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Article

Underwater Superoleophobic Carbon Paper/Pt Composite Electrodes for Improving Kolbe Electrochemical Production

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Abstract

The acquisition of liquid energy sources and basic chemicals from washing water via Kolbe electrolysis is of great significance for achieving the goal of carbon-neutrality. However, the oleophilic products tend to adhere to Platinum (Pt) electrode, which results in a shortened working life of the Kolbe electrolysis. To address these issues, a novel method for endowing carbon fiber paper electrodes with underwater superoleophobic properties through simple electrodeposition is reported herein. The underwater superoleophobic electrodes improve the efficiency of the Kolbe electrolysis reaction, as oleophilic products can be easily removed from the electrode surface, thereby exposing more active reaction sites. Importantly, the underwater superoleophobic electrodes have fully demonstrated their advantages of excellent electrochemical performance, stability and durability. This work provides a novel approach for the design of high-performance electrodes in organic electro-catalysis.

Keywords: kolbe electrolysis; underwater superoleophobic; carbon fiber paper; oleophilic products

1. Introduction

Driven by the goal of carbon-neutrality, the urgency of easily accessible renewable energies and sustainable sources of raw materials is steadily increasing [1–4]. With the help of Kolbe electrolysis, liquid energy sources and basic chemicals are efficiently obtained from various carboxylic acids of biomass-based compounds or surfactants of washing water [5–9]. However, the electrochemical activity of the superior Platinum (Pt) electrocatalysts in this electrolysis reaction gradually decreases, owing to the alkyl compound products inclined to adhere to the Pt plates. Innovative progress on improvement the electrochemical activity and working life of Kolbe electrolysis is still remains challengeable.

The strong dependence of electrochemical performance on the interfacial interactions, during which multiphase mass transfer and energy conversion occurs in the electrolysis process, is greatly relevant to the physical and chemical aspects of the electrode surface. Especially, the rapid release of products on the electrode surface is closed related to the wettability of the electrode surface^{10, 11}. Recently, lots of progress in obtaining superwetting electrocatalyst surface by construct the hierarchically micro/nanostructured interfaces to facilitate mass transport in the electrocatalytic reactions such as oxygen reduction reaction (ORR) [12,13], hydrogen evolution reaction (HER)

[10,11,14–16], and oxygen evolution reaction (OER) [17], etc. Theoretically, designing the superwetting of the electrode to endow the alkyl compound products overcoming the adhesion barriers, leading to a long working life and excellent electrochemical performances of the Pt electrode. In our previous work, underwater superoleophobic Pt electrodes with multiscale structures were designed inspired by the biological materials, enhancing the working life from about 500 to 30 000 s. However, the cost-effective is a non-negligible factor in the carbon-neutrality action, and the cost of the Pt plate and the laser etching obviously clash with the cost-effective of the carbon-neutrality implement approach. Therefore, designing the electrode with cost-effective, high electrochemical activity is urgency to match the production of the carbon-neutrality policy.

Herein, we report an economical and underwater superoleophobic electrode based on carbon fiber paper to the replace pure Pt plate electrode for steady performance in Kolbe electrolytic reaction. Nanostructured Pt particles were coated on carbon fiber by electrodeposition. The electrode surface roughness can be readily controlled by varying the electrodeposition time. The coated Pt particles improve the roughness of the electrode surface and enhance the surface superoleophobicity, thus the as-formed oil products can leave the electrode surface readily to expose more reaction active site, so that the reaction can maintain high performance for a long time. Such carbon paper/Pt composite electrodes fully demonstrated their advantages of high performance and cost-effective to Kolbe electrolysis, and would shed light on electrode design for other organic electrocatalysis reactions.

2. Materials and Methods

Fabrication of the underwater superoleophobic carbon paper/Pt composite electrodes: Pt NPs were electrodeposited on carbon fiber paper to obtain underwater superoleophobic electrodes. The electrodeposition process employed a three-electrode configuration, comprising of carbon paper as the working electrode, a carbon rod electrode as the counter electrode, and an Ag/AgCl electrode as the reference electrode. The electrolyte solution was consisted of a mixture containing 1 mmol·L⁻¹ K₂PtCl₆·6H₂O and 0.5 mol·L⁻¹ H₂SO₄, and different electrodes were obtained by varying the electrodeposition time from 4000 s to 9600s..

Catalytic activity evaluation: The electrochemical activity of the as-prepared electrodes for Kolbe reaction was evaluated in an electrolytic cell. The electrolyte was 15 wt% stearic acid and a mixture of water and ethanol (1:1) was used as the solvent. A flat Pt electrode was used as the counter electrode.

Characterization: Scanning electron microscope (SEM) images were acquired using a field-emission SEM (Quanta 250 FEG). The energy dispersive spectroscopy (EDS) analyses were performed using GeminiSEM 300 (Carl Zeiss, Germany). The electrolysis was carried out using an electrochemical workstation (CHI 760E). All the contact angles were measured using a OCA20 CA system (Data-Physics, Germany) at ambient temperature. The droplet adhesive force on the electrode surface was measured using a high sensitivity micro-electromechanical balance system (Dataphysics DCAT11, Germany). 1,2-Dichloroethane was used for the underwater oil droplet contact angle tests, with each measurement taken at five different regions of the sample surface. The composition of electrolytic product was characterized by mass spectrometry, (MS, Agilent qtof6550).

3. Results

Recently, the concept of mass transport manipulation on superwetting interfaces has extended to chemical reaction systems to improve the reaction kinetics [18]. Superwetting interfaces serve as a versatile platform in chemical reactions involved multiphases (i.e., oil, water and solid) for enhancing chemical reaction efficiency, which through high-rate mass transport and effective collection of products. Consequently, the hierarchical micro-nanostructures of the electrode turns to be an important factor to affect the equilibrium state of actant/product adsorption/detachment rapidly in the multiphase electrochemical reactions. Therefore, how to construct the electrode with specific morphology is crucial for the rapid mass transport.

The rich three-phase interfaces are conducive to fast mass transport during the electrocatalytic reaction process, and carbon paper is the optimal candidate due to its large surface area and high porosity. Furthermore, carbon paper with proper 3D architecture has shown unique advantage in improving Pt utilization when used as catalyst support. The schematic illustration is depicted in Figure 1a, the process employed a three-electrode configuration, with carbon fiber paper serving as the working electrode. Direct electrodeposition process can efficiently immobilize the Pt nanoparticles (NPs) on carbon paper without any binder, which can effectively reduce the mass transport resistance and improve the utilization efficiency of Pt. Besides, in-situ grown Pt NPs during electrodeposition process results in a strong adherent force between Pt and conductive substrate, preventing the degradation of Pt NPs such as detachment, migration and agglomeration caused by the weak adherent force between Pt NPs and carbon support [19]. The micro/nano structure even the special wettability of the electrode can be manipulated by tailoring the electrodeposition parameter. For traditional flat Pt electrodes, the SEM images clearly show that their surfaces are smooth at a large scale (Figure 1b), and the underwater contact angle of oil droplet is about 65.4° (Figure 1d), suggesting their oleophilic property. Generally, the Kolbe electrolysis is the decarboxylative dimerization reaction under the function of the electrical field, yielding oleophilic products. Thus, the as-formed oil products during the Kolbe reaction adheres to the Pt sheet, progressively reducing the catalytic active sites, eventually obstructing the reaction and causing the reaction ceases (Figure 1f). In sharp contrast, the composite electrode exhibits excellent micro-nano multi-scale structure (Figure 1c) and the underwater oil contact angles is about 151.1° (Figure 1e). Such an underwater superoleophobic and low adhesion effect enables the electrode to timely remove the as-formed oil products without leaving oil residue. Thus, a stable and continuous current density obtained during the electrochemical reaction (Figure 1f), identifying an extended working life of the electrode during the Kolbe reaction process.

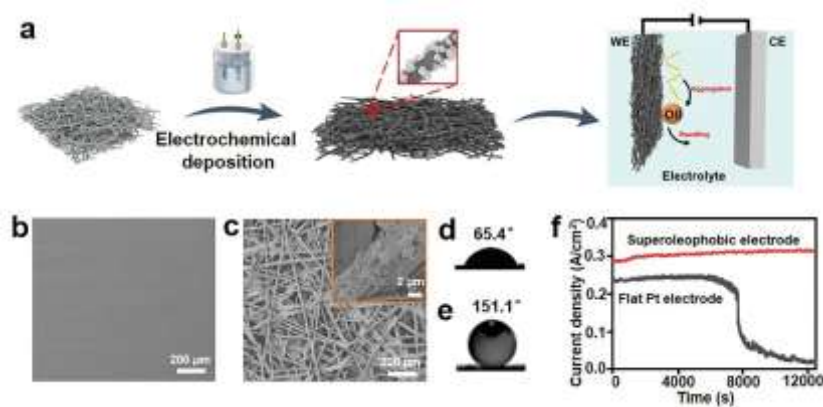


Figure 1. Schematic fabrication process and characterization of the underwater superoleophobic composite electrode. (a) The scheme mechanism of oil detachment during the Kolbe reaction and the electrode preparation process for Kolbe reaction. (b) SEM images of pure Pt plate electrode and (c) underwater superoleophobic composite electrode. (d) Underwater Oil contact angle on Pt plate (65.4°) and (e) the underwater superoleophobic composite electrode (151.1°). (f) Plots of current density on the reaction time using Pt flat and underwater superoleophobic composite electrode (deposition time: 8400 s) as the working electrodes.

To control the surface superoleophobicity, we tested micro-nanostructure and superwettability of as-prepared electrode at various electrodeposition time (Figure 2 a-j). The electrodeposition time of Pt NPs from 4800 s to 9600 s while maintaining a constant deposition voltage. Compared to the initial underwater oleophilic carbon fiber paper (Figure S1), SEM analysis confirmed the deposition of Pt NPs over the entire substrate, confirming that a large portion of the carbon fiber was exposed to the electrolyte. Most region of the carbon paper surface were uniformly covered with a layer of Pt NPs within the electrodeposition time of 4800 s, but partial regions of the carbon fiber surface were not completely covered by Pt NPs (Figure 2a). At 6000 s, the carbon fiber surface was almost entirely

covered of Pt NPs, displaying rough a nanospherical structures (Figure 2b). With the increase of electrodeposition time, the Pt NPs aggregate into microspheres on the carbon fiber surface (Figure 2c) and from more compact hierarchical micro-nanostructures (Figure 2d). However, the Pt NPs form a discursive pine-branch-like stacked structure on the carbon fiber surface when the electrodeposition time reaches 9600 s (Figure 2e), which may impact the stability of the electrode. The surface oleophobicity of the electrodes obtained at different electrodeposition times was assessed by measuring the underwater oil contact angles (1,2-Dichloroethane, 2 μ L). The underwater oil contact angles for electrodes with electrodeposition times of 4800 s, 6000 s, 7200 s, 8400 s, and 9600 s are 147.6°, 147.4°, 151.1°, 151.8°, and 154.2°, respectively (Figure 2 f-j). The electrode surface with only nanospherical structure exhibited high underwater oleophobicity, while the electrode with hierarchical micro-nanostructures even achieved underwater superoleophobicity. Therefore, the scale of coarse hierarchical micro-nanostructures, as well as their distribution, are crucial for the underwater oleophobicity of electrode. To further characterize the dynamic oil adhesion forces of the electrodes in underwater environments, dynamic adhesion tests were performed to assess the capacity of electrodes to repel oil generated during the reaction process. Figure 2 k, l display the adhesion force curves and numerical values of the electrode surfaces with different electrodeposition times, the underwater oil adhesion forces are about 22 μ N, 19 μ N, 21 μ N, 22 μ N, and 22 μ N, respectively. These results indicate that the electrodes possess low underwater oil adhesion, which is consistent with superoleophobic and low-adhesion characteristics. In aqueous media, the hierarchical micro-nanostructure of the electrode surface can capture a continuous water film, resulting in low adhesion of the electrode surface to oil droplets, that is, the oil droplets can maintain the Cassie-Baxter state and finally leave the electrode surface readily.

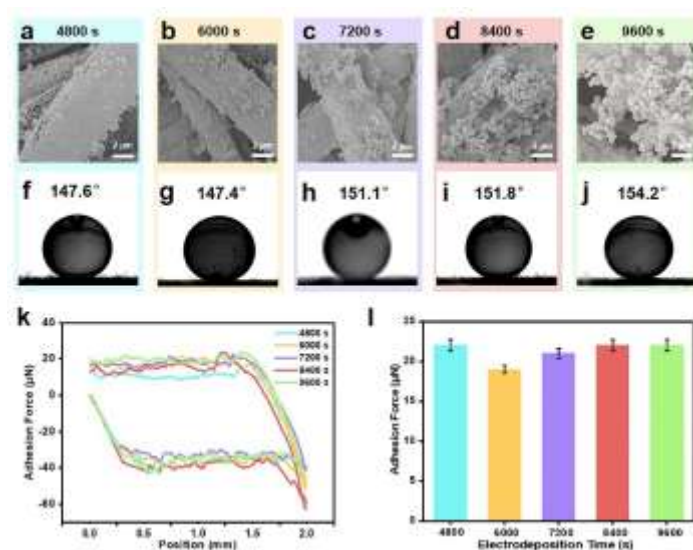


Figure 2. The effect of deposition time on the underwater wettability of the composite electrode. (a-e) SEM images of Pt deposited carbon fiber paper with different deposition time. (f-j) Underwater oil contact angle images of different Pt deposition electrode (1,2-Dichloroethane, 2 μ L). (k-l) Underwater oil adhesion force curves and adhesion values on different Pt deposition electrode.

Superwetting surfaces exhibit versatility in manipulating the mass transfer of ions liquids and gases on solid surfaces, which has been extended to chemical reaction systems to improve reaction kinetics. Superoleophobic electrodes can enhance three-phase interfaces and facilitate mass transport, thereby achieving the high catalytic performance and secular stability. To reveal the impact of the electrode surface microstructure on its electrocatalytic performance, Kolbe electrolysis tests of stearic acid were conducted using electrodes obtained at different electrodeposition times. As shown in Figure 2a, with the electrodeposition time increasing from 4800 seconds to 9600 seconds, the initial current density of the electrolytic reaction gradually increased, indicating that the reaction efficiency

could be significantly improved. This is attributed to the increased loading of Pt NPs on the carbon fiber surface with the extension of electrodeposition time, which greatly increases the reaction active sites. The current density of the electrodes with electrodeposition time of 4800 s and 6000 s were significantly lower, which is due to the insufficient coverage and loose structure of the micro-nanostructure formed by Pt NPs, resulting in insufficient reactive active sites. The electrode with an electrodeposition time of 9600 s showed a high initial current density, but, a decline in current density was observed around 10000 s, identifying a decrease in electrolysis efficiency. This is because the micro-nano multiscale structure on the electrode surface, though relatively abundant, lacks stability, with partial structure detachment occurring in the mid-reaction, leading to a decline in electrochemical performance. Remarkably, the electrode with an electrodeposition time of 8400 s exhibited optimal electrochemical performance, achieving the highest current density and maintaining a stable and efficient Kolbe reaction over an extended period. The occurrence of the Kolbe reaction was further identified by mass spectrometry (MS) analysis of the reactant (before Kolbe electrolysis) and product (after Kolbe electrolysis). By comparing the total ion chromatogram (TIC) of the reactant (Figure S2 a), a well-resolved peaks appeared in the TIC of the product after electrolysis (Figure 3 b), demonstrating the effectiveness of the superoleophobic electrode in catalyzing Kolbe reaction. Before the electrolysis reaction, the m/z data of the reactant were mainly located at 391.621, demonstrating that the main component was stearic acid (Figure S2 b). After the electrolysis reaction (10 h), the m/z signals of the as-formed oil products shifted to 391.014 and 701.496, which indicated the formation of tetratriacontane in the electrochemical reaction (Figure 3 c). The MS results clearly confirmed the presence of tetratriacontane, which was generated during the Kolbe electrolysis.

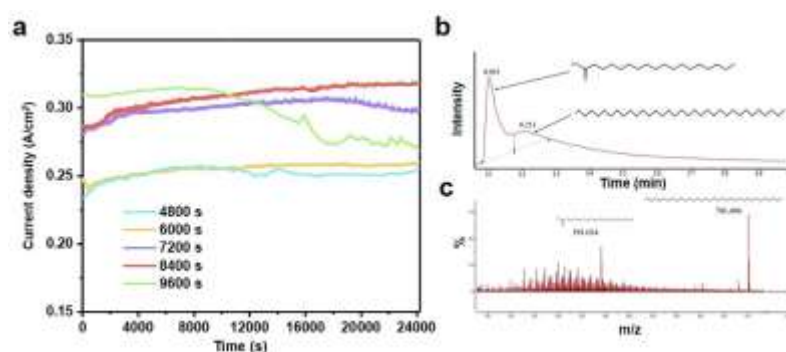


Figure 3. The Kolbe electrochemical performances of the underwater superoleophobic electrode. (a) Plots of current density on the reaction time using composite electrode with different electrodeposition time as the working electrodes. Mass spectra of as-formed oil products: (b) total ion chromatogram (TIC) and (c) extracted ion chromatogram (EIC).

The durability and stability of underwater superoleophobic electrode were further assessed and presented in Figure 4. SEM images reveal that the electrodes with electrodeposition times of 4800s, 6000s, 7200s, and 8400s exhibited minimal structural changes after the Kolbe reaction (10 h). Although the electrode with electrodeposition times of 9600 s initially displayed the most abundant micro-nano structures (Figure S3), the carbon fiber substrate was exposed after the reaction, which may lead to a reduction in its electrochemical performance. To further examine the structural detachment and elemental distribution of the electrode surfaces after Kolbe reaction, energy dispersive spectroscopy (EDS) mappings are shown in Figure 4. EDS results indicate that the micro-nano structures formed by Pt NPs on the electrode surface remained relatively stable, and the Pt elements were evenly distributed over the carbon fiber surface (Figure 4 a, b, c, d), maintaining its oleophobic or even superoleophobic properties after the Kolbe reaction (Figure 4 f, g, h, i). In contrast, the electrodes with electrodeposition times of 9600 s demonstrated significant Pt NPs detachment and carbon fiber substrate damage, resulting in disordered C and Pt elemental distribution (Figure 4 e2, e3). Thus, the

electrode lost superoleophobic property after Kolbe reaction (Figure 4 j). This change corroborates that the detachment of micro-nano structure formed by Pt NPs is the primary factor leading to the degenerate of electrochemical property. These facts are identical with the results of current density curve measurement. As a result, prolonged electrodeposition time can cause excessive deposition of Pt NPs, leading to unstable growth and cracking of micro-nano structures.

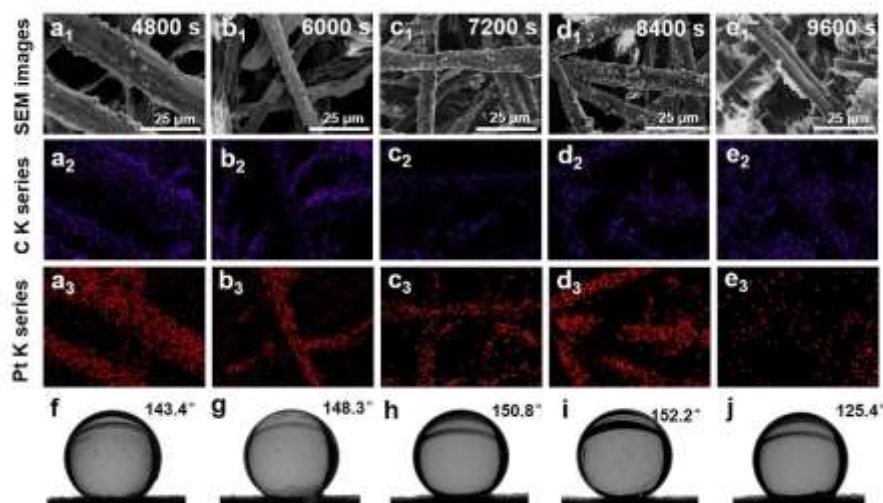


Figure 4. SEM micrographs and underwater oil contact angle of different Pt deposited electrodes after Kolbe reaction. (a-e) SEM images and EDS mappings of Pt distribution of different Pt deposited electrodes after Kolbe reaction. (f-j) Underwater oil contact angle on different Pt deposited electrodes after Kolbe reaction.

Figure 5 illustrates the suggested mechanism for the adhesion behavior of as-formed oil products on flat Pt electrodes and underwater superoleophobic carbon paper/Pt composite electrodes. Due to the inherent oleophilic property, the as-formed oil products form a continuous contact line with the traditional flat Pt plate electrodes and establish stable attachments (Figure 5 a). This adhesion behavior can mask the active sites and hinder catalytic reactions, leading to a decline in electrode performance, especially in long-term electrochemical reactions. Contrarily, benefiting from the unique micro-nano structure formed by Pt NPs on the surface of the composite electrode, the contact area between the electrolytic product and the electrode is reduced, forming discrete contact lines and endowing the electrode with underwater superoleophobic capability (Figure 5 b). This underwater superoleophobic property effectively minimizes product adhesion on the electrode surface and promptly removes the as-formed oil products, thereby greatly improving the working life of the electrode and maintaining high electrochemical activity. Moreover, the discrete contact points provide rich three-phase interfaces, which facilitate rapid mass transport during the electrocatalysis reaction process. Thus, in principle, our strategies can also be applied to Kolby electrolysis reactions of various carboxylic acid molecules.

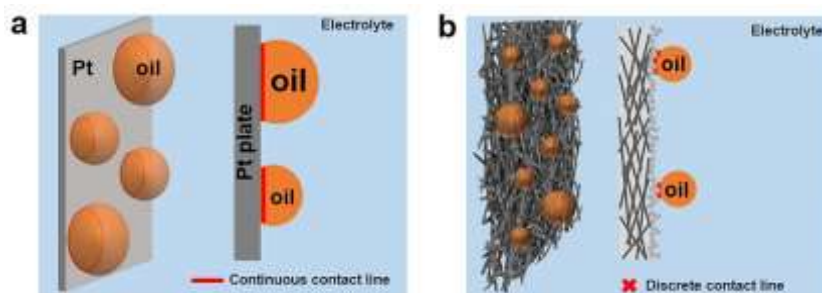


Figure 5. Schematic illustrations of adhesion behavior of oleophilic oil products on the (a) flat Pt electrode and (b) underwater superoleophobic Pt deposited carbon fiber paper electrode.

4. Conclusions

In this contribution, we report that the property of electrodes for Kolbe electrolysis reaction can be significantly improved even without complicated processes by modifying carbon fiber paper with electrodeposited Pt NPs. Briefly, the micro-nanostructure, underwater oleophobicity, electrochemical performance of the electrodes can be controlled by adjusting the electrodeposition time. Due to facile removal of as-formed oil products, the underwater superoleophobic carbon paper/Pt composite electrodes outperformed traditional flat Pt plate electrodes and exhibited excellent stability and durability. The fabricated underwater superoleophobic electrodes provide a new strategy for enhancing Kolbe electrochemical production, and have far-reaching implications for the treatment of wastewater containing surfactant pollutants.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org: Figure S1: SEM micrographs and underwater wettability of pure carbon fiber paper; Figure S2: Mass spectra of the reactant (before Kolbe electrolysis); Figure S3: SEM image and EDS mappings of the electrode with electrodeposition time of 9600 s before Kolbe reaction..

Author Contributions: Conceptualization, J.L. and Q.L.; methodology, L.W., J.Z. and L.G; software, S.S., J.Z. and W.L.; validation, J.L., Q.L. and L.W.; investigation, L.W. and L.G; data curation, L.W.; writing—original draft preparation, J.L. and Q.L.; writing—review and editing, Y.N. and Z.Z.; supervision, J.L.; project administration, Y.N., Z.Z. and K.L.; funding acquisition, Y.N., Z.Z. and K.L. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Data will be made available on request.

Conflicts of Interest: The authors declare no conflicts of interest.

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