



Electrochemical degradation of chlorobenzene on conductive-diamond electrode

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ABSTRACT

Conductive-diamond powder was co-deposited with nickel to form Ni-conductive-diamond composite electrode. Conductive-diamond electrode, which can be used for stable anode, was successfully fabricated by Ni-conductive-diamond composite electrode dip coating with polyvinylidene fluoride (PVDF) and reveal the conductive-diamond by polishing the PVDF coated on the conductive-diamond powder surface. The conductive-diamond electrode possesses extremely wide potential window, extending almost 2 V in acid, 2.5 V in alkali and 1.75V in salt solution. The electrochemical degradation of chlorobenzene (CB) with the prepared conductive-diamond electrode and Pt electrode was evaluated with the parameter of total organic carbon (TOC) removal, the TOC removal efficiency reached 92.6% on the conductive-diamond electrode, much higher than 82.5% on Pt electrode under the same experiment condition.

Prime novelty statement:

- (1) Conductive-diamond powder was co-deposited with nickel to form composite electrode.
- (2) The electrochemical degradation of chlorobenzene with the prepared conductive-diamond electrode and Pt electrode was evaluated. With the parameter of total organic carbon (TOC) removal, the TOC removal reached 92.6% on the conductive-diamond electrode, much higher than 82.5% on Pt electrode under the same experiment condition.

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1. Introduction

The increasing public concern regarding the environmental contamination that arises from hazardous pollutants present in a wide variety of industrial effluents has brought about stricter legislation and lower disposal limits. Among polluting effluent, halogenated aromatic hydrocarbons have been introduced to the environment from a variety of sources, such as pharmacy, dyeing, petroleum refining and plastic [1]. As a representative halogenated aromatic compound, chlorobenzene (CB) is easy to get caught up in large variety of industrial effluent, CB might cause severe problems to human health and aquatic life [2–4]. Thus a new effective technology should be applied to treat CB before disposal.

Several methods including photocatalytic oxidation [5], Fenton oxidation [6], H₂O₂ oxidation [7], Mn catalytic oxidation [8] and ultrasonic oxidation have been evaluated for the CB effluent treatment [9]. Electrochemical oxidation technology as an advanced oxidation treatment for hazardous pollutants has attracted a great deal of attention recently due to its high energy efficiency, ease of operation and cost effectiveness [10]. To find a high performance electrode is a focus point in the research on electrochemical degradation of CB. Up to now, such a diversity of electrodes including carbon materials (for instance activated carbon fiber, graphite and boron doped diamond (BDD)) metal materials (for instance platinum and titanium metal and stainless steel) and dimensionally stable electrode (DSA) (for instance RuO₂, SnO₂ and PbO₂) [11–13] were studied. The boron doped diamond (BDD) has emerged as a very promising anode material. It possesses advantageous properties such as an extremely wide potential window for water discharge, robust oxidation capacity, corrosion stability in very aggressive media, an inert surface with low adsorption and resistance to corrosion [14, 15]. However BDD anode also has some disadvantages such as the

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preparation process is very complicated subject to irregular base surface with complex geometry shape, expensive and so on. These disadvantages have limited its industrial application [16–19].

This paper demonstrates that the conductive-diamond electrode could be fabricated by co-deposit conductive-diamond powders with Nickel electroplating, dip-coating with polyvinylidene fluoride (PVDF), and the electrochemical degradation of CB on the conductive-diamond electrode were investigated.

2. Experimental

2.1. Materials and reagents

Commercial available conductive-diamond powder (provided by the Henan Huanghe Whirlwind Co., Ltd. and the resistivity of conductive-diamond powder was $1.08 \Omega \cdot \text{cm}$, average diameter $60\text{--}70 \mu\text{m}$) and brass rod (diameter $2 \text{ mm} \times$ length 60 mm) were used. All the reagents were of analytical degree and purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All the chemicals used in the experiments were received without further purification. All the solutions were prepared using double-distilled water.

2.2. Electrode preparation

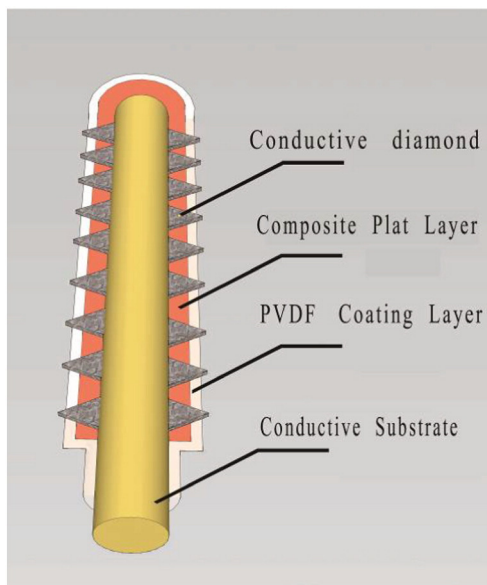
The process for conductive-diamond electrode preparation was shown in Scheme 1, and the structure of the conductive-diamond electrode shown in Scheme 2. Details of the process were described as follow.

2.2.1. Conductive-diamond powder treatment

Conductive-diamond powder was suspended in 10 wt.% nitric acid, and refluxed for 2 h. Then repeatedly washed with deionized water to remove the acid solution and finally dried in a drying oven. Next step in the process, conductive-diamond powder was suspended in 10 wt.% sodium hydroxide solution and refluxed for 2 h, then washed and dried. Finally, conductive-diamond loaded into the cathode cloth bag and marinated it into nickel plating solution.

2.2.2. Brass rod surface treatment

First step, a brass rod was mechanically polished with 600-mesh abrasive papers. Then the brass rod was cleaned with deionized water and acetone to remove solid particles and grease. Second step, it was subsequently immersed in brass chemical polishing solution (HNO_3 10 mL/L, H_3PO_4 54 mL/L, CH_3COOH 30 mL/L, temperature $55\text{--}65 \text{ }^\circ\text{C}$, time 3–5 min), and then we adopted the brass rod which had

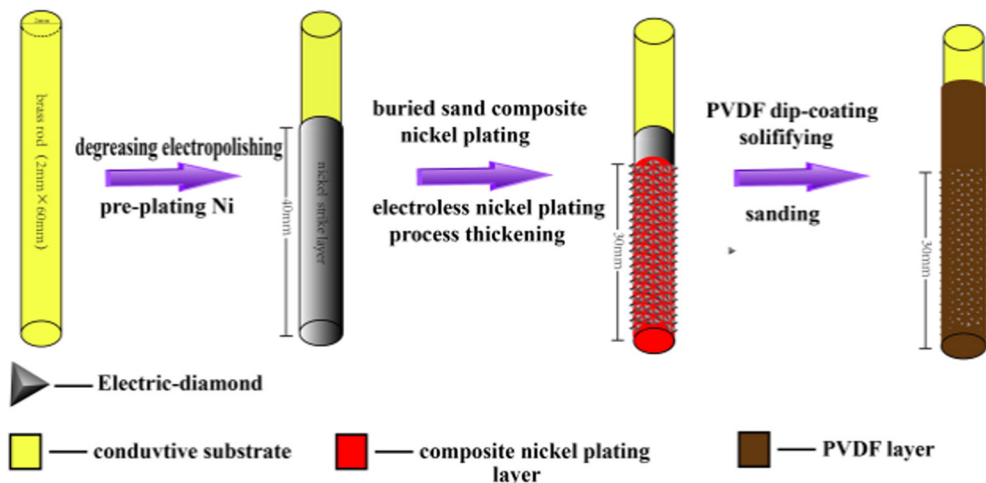


Scheme 2. Sectional axonometric drawing for conductive-diamond electrode.

been above processed treated as anode, lead as cathode, electrochemical polish operation was carried out through constant voltage. The brass electrochemical polishing solution and parameters are (H_3PO_4 72 mL/L, voltage 2.5 V, time 5 min). Third step, impact nickel plating was performed in the solution ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ 250 g/L, chloric acid 120 mL/L) and condition (room temperature, current density $5 \text{ A}/\text{dm}^2$, time 5 min).

2.2.3. Nickel/(conductive-diamond) powder composite electroplating

The brass rod was buried in the conductive-diamond powder loaded in cathode cloth bag for bright nickel plating in the solution ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$: 300 g/L, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$: 40 g/L, HBO_3 : 40 g/L, sodium dodecyl sulfate: 0.1 g/L, butynediol: 0.5 g/L, saccharin: 1 g/L). To keep the conductive-diamond powder stick on the brass rod surface and with good electric conduct, nickel coating thickness is supposed about 10% of the conductive-diamond powder diameter and the resulted plating parameter is with current density $2 \text{ A} \cdot \text{dm}^{-2}$ for 25 min. Then the brass rod with conductive-diamond powder on its surface was moved out from the cathode cloth bag to commercial electroless nickel plating solution for thickening the nickel coating. Final nickel coating thickness is about 50% of conductive-diamond diameter.



Scheme 1. Schematic illustration for preparing conductive-diamond electrode by using the composite electroplating and dip coating method.

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