Electrochemistry Communications 124 (2021) 106944

Contents lists available at ScienceDirect

Electrochemistry Communications

journal homepage: www.elsevier.com/locate/elecom

Au-catalyzed electrochemical oxidation of alcohols using an electrochemical column flow cell

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ARTICLE INFO

Keywords:
Electrosynthesis
Electrochemical oxidation
Alcohol oxidation
Au catalyst
Electrochemical column flow cell

ABSTRACT

A novel green system for the electrochemical oxidation of alcohols is demonstrated using a column flow cell. Voltammetric analysis revealed that the oxidation of 1-phenylethanol and benzaldehyde are promoted by using both an Au-electrode and an alkaline medium. To conduct such reaction with a column flow cell, we developed a method to modify a carbon-fiber thread with Au nanoparticles. The column carbon-fiber thread electrode modified with Au nanoparticles showed a high surface area, enabling the efficient electrochemical oxidation of various alcohols.

1. Introduction

The oxidation of alcohols is a fundamentally important class of reaction at both the laboratory and commercial scale [1,2]. Classical alcohol oxidations have been carried out using stoichiometric or excess amounts of oxidants such as hypochlorite, chromium, manganese, chlorine gas, and peroxides [3]. These oxidants are hazardous, corrosive, and/or toxic and also result in an equimolar amount of chemical waste. Hence, alternative environmentally benign strategies for the oxidation of alcohols are desired from the viewpoint of sustainability. Oxidation with heterogeneous or homogeneous catalysts using O_2 as a terminal oxidant has been developed, with excellent examples reported in the literature [1,4–6]; however, achieving high levels of productivity, durability, chemoselectivity, and sustainability is difficult. Another option for the development of a green oxidation process is electrochemistry, where redox reactions occur at the electrode surface, driven by electric energy instead of by chemical reagents. When the cathodic reaction is a proton reduction reaction, hydrogen gas is generated as the sole byproduct. In addition, the use of electricity generated from renewable sources makes the whole process even greener; thus, the electrochemical oxidation of alcohols is regarded as an ideal sustainable system.

To enable the efficient electrochemical oxidation of alcohols, researchers have developed numerous systems that use electrocatalysts. Aminoxyl radicals, as a homogeneous electrocatalyst, have been extensively used for the production of complex and valuable chemicals such as pharmaceuticals and natural products [7]. On the other hand, heterogeneous transition-metal electrocatalysts are also being actively investigated because of their high productivity and durability.

Gold (Au) is a representative heterogeneous electrocatalyst for alcohol oxidation and has been investigated for decades [8,9]. As an electrocatalyst, Au enables the oxidation of alcohols with a small overpotential under alkaline conditions via the on-surface formation of adsorbed hydroxide ($Au(OH)_{ads}$) [8–10]. Zhu and coworkers observed $Au(OH)_{ads}$ species using *in situ* liquid secondary-ion mass spectrometry and elucidated the critical role of $Au(OH)_{ads}$ in the catalysis of alcohols. They also reported that Au oxide (Au—O—Au), which is generated at higher potentials, exhibits no catalytic activity toward alcohol oxidation [11]. Xu and coworkers showed that the peak intensity of $Au(OH)_{ads}$ in attenuated-total-reflectance surface-enhanced infrared absorption spectra increased with increasing potential, reached a maximum at a certain potential, and then gradually decreased at higher potentials [12].

In the present study, we report a novel green system for the electrochemical oxidation of alcohols. Electrochemical measurements using an Au electrode and a carbon electrode modified with Au nanoparticles (NPs) were performed with various alcohols, including benzylic, allylic, and aliphatic alcohols with primary or secondary hydroxy groups, to confirm the general catalytic activity of Au. To scale up the system, we used a continuous-flow column cell (Fig. 1), which was originally developed for the highly sensitive coulometric analysis of trace chemical substances. Although the application of a column flow cell for preparative-scale electrosynthesis is rare [13–16], the extremely large electrode surface area of the system seems suitable for the large-scale

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https://doi.org/10.1016/j.elecom.2021.106944
Received 21 December 2020; Received in revised form 9 January 2021; Accepted 18 January 2021
Available online 28 January 2021

oxidation of alcohols. An Au NP-modified carbon-fiber-thread electrode worked well as an electrocatalyst for alcohol oxidation in a column flow cell, resulting in a small overpotential, high productivity and unique selectivity were confirmed in preparative-scale experiments.

2. Results and discussion

2.1. Voltammetric study with an Au disk electrode and plausible reaction mechanism

First, CV measurements were performed in various electrolyte solutions using an Au disk electrode (Fig. 2, Fig. S1). Under conditions where neither base nor alcohol was present, no oxidation peak was observed (Fig. 2a). Water oxidation did not occur at 1.5 V (*vs* Ag/AgCl) under neutral conditions because of the high overpotential of the oxygen-evolution reaction (OER) at the Au electrode [17]. When 1-phenylethanol was added to the neutral solutions, direct alcohol oxidation did not occur (Fig. 2b). The alkaline background solution, which did not contain 1-phenylethanol, showed an oxidation current in the potential region beyond 1.2 V (Fig. 2c), which we attributed to the OER, concomitant with the formation of Au oxide (Au—O—Au) [11]. A small cathodic current was observed in the reverse scan at ~0 V, presumably due to the reduction of the Au oxide. When both 1-phenylethanol and KOH were present, three characteristic oxidation waves (A–C) were observed (Fig. 2d). Peak **A** was the first oxidation peak observed during the measurement, with an onset potential of approximately −0.4 V, demonstrating a substantial decrease in the overpotential for alcohol oxidation under catalysis by the Au electrode. This anodic current originated from the electrocatalytic oxidation of both 1-phenylethanol and hydroxide via the formation of $Au(OH)_{ads}$ species, as also proposed for the electrochemical oxidation of ethanol [10,11]. The anodic current substantially decreased at 0.55 V, presumably because of the on-surface formation of catalytically inactive Au—O—Au species. Peak **B** was attributed to the OER, in accordance with the results observed for the background solution. Oxidation current **C** appeared at ~0 V, which is a potential where the reduction of Au—O—Au was observed (Fig. 2, blue line). Thus, peak **C** was assigned to the oxidation of 1-phenylethanol. The reappearance of this peak was concomitant with the regeneration of $Au(OH)_{ads}$ species upon reduction of Au—O—Au. Similar voltammetric behavior was also observed for benzaldehyde (Fig. S2).

The voltammetric data for 1-phenylethanol and benzaldehyde suggest that the oxidation of these compounds likely follows a mechanism similar to that previously proposed for ethanol oxidation (Fig. 3) [10]. Under basic conditions, alcohols are deprotonated to give alkoxides in the electrolyte. The alkoxides and hydroxide ions are oxidized on the electrode and adsorbed onto the surface of the Au NPs. Aldehydes are generated upon further oxidation, potentially via H-atom abstraction from the adsorbed substrates by $Au(OH)_{ads}$. The aldehydes are in equilibrium with *gem*-diols under alkaline conditions, and *gem*-diols undergo further oxidation via the same mechanism to give carboxylic acids.

2.2. Au nanoparticle modification of a carbon-fiber thread

To enable the electrocatalytic oxidation of alcohols in a column flow system, we decorated the Au NPs onto a carbon-fiber thread electrode. Although Sakata and coworkers [16,18] have reported modifying carbon-fiber thread electrodes with Ag, Cu, and Zn, they did not characterize the particles microscopically. Thus, we optimized the method to fabricate Au NPs on a carbon-fiber thread inserted into a column flow cell.

We first investigated the direct electrodeposition of Au onto a pristine carbon-fiber thread (Fig. S3) [19]. The electrochemical deposition was carried out via a one-pass potential-sweep method with the electrolyte introduced at flow rates of 1.0, 3.0, and 5.0 mL min^{-1} and using 0.5 M H_2SO_4 (aq) containing 5 mM $NaAuCl_4{\cdot}2H_2O$. UV–vis analyses were performed on the outlet solution to confirm the conversion of chloroauric acid ions (Fig. S4). At flow rates<3.0 mL min^{-1}, no $AuCl_4^-$ anion was detected, indicating that the carbon fiber near the outlet was

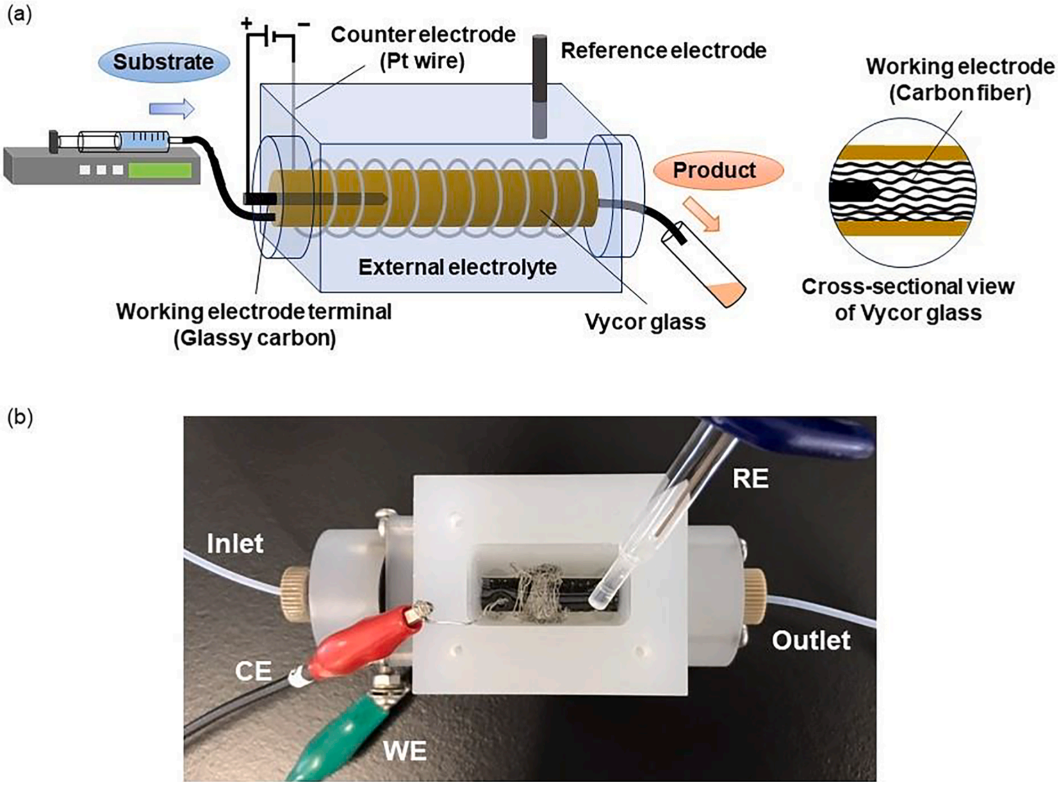


Fig. 1. Structure of an electrochemical column flow cell. (a) Flow scheme of an electrosynthetic reaction in the column flow cell. (b) Photograph of an assembled column flow cell (top view).

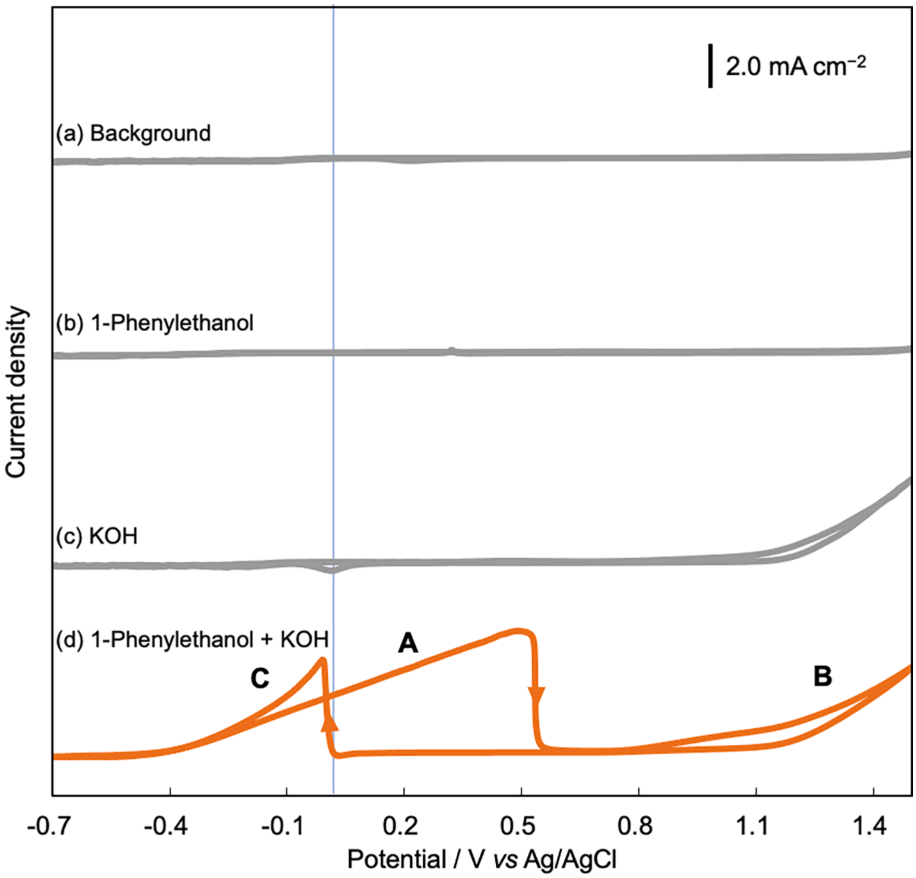


Fig. 2. Cyclic voltammograms recorded at a scan rate of 10 mV s^{-1} with an Au disk electrode using water/acetonitrile solution (80/20 in vol%) containing 200 mM $LiClO_4$: (a) background solution containing no additional substrates; (b) 50 mM of 1-phenylethanol; (c) 50 mM of KOH; and (d) 50 mM of 1-phenylethanol and 50 mM of KOH.

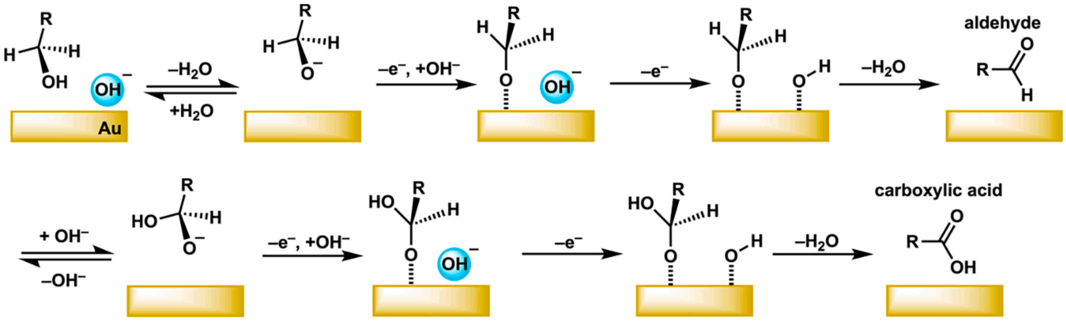


Fig. 3. Plausible reaction mechanism for the oxidation of a primary alcohol at an Au electrode in an alkaline medium.

not sufficiently modified. Therefore, a cycle method was adopted with a flow rate of 5.0 mL min^{-1}. The effect of the sweep rate on the particle size was confirmed by scanning electron microscopy (SEM) analysis. SEM images show that the smallest average particle size (96 nm) and narrowest size distribution were obtained at a sweep rate of 0.5 V s^{-1} (Fig. 4, top and middle; Fig. S5). However, the extent of modification remained small.

To increase the loading amount of Au NPs, we developed a new methodology—an electrochemical seed-mediated growth method—to modify a carbon-fiber thread with Au NPs. This method is based on a combination of the electrochemical deposition method and the seed-mediated growth method [19–21]; specifically, it is a two-step method consisting of the physical absorption of Au nanoseeds followed by the electrochemical reduction of $NaAuCl_4 \cdot 2H_2O$ for the further growth of Au NPs (Fig. S3, bottom). Field-emission scanning electron microscopy (FE-SEM) images of the Au NP-modified column electrode show that the Au NPs were uniformly distributed after the modification, whereas the carbon fibers remained intact (Fig. 4, bottom). Energy-dispersive X-ray spectroscopy (EDX) analysis confirmed that the particles in the SEM images contained Au, suggesting the formation of Au NPs (Fig. S6a). The average size of the Au NPs was 159 nm (Fig. S6b). Electrochemical measurements revealed that the total surface area of the Au NPs was 1032 cm^2, which represents an extremely large catalytic area (Fig. S6c) [22].

2.3. Au-catalyzed electrochemical oxidation using a column flow cell

2.3.1. Cyclic voltammetry study in column flow cell

To validate the electrochemical behavior of the Au NP-modified electrode, cyclic voltammetry (CV) measurements were performed in

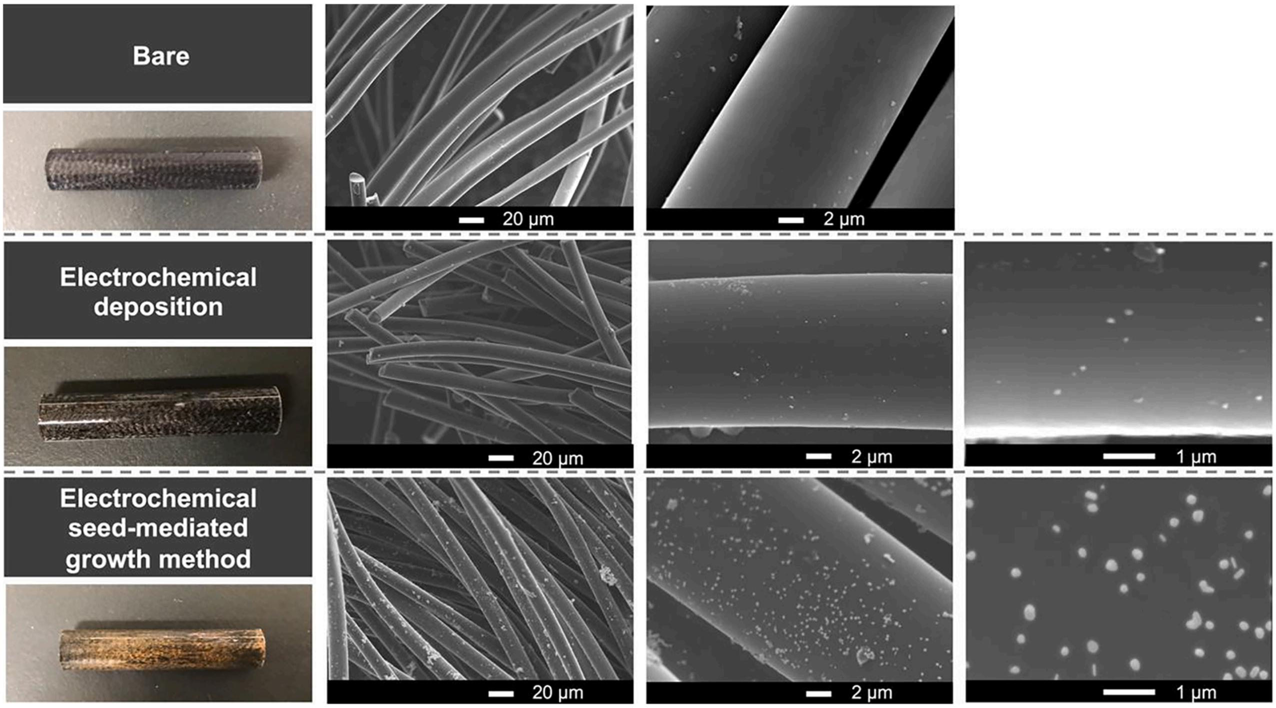


Fig. 4. Pictures and SEM and FE-SEM micrographs of a bare column electrode and Au NP-modified column electrodes prepared with the electrodeposition method and the electrochemical seed-mediated growth method; the images correspond to different magnifications.

the column flow cell with water/acetonitrile solution (80/20 in vol%) containing 200 mM $LiClO_4$, 50 mM KOH, and 50 mM alcohol (Fig. 5). Measurements were performed in the potential region more negative than 0.4 V to avoid the formation of catalytically inactive Au—O—Au species. Benzylic, allylic, or aliphatic alcohols with a primary or secondary hydroxy group were used for the measurements. Only small oxidation waves associated with the direct oxidation of alcohols were observed for the pristine carbon-fiber thread electrode (Fig. 5, left). By contrast, substantial enhancements in the anodic current were observed for all of the substrates when the Au NP-modified electrode was used (Fig. 5, right), suggesting electrocatalytic oxidation. Koper et al. have reported that the onset potentials for electrocatalytic oxidation of

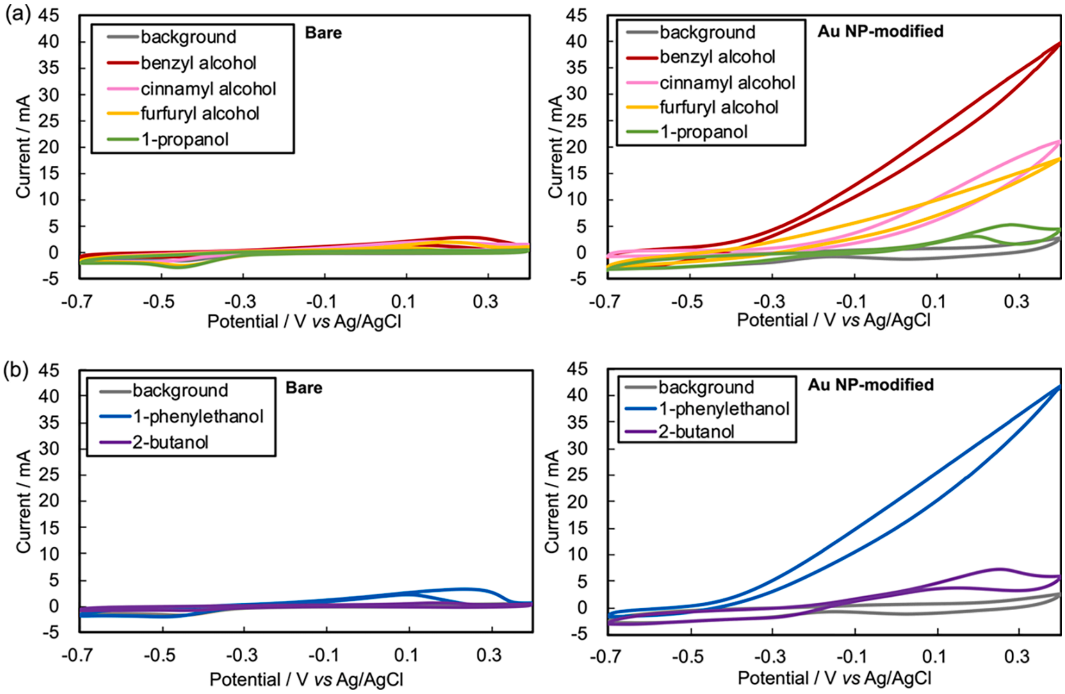


Fig. 5. Cyclic voltammograms of the electrochemical oxidation of (a) primary alcohols (benzyl alcohol, cinnamyl alcohol, furfuryl alcohol, and 1-propanol) and (b) secondary alcohols (1-phenylethanol and 2-butanol) at a bare column electrode (left) and at an Au NP-modified column electrode (right). CV measurements were performed at a scan rate of 10 mV s^{-1} in water/acetonitrile solution (80/20 in vol%) containing 200 mM $LiClO_4$, 50 mM KOH, and 50 mM of alcohol. The flow rate was 0.5 mL min^{-1}.

alcohols on an Au electrode are dependent on the pK_a of the substrates and that the catalytic current increases with substrates associated with lower onset potentials because of their propensity to form alkoxides [23]. In Fig. 5, a similar trend was observed (furfuryl alcohol (pK_a = 9.6) < 1-phenylethanol (14.4) < benzyl alcohol (15.4) < cinnamyl alcohol (15.6) < 1-propanol (16.1) < 2-butanol (17.6) [24–27]).

2.3.2. *Controlled-potential electrolysis under various applied potentials*

Prompted by the results of CV measurements, we conducted bulk electrolysis under controlled-potential conditions to gain insights into the identity of the products generated by the electrochemical oxidation of alcohols. In addition to analyzing the products by high-performance liquid chromatography (HPLC), we monitored steady-state current values. Current efficiency was calculated on the basis of the integral of the current corresponding to the time of sample collection (see Fig. S7 and related description).

Electrolysis was performed under different applied potentials within the range of oxidation wave **A** in Fig. 2d. Bulk electrolysis of 1-phenylethanol was examined first, revealing that the application of a higher potential increased the current values, whereas the applied potential had little effect on the current efficiency, which was ~50% for all entries (Table S1). Despite efforts to identify the byproducts using HPLC and gas chromatography (GC), the substrates generated with the rest of the charge passed during the electrolysis remain unclear.

In the reaction with benzyl alcohol, the current also increased at higher potentials, suggesting that the applied potential strongly affected the reaction kinetics in the Au-catalyzed electrochemical oxidation of alcohols (Table S2). The oxidation of benzyl alcohol gave a higher current efficiency (78–89%) than the oxidation of 1-phenylethanol, presumably because the less-bulky nature of the primary alcohol enabled stronger adsorption onto the Au NPs. The current efficiency and the product selectivity were insensitive to the applied potential for benzyl alcohol, where both benzaldehyde and benzoic acid were obtained with an approximately 3:7 M ratio (Table S2). These results suggest that the oxidation power of Au(OH)$_{ads}$ species is essentially constant within the investigated potential range.

2.3.3. *Effect of stoichiometry of KOH and alcohol on the bulk electrolysis*

We next investigated the effect of the stoichiometry of KOH and alcohol on the outcomes of bulk electrolysis. The applied potential was 0 V *vs* Ag/AgCl, where the reaction sufficiently proceeded (Fig. 5).

The current efficiency in the oxidation of 1-phenylethanol in the column flow cell was independent of the amount of base (Table S3), whereas the current continued to increase as the amount of base was increased to 1 equiv with respect to the substrate and became constant as the amount of base was increased further (Fig. S8). Similar experiments were also performed using the Au disk electrode (Fig. S9a) previously used in the CV measurements (Figs. 2 and S2). When the Au disk electrode was used, the current value increased with increasing concentration of KOH; it then became constant at a certain point, indicating that the electrocatalysis on the Au NPs and that on bulk Au proceed in a similar manner. Interestingly, the current as a function of the concentration of 1-phenylethanol followed the same trend (Fig. S9b). These results suggest that the kinetics of the Au-catalyzed electrochemical oxidation are determined by the concentration of both KOH and 1-phenylethanol, consistent with the mechanism depicted in Fig. 3.

In the oxidation of benzyl alcohol, the total current efficiency for the production of benzaldehyde and benzoic acid increased with increasing amount of base (Fig. 6, Table S4). The current efficiency became quantitative with the addition of 10 equiv of KOH to benzyl alcohol. In the absence of KOH, benzaldehyde was predominantly formed with 97% selectivity, whereas the selectivity for benzoic acid increased with increasing amount of KOH. These results are attributed to the shift of the equilibrium from aldehyde to *gem*-diol (Fig. 3) as well as to an enhancement of the formation of Au(OH)$_{ads}$ species under basic conditions.

2.3.4. *Bulk electrolysis using various alcohols and long-term electrolysis*

The oxidations of various alcohols were subsequently investigated. Detailed results for the bulk electrolysis reactions are shown in Tables S3–S8. Table 1 summarizes the data for each substrate oxidized in the presence of 1 equiv of KOH.

In the oxidation of primary alcohols, the total current efficiencies for the corresponding aldehydes and carboxylic acids formation were higher than 90%. Interestingly, the selectivity of carboxylic acids greatly varied depending on the substrate (Table 1, entries 1–4). Notably, cinnamyl alcohol gave predominantly cinnamaldehyde, along with a very small amount of cinnamic acid (entry 2). When cinnamaldehyde was used as a starting material for the electrolysis under the same conditions, cinnamic acid was obtained with sufficiently high current efficiency; however, the current value was much lower than that observed in the oxidation of cinnamyl alcohol (Table S9), suggesting a kinetically controlled selectivity for the production of cinnamaldehyde. In the oxidation of secondary alcohols, the corresponding ketones were obtained with ~ 50% current efficiency (Table 1, entries 5 and 6).

We hypothesized that the differences in oxidation potentials of the substrates (ΔE_{ox}) or differences in the bond dissociation energy (ΔBDE) between alcohols and aldehydes might be responsible for the selectivity of the aldehyde/carboxylic acid production. The ΔE_{ox} values were calculated by quantum simulation using density functional theory [28], whereas the BDEs of the C—H bonds in the —CH$_2$OH or —CHO moiety were obtained by ALFABET [29]. Table S10 and Fig. S10 summarize the ΔE_{ox} and ΔBDE. Compared with ΔE_{ox}, ΔBDE shows better correlation

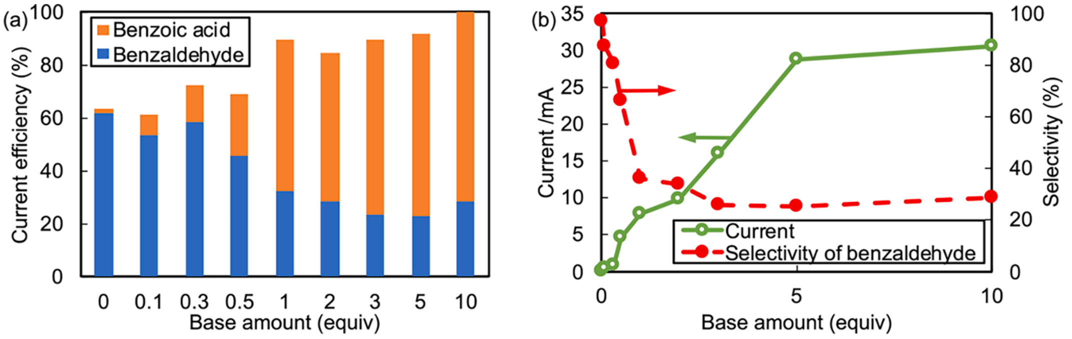
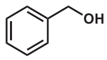
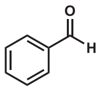
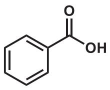
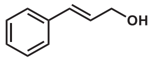
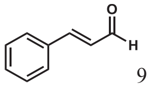
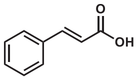
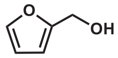
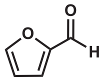
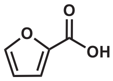
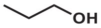
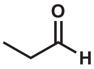
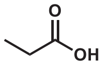
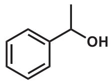
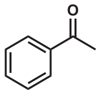
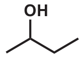
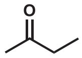


Fig. 6. Influence of the amount of base on (a) the current efficiencies and (b) current and selectivity of benzaldehyde in the electrooxidation of benzyl alcohol using an electrochemical column flow cell with an Au catalyst. Experimental conditions: anode, Au NP-modified column electrode; cathode, Pt spiral wire; solvent, water/acetonitrile solution (80/20 in vol%); supporting electrolyte, 200 mM LiClO$_4$; substrate, 50 mM benzyl alcohol; base, KOH; applied potential, 0 V *vs* Ag/AgCl; cell temperature, room temperature. Current efficiency was determined by HPLC. The electrolysis current was too small to obtain sufficient electricity when the amount of added base was<0.3 equiv. When the amount of added base was 10 equiv, the electrolyte was used without a supporting electrolyte.

Table 1
Controlled-potential electrolysis of alcohols at an Au NP-modified electrode using an electrochemical column flow cell.[a]

Entry	Substrate	Current/mA	Current efficiency[b] (%)			Aldehyde/Carboxylic acid
			Aldehyde, ketone (2e$^-$ oxidation)	Carboxylic acid (4e$^-$ oxidation)	Total	
1		7.9	 32	 57	89	36/64
2[c]		9.7	 96	 3	99	97/3
3		2.3	 52	 38	90	58/42
4[d]		2.7	 Trace	 >99	>99	0/100
5		9.5	 51	–	51	–
6[e]		0.2	 56	–	56	–

[a] Experimental conditions are as follows: anode, Au NP-modified column electrode; cathode, Pt spiral wire; solvent, water/acetonitrile solution (80/20 in vol%); supporting electrolyte, 200 mM $LiClO_4$; substrate, 50 mM of alcohol; base, 50 mM KOH; applied potential, 0 V *vs* Ag/AgCl; electricity, approximately 5C; cell temperature, room temperature. [b]Determined by HPLC. [c]Solvent, water/acetonitrile solution (70/30 in vol%). [d]Solvent, 100% water. [e]Current was too low to pass 5C of charge.

with the product selectivity. Because of the highly complicated reaction mechanism, completely elucidating the correlation between the chemical structure of alcohols and the selectivity for aldehyde/carboxylic acid production is difficult; however, these data could provide insights into the system and enable predictions of the products.

To confirm the feasibility of this system for mass production, long-term bulk electrolysis was performed for the oxidation of benzyl alcohol as a model reaction. A constant electrolytic current continued to flow for 2 h without deactivation of the catalyst (Fig. S11). In addition, the total current efficiency for the production of benzaldehyde and benzoic acid remained >90% during 2 h of electrolysis. To the best of our knowledge, this result is the first report on prolonged bulk electrolysis in an electrochemical column flow cell. SEM analysis of the electrodes post-electrolysis revealed that the morphological features of the Au NPs were unchanged, suggesting that the system is durable (Fig. S12).

3. Conclusion

We have developed a novel system for the continuous electrochemical oxidation of alcohols using a column flow cell. A carbon-fiber thread electrode was successfully modified with Au NPs as a catalyst via a two-step modification method developed in the present work, which resulted in an extremely high surface area and high electrocatalytic activity, enabling the oxidation of alcohols with high efficiency and small overpotentials. Various alcohols were successfully oxidized under the continuous flow. This preliminary research proposes a new approach in the design of a scalable and productive alcohol oxidation system with high sustainability.

CRediT authorship contribution statement

Tatsuya Suga: Data curation, Formal analysis, Investigation, Methodology, Validation, Visualization, Writing - review & editing. **Naoki Shida:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Supervision, Validation, Visualization, Writing - original draft. **Mahito Atobe:** Conceptualization, Funding acquisition, Methodology, Project administration, Resources, Supervision, Validation, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was financially supported by a CREST (JST grant no.: JPMJCR18R1) and by a Grant-in-Aid for Scientific Research (JSPS grant no.: JP20H02513, JP20K21106), Japan.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.elecom.2021.106944.

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