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Novel electrocatalytic oxidation of benzyl alcohol on various electrodes in a biphasic medium using potassium periodate as a mediator

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Abstract

In a biphasic media, benzoyl alcohol can become the equivalent of benzaldehyde. The selection of the electrode material is crucial in synthetic organic electrochemistry to achieve the optimum yield and selectivity. This material has a significant impact on the thermodynamics and kinetics of electron transport and ultimately determines whether a transformation is effective or not. The reaction was conducted at room temperature in an undivided cell with several electrodes, in a continuous biphasic medium made up of dichloromethane containing benzyl alcohol, an aqueous solution of cyclic potassium as an intermediate, and a little quantity of catalytic sulfuric acid. Platinum electrodes with a high benzaldehyde yield (98%) are produced by the electrochemical oxidation process being optimized using various electrodes.

Keywords: biphasic medium, potassium periodate, electrochemical oxidation, electrodes, benzaldehyde.

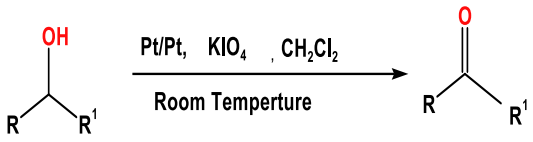
Full-length article **Corresponding Author*, e-mail: sjc_selvaraj@yahool.com

1. Introduction

Electrochemical oxidation of materials is a good substitute for chemical processes since it produces a lot less waste, especially when used on a big scale. Additionally, the electrochemical oxidation of alcohols is widely used in the pharmaceutical and fuel cell sectors synthesis, chemicals[1-4]. Electrochemical oxidation often occurs directly or indirectly. The direct oxidation technique demands high potentials when the majority of the functional groups are activated [5]. To address this the indirect approach was introduced to solve the issue. Methanol and toluene's oxidation potentials demonstrate that Methanol oxidation is more challenging than toluene oxidation. However, benzyl alcohol oxidation is considerably simpler than oxidizing aliphatic alcohols[6]. One of the most significant reactions in pharmaceuticals, dyes, fine chemicals, and prospective fuel reactions. Producing carbonyl compounds through the selective electrocatalytic oxidation of small organic molecules including methanol, ethanol, and benzyl alcohol [7-10]. Such materials may be made fairly easily in an aquatic environment and are sufficiently stable there[11-13]. The generation of artificial organic chemistry reactions needs meticulous attention to parameters that don't seem to be usually encountered by organic chemists as a result of the transport of electrons between the conductor and therefore the liquid part electrolyte. the interpretation is inconsistent, chemistry reactions are often performed in current cells or plots, divided or undivided cells, whereas optimizing the applied current density or potential across a cell. the foremost significant change, however, is at the electrodes, because the material has a major impact on the success or property of a given transformation. the space between the conductors,

their form and size conjointly have an effect on the submerged surface, the uniformity of the sector and as a result, this density; All of those factors will affect the tactic of lepton transfer, which can affect the reaction results. whereas the electrode substance is another parameter that has to be optimized, it is often accustomed tune and altering the property of the reaction and permits the electrode catalyst to alter the reactivity contact electrocatalyst, ready by a chemicals changed electrocatalyst or intermediate. The electrode material is critical for such a procedure. Consequently, the platinum electrode is the best option due to both its excellent electrical conductivity and its ability to resist corrosion [14]. In addition to its characteristics, platinum is effectively employed in a variety of sectors, including electronics, organic compounds and fuel cell oxidation[15]. Though its usage is sometimes constrained by its comparatively expensive cost. Numerous studies have focused on the electrocatalytic oxidation of alcohols over different catalysts, which are primarily based on platinum[16]. One of the finest anodic materials with catalytic characteristics enabling alcohol oxidation to occur at a suitable pace in fuel cells is platinum[17]. Numerous oxidizing substances have been observed to oxidize benzyl alcohols[18-23]. Recently created an In-Cell approach for biphasic electro-oxidation of benzyl alcohol, which is mediated by potassium periodate[24-25]. Simplicity of reaction conditions, low cell voltage, undivided cell, constant current electrolysis with affordable DC power supply, low cost, ease of expansion and virtually no waste problem are the advantages of the proposed technique. The electrochemical oxidation of alcohol to aldehydes or ketones is currently mediated by IO_3/IO_4. After electrolysis is finished, the organic layers are separated and evaporated to produce products.

The present work aims to explore the electrolytic activity of several different metal electrodes in the direction of electrooxidation of benzyl alcohol by a two-phase approach and the electrolytic deposition of benzyl alcohol on different electrodes, using the potassium cycle as an intermediate in experimental cases. The required components for this electrolysis are benzyl alcohol as a substrate, a supportive potassium cyclic electrolyte, and a small amount of organic solvent. To achieve efficient electrochemical oxidation, no additional materials such as catalysts, intermediates, oxidants or ligands are required.



Pt = Platinum electrodes, R = Aryl,substituted aryl,benzyl, Ph-CH=CH-, R^1 = H

Scheme 1. Electro oxidation of alcohols in biphasic medium

2. Materials and methods

In a single-chamber electrolytic cell, collect a solution of benzyl alcohol (10 mmol), 1.008 g of potassium periodate, and dichloromethane (20 mL) and add an aqueous solution (2.3 mmol) containing 1.36 M (concentrated sulfuric acid) (80 mL). Added if given the solution above. The aqueous phase contained 15 cm 2 of numerous metallic and non-metallic electrodes, some of which were close to the interface but not touching the organic phase. The organic phase itself was stirred at a speed of 40 rpm using a magnetic stirrer. Avoid contact of the electrodes with the biological layer. Galvanostatic electrolysis was performed at room temperature, 4 F/mol temperature and a current density of 30 mA/cm². The aqueous layer was reused for the next reaction. Aliquots were removed periodically from the organic phase and subjected to HPLC analysis using a stationary phase SHIMADZU LC-8A column (250 mm x 4.6 mm). Dichloromethane and water were used as eluents at a flow rate of 1 mL/min. Samples were examined using a UV detector with a wavelength of 254 nm. The lower organic phase was isolated after electrolysis, washed with water (2x25 ml), dried over anhydrous $Na_2SO_4$and the solvent is removed by distillation. HPLC analysis of the residue showed the presence of benzaldehyde in 98% yield. The incomplete product was passed through a silica gel column and eluted with a combination. To obtain a 95% pure product use ethyl acetate/n-hexane (1:9)

3. Results and Discussions

3.1. The parameters studied to optimize the conditions:

To improve product yield, the following electrochemical parameters were analyzed for optimization Conditions and results are presented in Table 1.

3.2. Effect of various electrodes on the oxidation of benzyl alcohol:

When looking for favorable reaction conditions, one should start with the electrode to see if it is inert to the reduction/oxidation potential of the substrate. Pt/Pt is a better electrode choice than any other for the conversion of benzyl alcohol to benzaldehyde. Graphite/graphite, S.S/S.S,Cu/Cu, zinc/zinc, and tin/tin electrodes can degrade during processing, reducing yield. Various types of electrode pairs shown in Table 2 are used in two-phase electrolysis for the electrochemical oxidation of benzyl alcohol. Effects of different electrodes on electrochemical synthesis of benzyl alcohol at room temperature

Reaction conditions:

Electrode - Anode/Cathode
Organic layer - Benzyl alcohol (10 mmol) + CH_2Cl_2(20 ml)
Aqueous layer - KIO_4(1.2g,10 mmol) +10.2mmol H_2SO_4 in H_2O (80ml)
The crucial factor in the oxidation of benzyl alcohol is electrode variation. Anode and cathode pair effects on product yield were researched. When using different anode and cathode material combinations for electrooxidation, such

as Pt/Pt, platinum/graphite, Pt/S. S, S.S/S.S, Cu/Cu, zinc/zinc, and graphite/graphite (Fig: 2) the product yield varied marginally .

The earlier report specifically highlights the advantages of using platinum as an anodic material for electrolytic conversion[26-27]. However, platinum is a very expensive material, and it has been discovered that using carbon as an anodic material for electrolytic conversion results in yields that are comparably lower than those of the present investigation. Platinum is often used as a counter-electrode material, but recent developments in our knowledge of Pt electrochemistry and electrocatalysis show that it can experience gradual but inevitable electrochemical and chemical deterioration under certain circumstances. This is significant in non-elegant (non-precious) electrode research electrochemical energy storage and production materials tiny quantities of electrodeposited Pt can change the electrocatalytic characteristics of new-generation technologies. improper information might provide erroneous outcomes and incorrect. For measurements under high pressure and in a variety of electrochemical systems, including aqueous, non-aqueous, gel, or frozen electrolytes, Pt electrodes serve as suitable reference electrodes. Peak separation results from the study of single or multiple ones- or two-electron redox systems show that Pt may be utilized consistently as a reference electrode under a range of circumstances[28]. Because platinum is resistant to oxidation and does not react easily, it is used in electrochemical cells as an electrode because it does not participate in the redox processes that take place there. Inert metal platinum can rapidly absorb hydrogen. Unlike other metals, platinum electrodes do not participate in redox processes during cellular activity. Therefore, from the results described above, we conclude that the best and most promising electrodes for benzyl alcohol conversion are the platinum anode and the platinum cathode. The percentage of the yield of the product is shown in chart 1.

3.3. Effects of recycling Potassium periodate:

The reusability of potassium periodate for the oxidation of benzyl alcohol (10 mmol) as a modal substrate was investigated in this case, where both anodic and cathodic reactions were conducted in an undivided cell (biphasic medium). The aqueous phase was recycled for another cycle after the electrolysis. Almost comparable yields (95–98%) were achieved. The effects of recycling potassium periodate are shown in Table 3 and chart 2.

3.4. Benzylic alcohols are electro-oxidized in a biphasic medium:

We have performed oxidative electrolysis of different alcohols to extend the use of cyclic potassium oxidation electrolysis of benzyl alcohols. The results are shown in Table 4. A good product (98%) was produced by the electrochemical oxidation of benzyl alcohol using cyclic potassium as an intermediate in a two-phase system. This two-phase approach converted the substituted alcohols into corresponding carbonyl compounds with high single-chamber yields. The yields of different products are shown in Table 4. Various benzyl alcohols have been subjected to homogenous electrolysis at room temperature in a single cell to evaluate the range of this optimized condition and to check the generality of the method. The corresponding results are listed in Table 3 with their respective results. Benzyl alcohol (entry 1) was smoothly oxidized under ideal reaction circumstances to produce only the necessary aldehyde, with no extra oxidation to acids. While the yield was 78, 80 and 82% for benzyl alcohols replaced with an electron-withdrawing substituent like Chloro, Bromo, Fluro (entry 2,3,4) the energy requirement was 3F/mol. With 3 F/mol of current, electron-donating substituents such as the methoxy and methyl groups (entry 5,6) on the phenyl ring have produced high yields of substituted benzaldehyde without affecting the side chain of the C=C double bond, cinnamyl alcohol (entry 7) underwent biphasic electro-oxidation to give substantial yields of cinnamaldehyde.

3.5. Characterization of the Product:

The product obtained after electrolysis was characterized by different spectroscopic analyses such as UV, FTIR, and ^{1}H-NMR.From HPLC data the percentage of product yield was calculated.

Spectral details of benzaldehyde

Yellowish-brown oil yields 98%

HPLC retention time of benzaldehyde: 120 min.

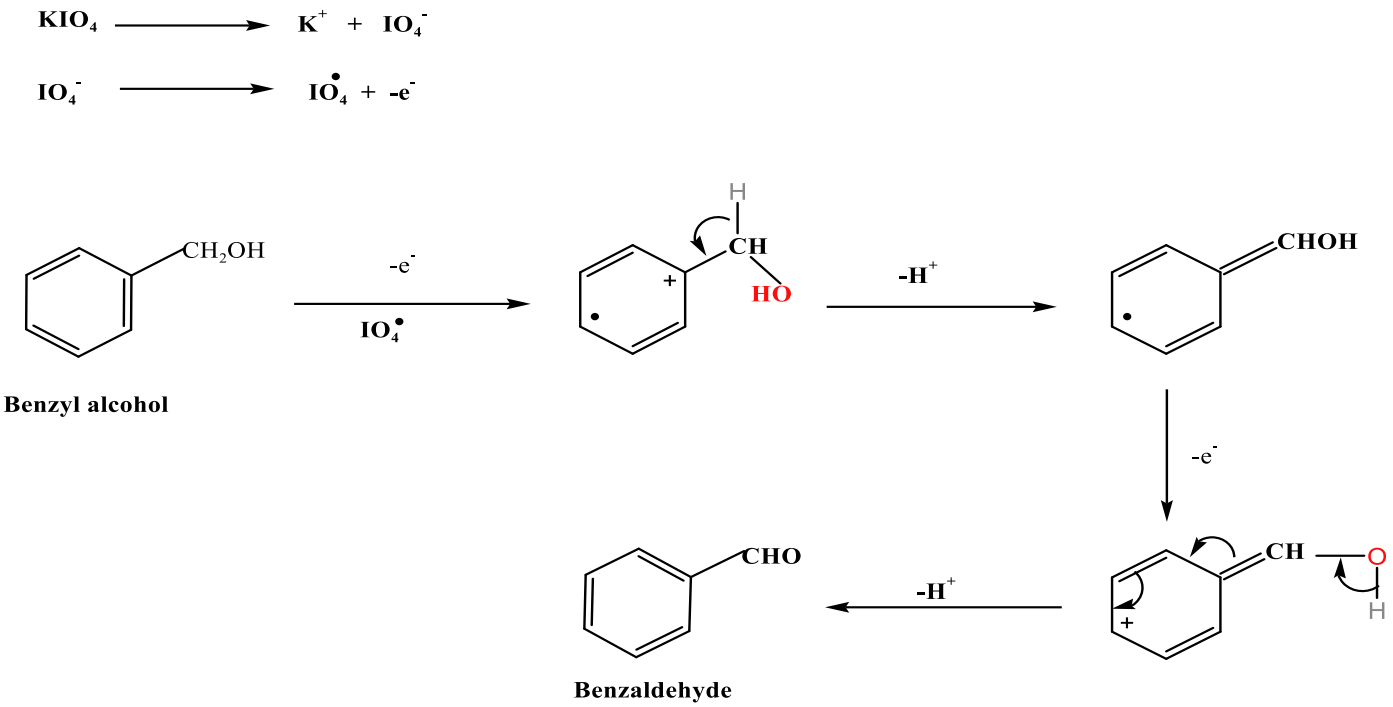
UV Spectrum: The maximum wavelength λ_{max} at 244 nm

FTIR Spectrum: 1696(alpha beta-unsaturated aldehyde) 2000 (C=O stretching frequency) 2745-2827 (aldehydic C-H Stretching) 3073 (aromatic C- H stretching)

^{1}H-NMR: 400 MHz, $CDCl_3$ δ: 10.02 (ppm) (1H, S) (O=C-H) 7.90-7.87(2H, t, benzene-H) 7.66-7.61(1H,t benzene=H)7.56-7.51(2H,t,benzene-H)

3.6. Proposed mechanism

The scheme below depicts a potential process for the electrocatalytic oxidation of benzyl alcohol to benzaldehyde in a biphasic medium[29].



Scheme 2: Mechanism for the biphasic electrooxidation of benzyl alcohol

Table 1: Parameters studied to optimize conditions

S. No	Parameters	Variation Studied	Optimum Condition
1	Current Density	20 to 60 mA\Cm^2	40 mA\Cm^2
2	Charge Passed	1 to 5 F\mol	3 F/mol
3	Medium	Acidic, Basic, Neutral	Acidic
4	Solvent	$CHCl_3$, CH_2Cl_2 $C_2H_4Cl_2$, CCl_4	CH_2Cl_2
5	Electrode Combination	Pt\Pt,Cu\Cu, Zn\Zn,C\C, Sn/Sn S.S/S.S	Pt\Pt
6	Agitation	20 to 60 rpm	40 rpm
7	Mediator	KI, KIO_3, KIO_4	KIO_4

(S.S- Stainless Steel)

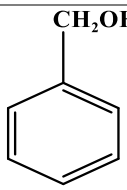
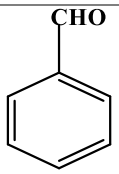
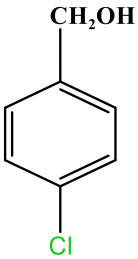
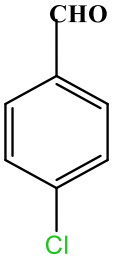
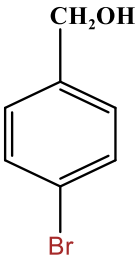
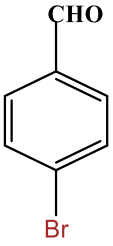
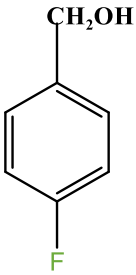
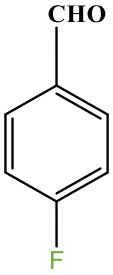
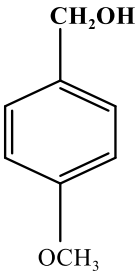
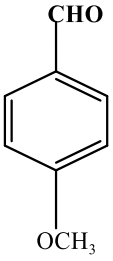
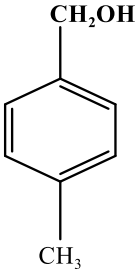
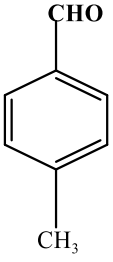
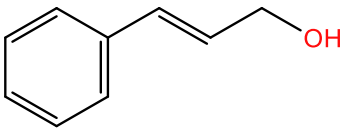
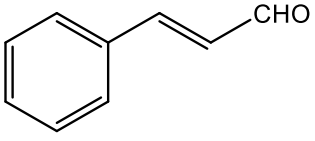
Table 2: Effect of various electrodes

S. No	Anode	Cathode	Time (min)	Benzaldehyde Yield (%)	Charge passed F/mol
1	Platinum	Platinum	120	98	3
2	Platinum	Graphite	90	92	2.5
3	Graphite	Graphite	90	95	2.5
4	Graphite	Pt	90	92	2.5
5	Graphite	S. S	90	90	2.5
6	Pt	S. S	90	89	2.5
7	S. S	S. S	90	93	2.5
8	S. S	Pt	90	89	2.5
9	Pt	Cu	60	66	2.0
10	Cu	Cu	40	65	1.5
11	Pt	Zinc	40	59	1.0
12	Zinc	Zinc	40	52	1.0
13	Tin	Tin	40	26	1.0

Table 3. Effect of Reuse

S. No	Amount of Potassium periodate (mmol)	Benzaldehyde Yield (%)
1	1.2	98
2	Reuse 1	97
3	Reuse 2	96
4	Reuse 3	95

Table 4. Effect of various Benzyl alcohols

Entry	Substrate	Product	Yield (%)	Time (min)	Charge Passed (F/mol)
1			98	120	3.0
2			78	90	3.0
3			80	90	3.0
4			82	90	3.0
5			88	60	3.0
6			90	75	3.0
7			92	60	3.0

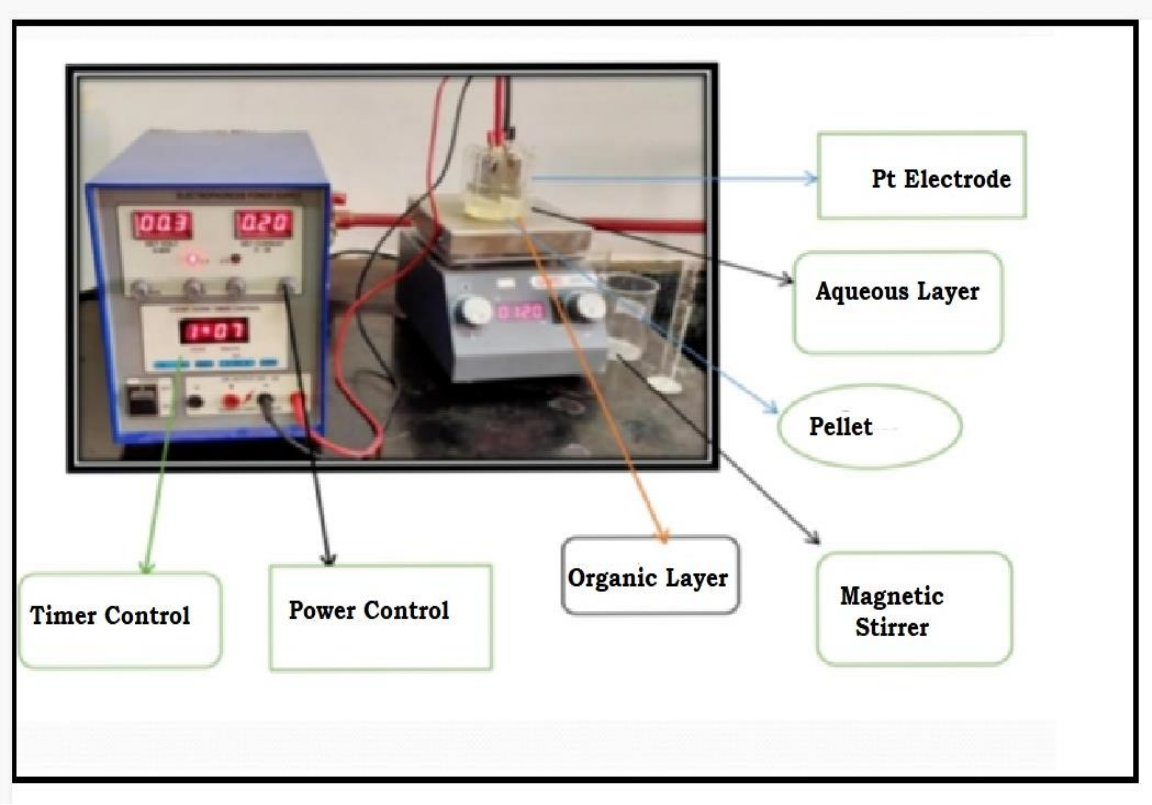


Figure 1. Instrumentation for electrochemical oxidation of benzyl alcohol

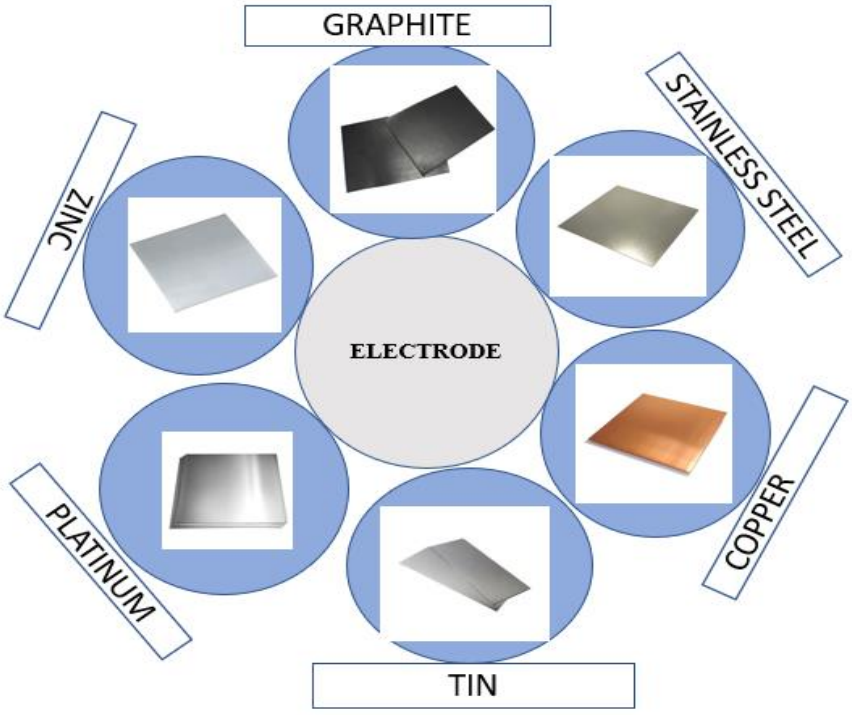
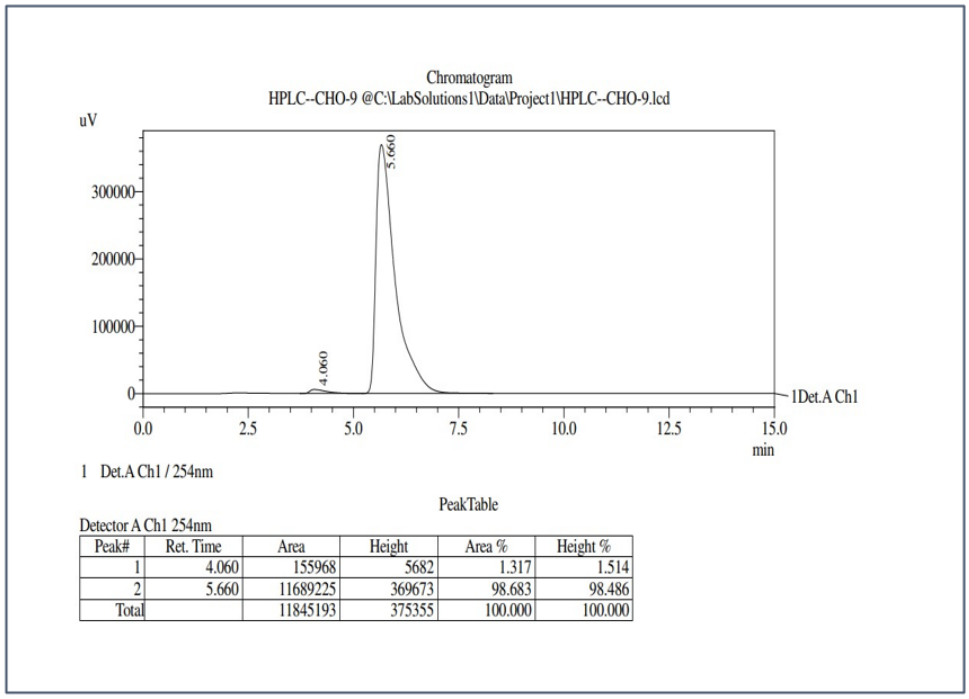


Figure 2. Image of the various electrodes



PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.060	155968	5682	1.317	1.514
2	5.660	11689225	369673	98.683	98.486
Total		11845193	375355	100.000	100.000

Figure 3: HPLC data for benzaldehyde

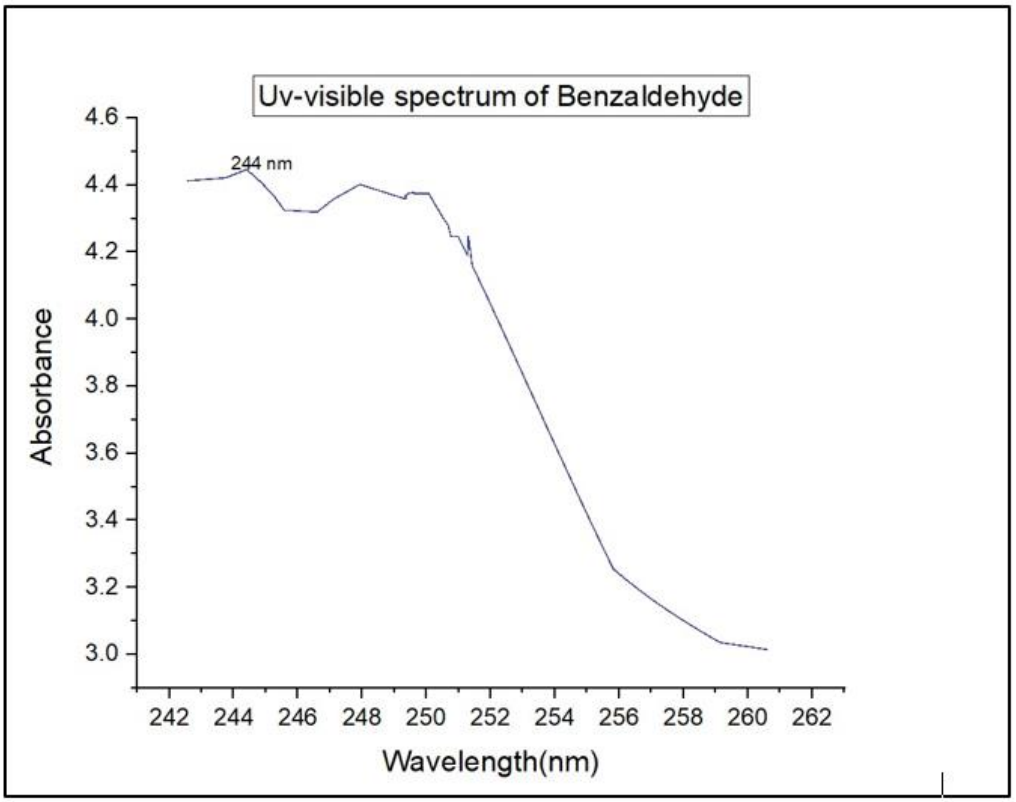


Figure 4: UV spectrum of Benzaldehyde

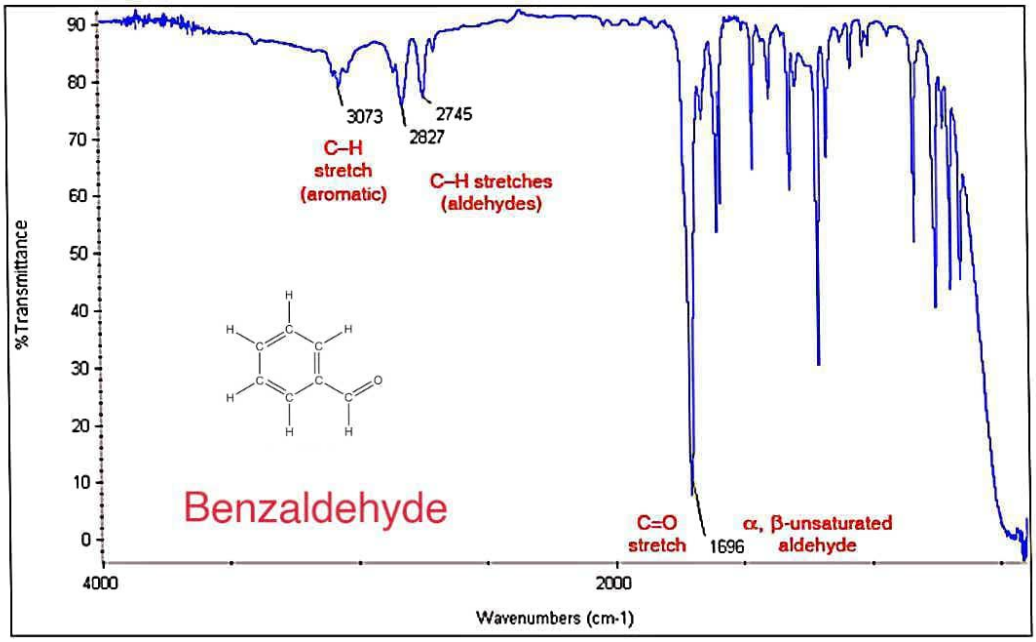


Figure 5: FTIR Spectrum of benzaldehyde

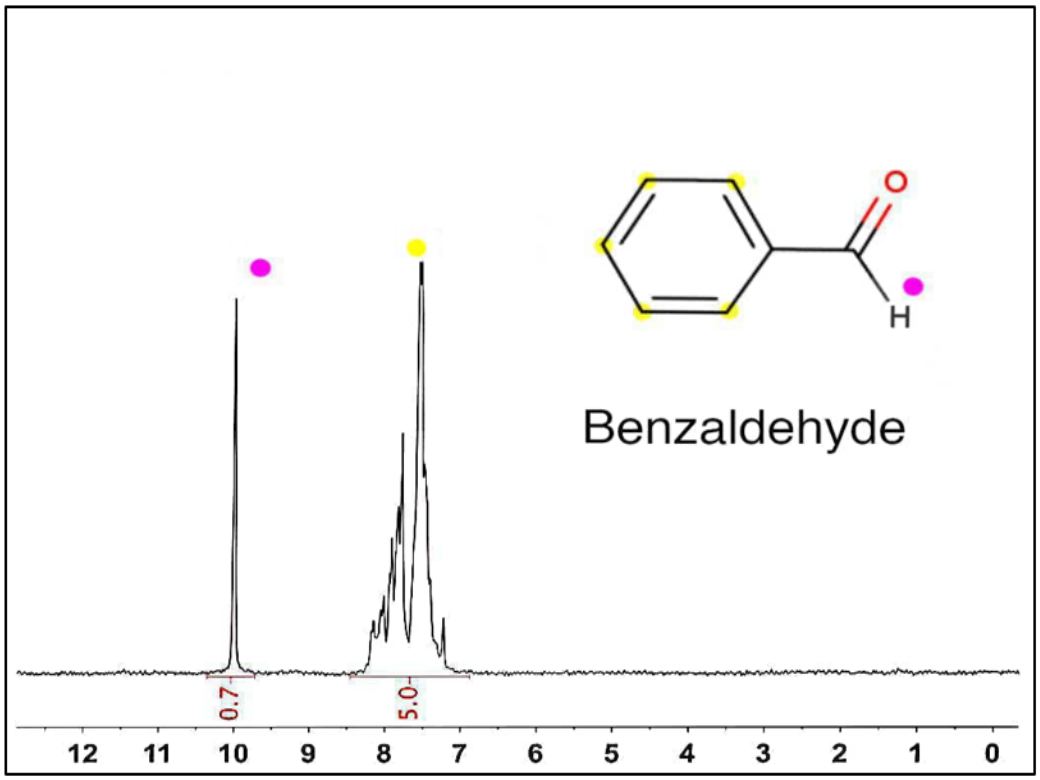
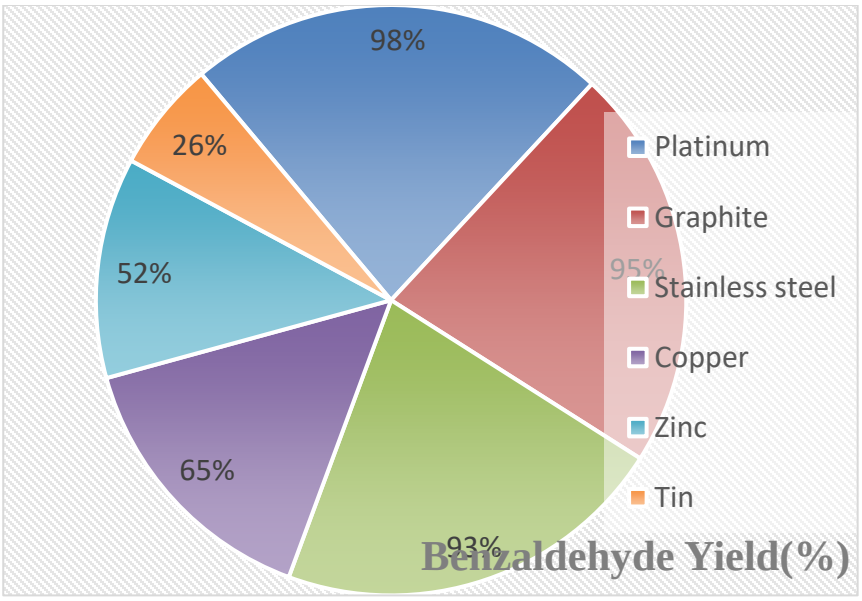
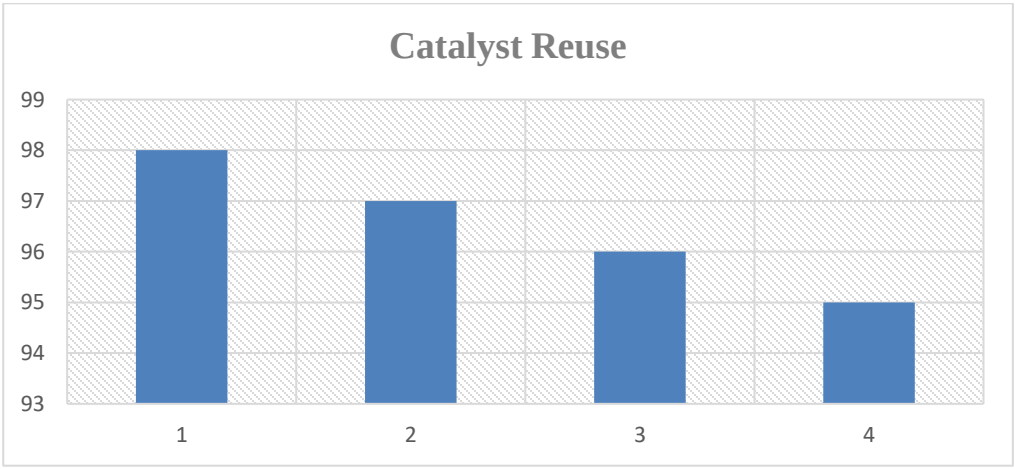


Figure 6: [1] H-NMR Spectrum of benzaldehyde



Charts 1. Effect of various electrodes



Charts 2. Reusability of Potassium periodate for the electrochemical oxidation of benzyl alcohol

4. Conclusions

We have demonstrated that the potassium cycle is a useful intermediate for the electrochemical oxidation of alcohols in a biphasic environment promoting gradual oxidation and selective generation of the corresponding carbonyl products. Potassium can also be used repeatedly with only a small reduction in product yield. The fundamental advantage of our method is that we have used a platinum electrode and a two-phase medium which simplifies the separation of the results. Clarifying trends is often the result of efforts to understand the impact of electrode materials using analytical electrochemistry and physical organic chemistry approaches. Increased efficiency and new possibilities will come from the broader adoption of these trends and insights. Even if the decision is informed in principle, testing may still be necessary due to the complexity and variety of electrode-substrate interactions in organic processes. On electrolysis of potassium periodate, an iodate ion is produced that allows the benzyl alcohols to be converted to their respective aldehydes. The current approach has the following advantages: simple test setup; inexpensive electrodes and iodate salts; ambient temperature; quick response time; maximum use of solvent-based green water; and no by-products. The aforementioned capabilities also lead to rapid oxidation without oxidants and electrochemical regeneration with excellent current efficiency at high current densities.

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