authentic samples of *p*-methylbenzaldehyde and 4-methylbenzoic acid were used to calculate the peak area of the corresponding experimental product for yield calculation

2.2 Analysis of the Products.

The FT-IR spectra of the products were recorded with a double-beam Mattson Galaxy Series FTIR-3000 spectrometer in the range of 4000–400 cm^{-1} using KBr pellets. 400 MHz ^{1}H spectra were measured on a 400 MHz FT-NMR spectrometer equipped with a Bruker S2 CryoPlatform in the indicated deutero solvent and at ambient temperature. Chemical shifts are reported in parts per million. DMSO as a solvent at 25°C.

2.3Optimization of *p-methylbenzaldehyde* production:

Experimental condition for In-Cell method.

A solution of *p*-methylbenzyl alcohol (1.045 g, 10 mmol) dissolved in 20 mL chloroform was transferred to an undivided electrolytic cell. An aqueous solution (60 mL) of sodium chloride(7.04g, 12%) containing 0.36 M HCl was added to the above solution. Deionized water was used for preparing sodium chloride solution. Two platinum electrodes each of 15 cm^2 area were placed in the upper layer of the aqueous phase without touching the organic phase but very close to the interphasial region. The aqueous upper phase acted as the supporting electrolyte and chloride as cationic source. Biphasic electrolysis was achieved with a magnetic stirrer at the lowest speed (30 r.p.m.) necessary to prevent the separation of the two phases. The temperature of the electrochemical cell contents was maintained at 30–34°C throughout the electrolysis. The electrolysis was conducted Galvanostatically at a current density of 30 mA/cm^2 until the quantity of 3F charge was passed. An aliquot was drawn periodically from the organic phase and was monitored by HPLC. After completion of the electrolysis, the lower organic phase was separated; washed with water (2 x 25 mL), dried over anhydrous Na_2SO_4 and the solvent was removed by distillation. HPLC analysis of the residue indicated the presence of 96% of 4-methylbenzaldehyde (1.172 g) along with 4% unconverted 4-methylbenzyl alcohol. All the isolated products were characterized by the usual spectroscopic techniques: IR, proton NMR and Product isolation by Column Chromatography. The spectroscopic characteristics were in agreement with those reported in the literature [26-27]

Experimental condition for Ex-Cell Method.

A solution of *p*-methylbenzyl alcohol (1.045 g, 10 mmol) dissolved in 20 mL chloroform was transferred to an undivided electrolytic cell. An aqueous solution (60 mL) of sodium chloride (7.04g, 12%) containing 0.36 M HCl was added to the above solution. Deionized water was used for preparing sodium chloride solution. The cell doesn't contain any electrodes ,the aqueous upper phase acted as the supporting electrolyte and chloride as cationic source. Biphasic electrolysis was achieved with a magnetic stirrer at the lowest speed (30 r.p.m.) necessary to prevent the separation of the two phases. The temperature of the electrochemical cell contents was maintained at 30–34°C throughout the electrolysis. The reaction was allowed to stir up to 71 mts.. After completion of the electrolysis, the lower organic phase was separated; washed with water (2 x 25 mL), dried over anhydrous Na_2SO_4 and the solvent was removed by distillation. HPLC analysis of the residue indicated the presence of 6 % of the yield 4-methylbenzaldehyde and 94% unreacted 4-methylbenzylalcohol .

In the electrochemical anodic oxidation of p-methylbenzyl alcohol was carried out in order to determine the influence of the following parameters.

Effect of Cell methods : (In-Cell and Ex-Cell)
Effect of medium : (Acidic, Basic and Neutral)
Effect of Various Supporting Electrolytes : (HCl, HNO_3, H_2SO_4, $HClO_4$)
Effect of Supporting Electrolyte Concentration : (0.18 0.27 0.36 0.45 0.55M)
Effect of Anodic materials : (C/C, Pt/Pt, Pt/C, C/SS)
Effect of Initial concentration of p-methylbenzylalcohol : (05, 10, 15, 20 mmol)
Effect of Chloride Mediator : NaCl, BaCl, KCl, NH_4Cl, $MgCl_2$, $SrCl_2$
Effect of Reusability of Spent Mediator : (Reuse 1, 2 and 3)

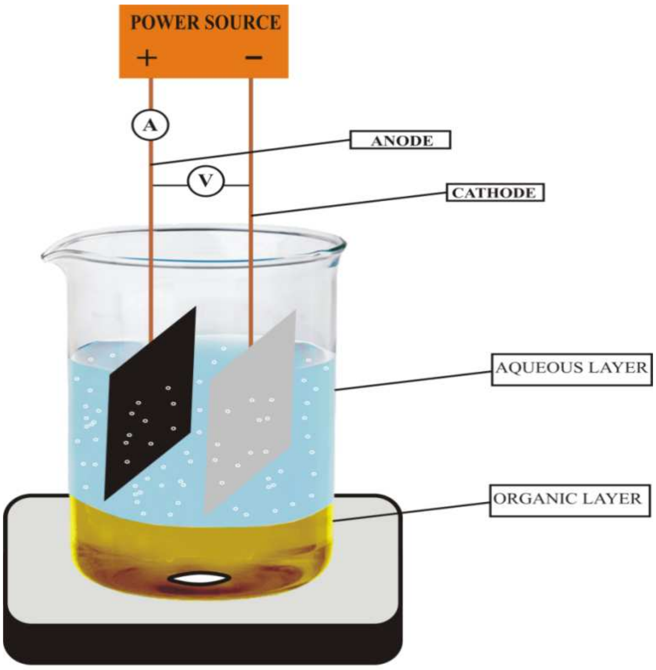


Figure 1. Electrochemical Cell set up for the anodic oxidation 4-methylbenzyl alcohol.

RESULTS AND DISCUSSION

3.1. Biphasic Electrolysis.

Biphasic electrolysis is one where the electrolyte and the substrate are present in different phases. When electrolysis occurs, electrolytically generated species travels from aqueous phase to organic phase and attacks the substrate. The products obtained in two-phase electrolysis will not be the same as in the homogenous electrolysis. The studies revealed that when the two-phases (aqueous and organic phases) are stirred in such a way without disturbing the two layers i.e., leaving the immiscible phases separated (stationary electrolysis), the products obtained from Biphasic electrolysis is entirely different from homogenous electrolysis.

3.2. Ratio of Electrolyte and Organic Phase.

In the present study saturated sodium chloride solution is used as upper phase and chloroform containing 4-methylbenzyl alcohol as lower phase. The unstable hypochlorous acid reactive species is generated electrochemically in aqueous phase by oxidation of chloride ions and is transported to the organic phase i.e. the reaction takes place at the interphase of the two-phases. Hence more the interphase area higher the transport of chlorinating species which makes the reaction fast. The cell is designed in such a way that the height–diameter ratio of the cell is 1:1. The electrodes were placed close to the interphase of the two-phases without touching the organic phase.

3.3 Effect of Method:

The experimental result reveals that Ex-cell method has only 6% of yield *p*-methylbenzaldehyde.

But in the case of In-Cell method the maximum yield of 96% was obtained. So, the electrode plays a pivitoal role in order for the anodic oxidation of *p*-methylbenzylalcohol in the In-Cell method.

3.4Effect of Medium:

Indirect electrochemical oxidation of *p*-methylbenzyl alcohol was carried out in a different medium. Acidic medium HCl has preferred, NaOH for basic medium and Neutral medium. Sensibly says, the acidic medium yields maximum 96%for the production of *p*-methylbenzaldehyde production. The results are reported in Table1

Table 1. Effect of Medium on the in-direct electrochemical oxidation of4-methylbenzylalcohol by biphasic electrolysis

S.No	Medium	Yield %		Current Efficiency % for the formation of 4-methylbenzaldehyde
		4-methylbenzylalcohol	4-methylbenzaldehyde	
1	Acidic [a]	4	96	64
2	Basic [b]	38	62	44
3	Neutral	31	66	41

Experimental conditions: Organic layer:p-methylbenzylalcohol (10mmol) in 20mL chloroform;
Aqueous layer: 12%NaCl (60mL H_2O); Supporting electrolyte: 0.36M[a] HCl, 0.05[b]NaOH; Current density: 30mA/cm2 Electrode: Pt/Pt Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.
[a] *AcidicMedium (HCl)*
[b] *Basic (NaOH)*

3.5. Effect of Supporting electrolyte.

Acid which acts as a supporting electrolyte for the biphasic electrolysis. This supporting electrolyte plays a key role in defining the environment surrounding an electrode. To find the optimum conditions for acid, studies were conducted at varying the acids on the controlled oxidation of 4-methylbenzyl alcohol. The Biphasic electrolysis was attempted with various acid media such as H_2SO_4, HNO_3 and $HClO_4$. It was observed that all other acids performed equally well, this means that current utilization will be the same for all the three acids expect HNO_3.Hence the suitable acid for the indirect electrochemical oxidation is HCl due to its low cost. The oxidant capacity of Sodium Chloride increases in presence of the acids in the following order HCl>$HClO_4$>H_2SO_4>HNO_3.Table 2 represents the effect of acid.

Table 2. Effect of supporting electrolyte on the in-direct electrochemical oxidation of 4-methylbenzylalcohol by biphasic electrolysis

S.No	Acids	Yield %		Current Efficiency % for the formation of 4-methylbenzaldehyde
		4-methylbenzylalcohol	4-methylbenzaldehyde	
1	HCl	16	84	56
2	H_2SO_4	18	82	55
3	$HClO_4$	16	84	56
4	HNO_3	34	65	43

Experimental conditions: Organic layer: p-methylbenzylalcohol (10mmol) in 20mL chloroform;
Aqueous layer: 12% NaCl (60mL H_2O); Supporting electrolyte: Various acids (2mL); Current density: 30mA/cm^2 Electrode: Pt/Pt Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

3.6. Effect of Acid Concentration.

Acid concentration takes an important vital role in the indirect oxidation systems. The effect of HCl concentration carried out for the oxidation of 4-methylbenzyl alcohol to 4-methylbenzaldehyde. Almost identical yields were obtained for 1mL, 1.5mL and 3mL (65-84%), on addition of 2mL of HCl concentration there is large increase in the4-methylbenzaldehyde yield. It is observed from the table that oxidation of 4-methylbenzyl alcohol to 4-methylbenzaldehyde is favored with the use of medium acid concentration of around 0.36 M HCl (2mL) the result reveals that this method is eco-friendly.Table 3 represents the effect of acid concentration and Figure 4 shows the correlation between acid concentration and yield of 4-methylbenzaldehyde.

Table 3. Effect of acid concentration on the in-direct electrochemical oxidation of 4-methylbenzylalcohol by biphasic electrolysis

S.NO	HClAcid Concentration (M)	Yield %		Current Efficiency % for the formation of 4-methylbenzaldehyde
		4-methyl benzyl alcohol	4-methylbenzaldehyde	
1	0.18	21	73	49
2	0.27	17	78	52
3	0.36	15	84	56
4	0.45	19	65	43
5	0.55	30	70	58

Experimental conditions: Organic layer: p-methylbenzylalcohol (10mmol) in 20mL chloroform;
Aqueous layer: 12 % NaCl (60mL H_2O); Supporting electrolyte: 0.18- 0.55 M HCl (Table 3) Current density: 30mA/cm^2 Electrode: Pt/Pt Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

3.7. Effect of Various Chloride Mediator.

The Effect of Salt variations was studied extensively to get the good response of various chloride mediator. The results are reported in Table 4. Prior to the various chloride mediator, NaCl will give good response for the oxidation of 4-methylbenzyl alcohol to 4-methylbenzaldehyde. It is observed from the table that oxidation of 4-methylbenzyl alcohol to 4-methylbenzaldehyde is favored with the use of NaCl.

Table 4. Effect of Chloride mediator on the in-direct electrochemical oxidation of 4-methylbenzylalcohol by biphasic electrolysis

S.No	Various Chloride mediator	Yield %		Current Efficiency %for the formation of 4-methylbenzaldehyde
		4-methylbenzyl Alcohol	4-methylbenzaldehyde	
1	NaCl	4	96	64
2	BaCl	11	87	58
3	KCl	22	70	47
4	NH_4Cl	19	76	51
5	$MgCl_2$	21	66	44
6	$SrCl_2$	12	85	57

Experimental conditions: Organic layer: p-methylbenzylalcohol (10mmol) in 20mL chloroform; Aqueous layer: 1.66% Various mediators (60mL H_2O); Supporting electrolyte: 0.36M HCl; Current density: 30mA/cm^2 Electrode: Pt/Pt Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

3.8. Effect of Chloride Mediator Concentration.

To find the optimum total chloride concentration, the experiments were carried out in presence of HCl acid medium at 30-34°C. Sodium chloride was used as mediator for the controlled oxidation of4-methylbenzyl alcohol in a biphasic electrolysis system. The result shows that 12% of mediator concentration is required for the formation of hypochlorous acid species. This *in-situ* generated hypochlorous acid species reacted instantly at interphasial region of organic and aqueous phase for the controlled oxidation of 4-methylbenzyl alcohol. Eventually, the mediator did not induce the product(4-methylbenzaldehyde) for further oxidation.Table 5 represents the effect of chloride concentration.

Table 5:Effect of Chloride mediator concentration on the in-direct electrochemical oxidation of4-methylbenzylalcohol by biphasic electrolysis

S. No	Mediator Concentration (%)	Yield %		Current Efficiency % for the formation Of4-methylbenzaldehyde
		4-methylbenzyl alcohol	4-methylbenzaldehyde	
1	1.66	26	69	46
2	5.00	17	45	30
3	8.33	23	56	37
4	12.01	4	96	64
5	15.00	12	75	50
6	Saturated Solution	7	93	62

Experimental conditions: Organic layer: p-methylbenzylalcohol (10mmol) in 20mL chloroform; Aqueous layer: 1.66-15% NaCl (60mL H_2O); Supporting electrolyte: 0.36M HCl; Current density: 30mA/cm^2 Electrode: Pt/Pt Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

3.9. Effect of Various Electrode Materials.

In the search for good reaction conditions one should begin with the electrode and sees that are inert at the reduction/oxidation potential of the substrate. With regards to all other electrodes Pt/Pt which is a good choice for the conversion of 4-methylbenzyl alcohol to 4-methylbenzaldehyde. Carbon/carbon and Carbon/Stainless electrodes get dissolved during the reaction this may leads to the fewer yields. Table 6 lists various types of electrode pairs employed in selective oxidation of4-methylbenzylalchol by the biphasic electrolysis method.

Table 6: Effect of anodic materials on the in-direct electrochemical oxidationof4-methylbenzylalcohol by biphasic electrolysis

S.No	Electrode Materials	Yield %		Current Efficiency %for the formation of 4-methylbenzaldehyde
		4-methyl benzyl alcohol	4-methylbenzaldehyde	
1	C/C	23	69	46
2	C/SS	21	76	51
3	Pt/SS	30	69	46
4	Pt/Pt	4	96	64

Experimental conditions: Organic layer: p-methylbenzylalcohol (10mmol) in 20mL chloroform; Aqueous layer: 12% NaCl (60mL H_2O); Supporting electrolyte: 0.36M HCl; Current density: 30mA/cm^2 Electrode: Various electrodes (Table 5) Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

3.10. Effect of Substrate Concentration.

Substrate concentration is the most important parameter for biphasic Electrolysis in the conversion of 4-methylbenzyl alcohol to 4-methylbenzaldehyde .At low substrate concentration from 05 -10 mmol gives an identical results (95-96%), when the substrate concentration getting increases from 15-20 mmol there is a large decrease in the yield of 4-methyl benzaldehyde, so the minimum substrate concentration is the optimum choice for the biphasic electrolysis system. Table 7 represents the substrate concentration.

Table 7:Effect of substrate concentration on the in-direct electrochemical oxidation of 4-methylbenzylalcohol by biphasic electrolysis

S.No	Substrate Concentration (mmol)	Yield %		Current Efficiency % for the formation of 4-methylbenzaldehyde
		4-methyl benzyl alcohol	4-methylbenzaldehyde	
1	05	5	95	63
2	10	4	96	64
3	15	12	83	55
4	20	16	77	51

Experimental conditions: Organic layer: p-methylbenzylalcohol (10mmol) in 20mL chloroform; Aqueous layer: 12% NaCl (60mL H_2O); Supporting electrolyte: 0.36M HCl; Current density: 30mA/cm^2 Electrode: Pt/Pt ; Stirring rate: 30rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

3.11. Effect of Recycle use of Sodium Chloride.

The most attractive features mediated electrosynthesis are the use of Non-stoichiometric quantities of redox mediator and its recycle. The biphasic electrolysis were conducted for the oxidation of 4-methylbenzyl alcohol to 4-methylbenzaldehyde under the standard reaction condition with continuous recycling of sodium chloride, i.e., the freshly used sodium chloride was recovered, washed and reused for further reactions. Results are reported in Table 8.

Table 8:Effect of recycle spent mediator on the in-direct electrochemical oxidation of 4-methylbenzylalcohol by biphasic electrolysis

S.No	Recycle Use of Spent Mediator	Yield %		Current Efficiency %for the formation of 4-methylbenzaldehyde
		4-methyl benzyl alcohol	4-methylbenzaldehyde	
1	Fresh sample	15	85	63
2	Recycle 1	23	77	64
3	Recycle 2	30	70	55
4	Recycle 3	26	74	51

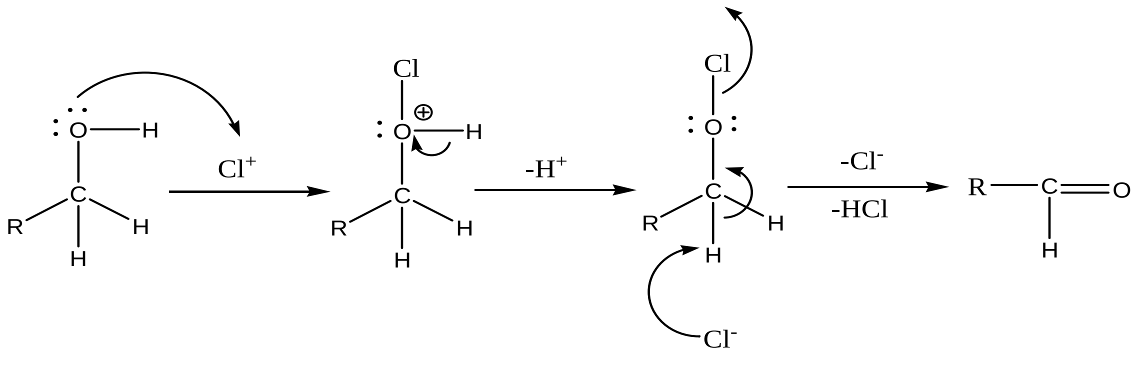
Biphasic electrolysis conditions: Current density = 30 mA/cm^2, Charge Passed: 3F; Anode/Cathode: Pt/Pt (area = 15 cm^2), Electrolyte: 60 mL of aqueous solution of (12%) NaCl and (0.36M) HCl, Solvent: (20 mL) Chloroform, Stirring rate: 30 rpm, Cell volume: 120 mL, Type of cell: undivided glass cell, Temperature: 30-34°C.

A possible mechanism for theoxidation, based on a literature report, is outlined in **Scheme 2** [28].As the electrolysis proceeds; the chloride ion is oxidized at the anode to chlorine which, on hydrolysis, results in the formation of hypochlorous acid and HCl. The unstable hypochloroite forms ClO $^-$ due to its ionic nature which subsequently oxidizes the 4-methylbenzyl alcohol to the corresponding4-methylbenzaldehyde (Scheme 2).

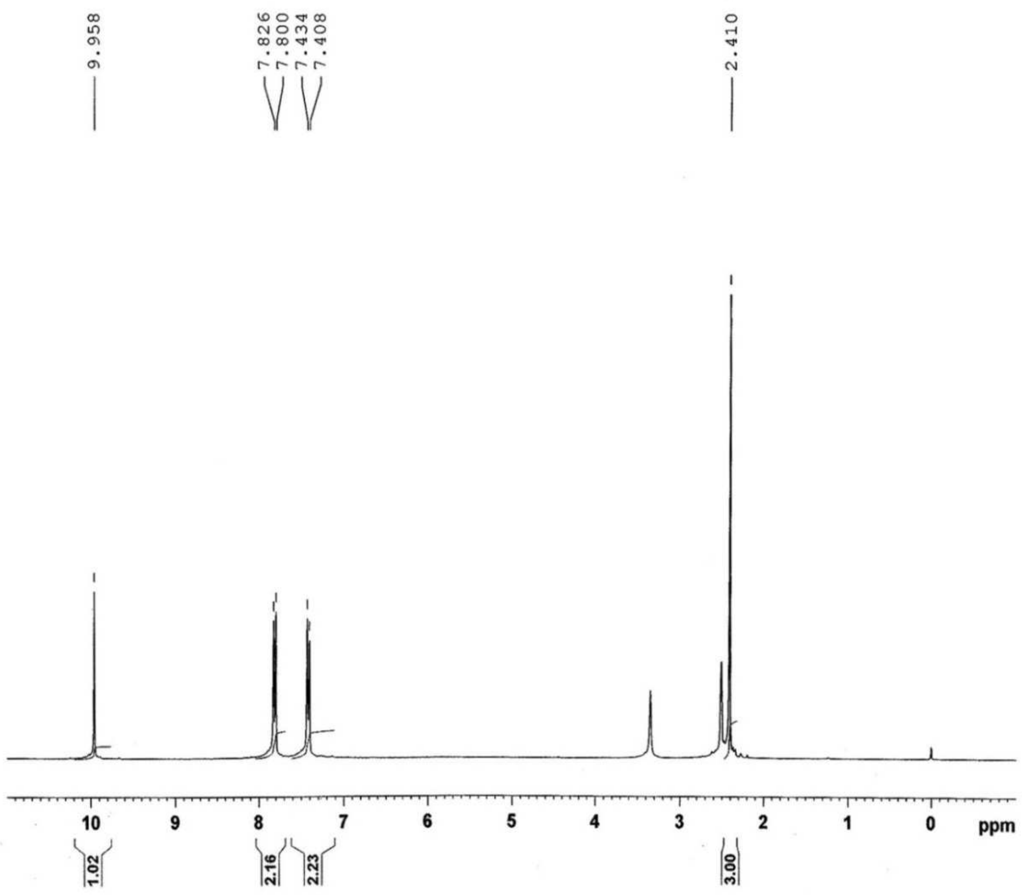
$$2Cl^- \xrightarrow{-2e^-} Cl_2$$

$$Cl_2 + H_2O \longrightarrow HOCl + HCl$$

$$HOCl \xrightarrow{H^+} Cl^+ + H_2O$$

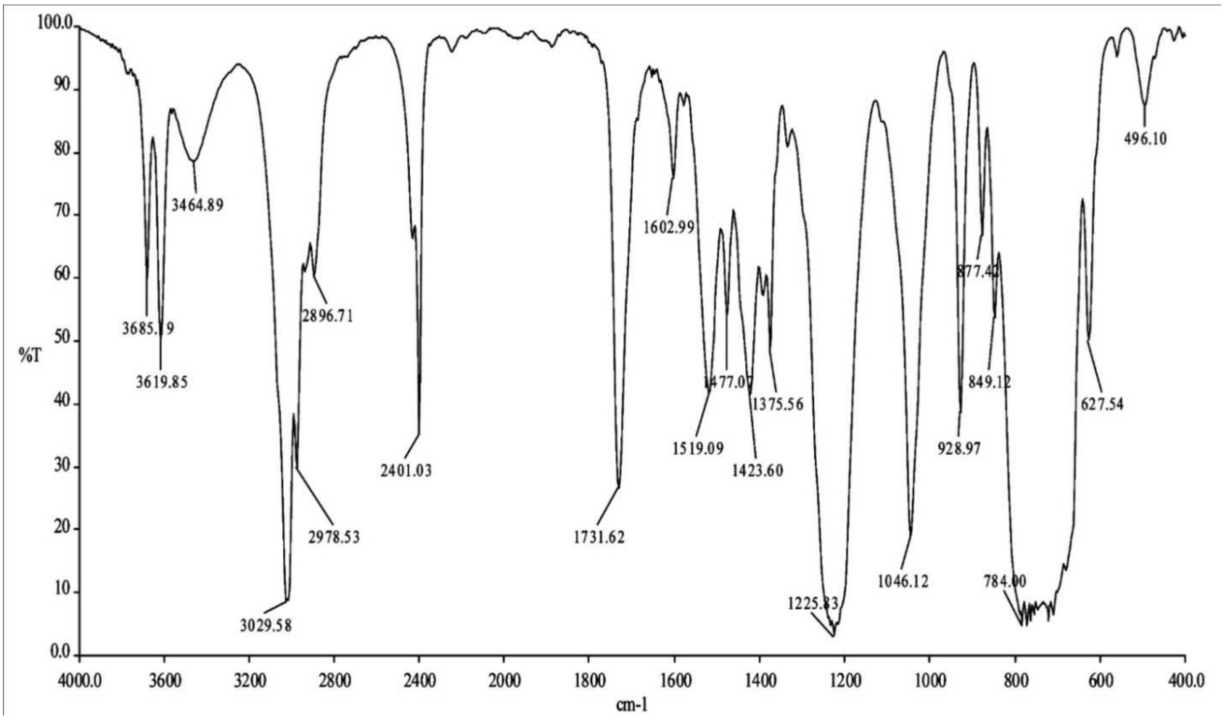


Scheme 2: The Possible mechanism for the biphasic electrolysis of *p*-methylbenzylalchol.



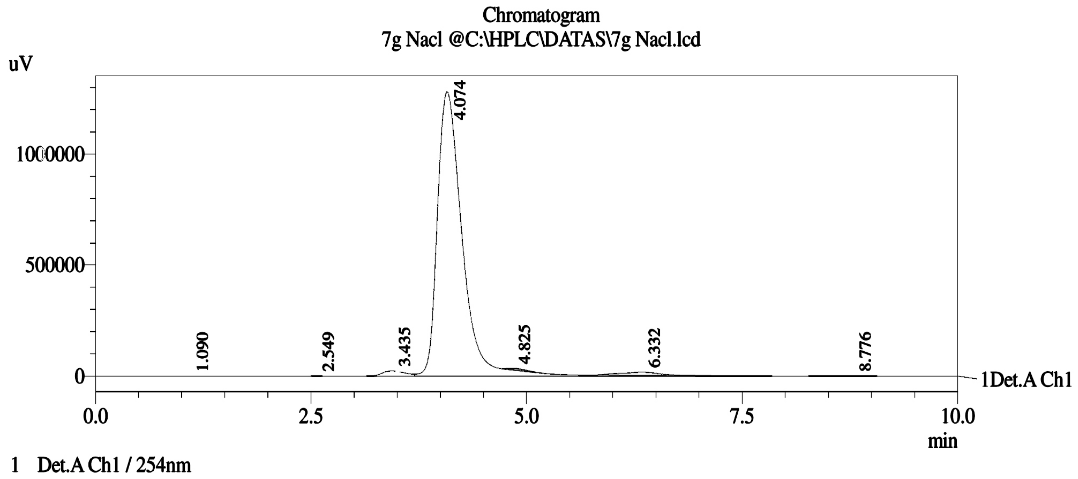
^{1}H NMR spectrum for 4-Methylbenzaldehyde

The spectrum shows the presence of aldehyde protons 4-Methylbenzaldehyde residues at 10.02 ppm. The chemical shifts at 7.93-7.59 are the multiplet protons of benzene ring. These results clearly confirmed that 4-methylbenzaldehyde has been successfully formed.



FT-IR spectrum for 4-methylbenzaldehyde.

The FT-IR spectrum for 4-methylbenzaldehyde shows a broad C-H stretching frequencies between 2978-3029 cm^{-1} represents the presence of aldehyde group (-CHO).The other major absorption band at 1731.62 cm^{-1} could assigned for the intramolecular H-bonding in aryl groups.The other peaks 849,877,928.97 cm^{-1} represents the peaks for c-c double bond at out of plane deformations.

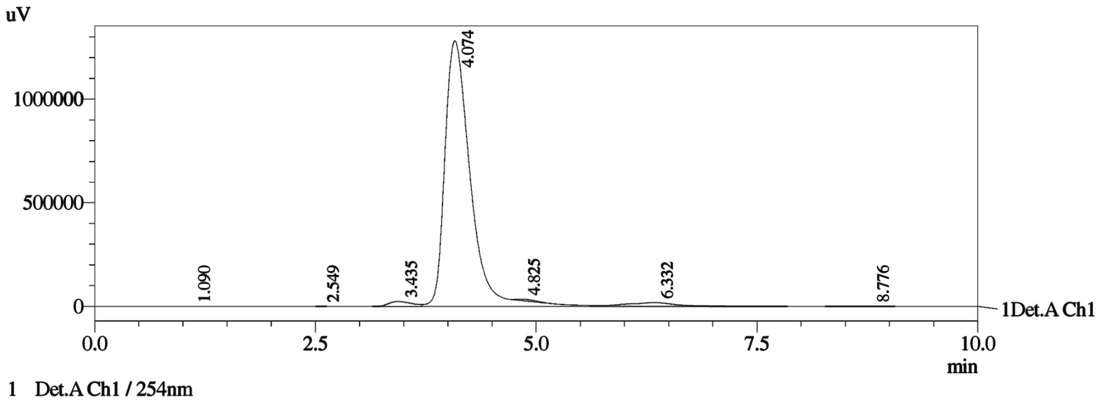


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	1.090	3713	132	0.014	0.010
2	2.549	5627	351	0.021	0.026
3	3.435	421352	22943	1.559	1.730
4	4.074	25991903	1280894	96.179	96.592
5	4.825	98823	5918	0.366	0.446
6	6.332	483632	15316	1.790	1.155
7	8.776	19501	535	0.072	0.040
Total		27024552	1326088	100.000	100.000

HPLC analysis for the Effect of Chloride Concentration for 7g NaCl.



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3	3.435	421352	22943	1.559	1.730
4	4.074	25991903	1280894	96.179	96.592
5	4.825	98823	5918	0.366	0.446
6	6.332	483632	15316	1.790	1.155
7	8.776	19501	535	0.072	0.040
Total		27024552	1326088	100.000	100.000

HPLC analysis for the Effect of the acidic range.

CONCLUSION

The reactions are carried out under mild condition with a precise modest electrochemical setup and present several compensations such as absence of secondary products, low cost of production, high renovation and yield. In conclusion, this anodic method for the oxidation of 4-methyl benzyl alcohol to the corresponding 4-methylbenzaldehyde in excellent yields using *in- situ* prepared hypochlorite ion via biphasic electrolysis. Easy separation of the product, a simple work-up, room temperature reaction conditions, and the reuse of the electrolyte are advantages of this biphasic electrolysis procedure.

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