



Electrochemical Oxidation of Para-Aminophenol With Rare Earth Doped Lead Dioxide Electrodes: Kinetics Modeling and Mechanism

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In this study, La and Ce doped PbO₂ electrodes were prepared and the characteristic of the electrodes were discussed with the help of structure analysis. The catalytic effects of the doped electrodes were explored through the degradation of para-aminophenol wastewater. The results showed that the para-aminophenol removal was 96.96%, 89.34%, and 77.55% after 180 min treatment with Ce-PbO₂, La-PbO₂, and PbO₂, respectively. The para-aminophenol enhanced degradation mechanism was discussed with rare earth element doping electrodes and a kinetic model was established based on radical reactions mechanism with genetic algorithm (GA) calculation. The reaction constants of these electrodes were calculated and the results showed that the reaction constant of Ce-PbO₂ electrode was the highest, which indicated that Ce-PbO₂ electrode could have a better treatment effect. The EE/O was used as the index of energy consumption efficiency and the results were calculated and compared. This paper could provide basic data and technique reference of the prediction the oxidation reaction process of different electrodes for the electrochemical oxidation application in wastewater treatment.

Keywords: electrochemical oxidation, rare earth, doped lead dioxide electrodes, kinetics modeling, mechanism

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INTRODUCTION

Para-aminophenol (PAP) is the primary material of paracetamol industry, which was widely used in a number of countries (Bloomfield, 2002; Bahrami and Salehabadi, 2014). Due to the characteristic of the pharmacy production process, the PAP would be contained in the wastewater. If this kind wastewater was discharged directly into water environment, there would affect human's health (Hallman and Tarloff, 2000; Li et al., 2004; Harmon et al., 2005; Khodaveisi et al., 2015). Therefore, the degradation and mineralization of PAP wastewater and relative pharmaceutical wastewater has received a great concern in the field of environment.

As one of advanced oxidation processes (AOPs), electrochemical oxidation could remove bio-refractory organic pollutants in wastewater and has the advantages of relatively high treatment efficiency with easy operation (Velegraki et al., 2010; Chen et al., 2014; Xia et al., 2015a). Electrode materials, such as boron-doped diamond, platinum, IrO₂, RuO₂, and PbO₂ and carbon relative materials (Arapoglou et al., 2003; Zhuo et al., 2011; Li et al., 2014; Wang et al., 2019), play an important role in electrochemical reaction. The β -PbO₂ electrode and rare earth element doped β -PbO₂ electrode were widely investigated due to the easy preparation technology and excellent degradation effect (Aquino et al., 2014; Mukimin et al., 2015; Xia et al., 2015b; Dai et al., 2016). However, the study of the reactions kinetic model on the doped and undoped PbO₂ electrodes is still limited.

In this study, PAP was selected as the model pollutant and the degradation of PAP wastewater was studied with the electrodes of PbO₂, La doped PbO₂ (La-PbO₂), and Ce doped PbO₂ (Ce-PbO₂). A kinetic model was established based on genetic algorithm (GA) calculation and used to quantify the performance of electrode materials. This paper could provide basic data and theoretical support for PAP wastewater treatment by electrochemical oxidation.

EXPERIMENTAL

Chemicals

PAP was selected as the model pollutant and obtained from Shanghai J&K Chemical Reagent (China) Co., Ltd. (purity 99.5%, wt%). The structure formula and general characteristics of PAP were given in **Table 1**. Other chemicals were purchased from Aladdin Reagent (China) Co., Ltd. All the other reagents were of analytical grad.

Electrode Preparation

The detail process for the preparation and characteristic of the electrodes has been fully described in previous literatures (Weng et al., 2013; Dai et al., 2014; Weng and Pei, 2016). Briefly, the electrode included three layers: the Ti substrate inner layer, thermally deposited Sn-SbOx intermediate layer, and the electrodeposited α -PbO₂ and β -PbO₂ outer layer. When La-PbO₂ and Ce-PbO₂ electrodes were prepared, certain amounts of La or Ce nitrate were added to the acid solution for the electrodeposition of β -PbO₂. Compared with undoped PbO₂ electrode, the surface and structure analysis results of doped PbO₂ showed that the electrodes after doped with rare earth could effectively improve the crystallinity of PbO₂ crystal on electrode surface. The tinier grain crystals on the doped electrodes could promote the conductivity, stability, and catalytic activity of electrode, which would be potential widely applied in dye wastewater pollution control in application filed.

Analysis Methods

The PAP concentration was analyzed by high performance liquid chromatography (Agilent 1200, USA) with C18 column and UV detector. The UV detector wavelength was set at 220 nm. The injection volume was 10 μ l. The mobile phase was methanol and H₃PO₄ solution (1%) and the ratio of methanol with H₃PO₄ solution was 22:78 (v/v). The flow rate was 0.4 ml min⁻¹. The crystal structure was investigated by X-ray diffraction (XRD) instrument with CuK α radiating (Rigaku D/MAX 2500PC,

Japan) under condition of 40 kV voltage and 300 mA tube current. The continuous scan mode was used and the scan speed was 4°·min⁻¹ with the range of 10°~90°.

The average current efficiency (ACE) was calculated by chemical oxygen demand (COD) and current data at time intervals (Comminellis and Pulgarin, 1991).

$$ACE = \frac{COD_0 - COD_t}{8It} FV \quad (1)$$

Where: COD₀ and COD_t are the CODs (in O, g·L⁻¹) of samples at different time (s), respectively. F is the faraday constant (96487 C·mol⁻¹), V is the volume of the electrolyte (L), I is the electrolysis current (A), and t is the reaction time (s).

Experimental Procedure

PAP wastewater degradation experiment was performed in a 250 ml customize reactor. During PAP degradation, samples were taken at presumed intervals to determine the concentration of PAP, COD, and total organic carbon (TOC). All tests were run three times. The results for each experimental condition were the means of the three experiments and the relative standard deviations (RSD) were <5%.

Modeling Approach

To establish model, the rate constants were calculated with the degradation data of PAP concentration. The minimum value of residual between experimental data and modeling data as the objective function was calculated by GA calculation (Goldberg, 1989; Ndiritu and Daniell, 2001).

The objective function (OF) was listed as follows:

$$OF = \sqrt{\frac{1}{n-1} \sum_{i=1}^n \left(\frac{C_{data,i} - C_{model,i}}{C_{data,i}} \right)^2} \quad (2)$$

Where: OF = objective function, dimensionless, n = number of data points, unitless

C_{data,i} = measured concentration of data point i, mmol L⁻¹,

C_{model,i} = predicted concentration of data point i, mmol L⁻¹

All the reaction constants (k) were calculated by the GA method.

RESULTS AND DISCUSSION

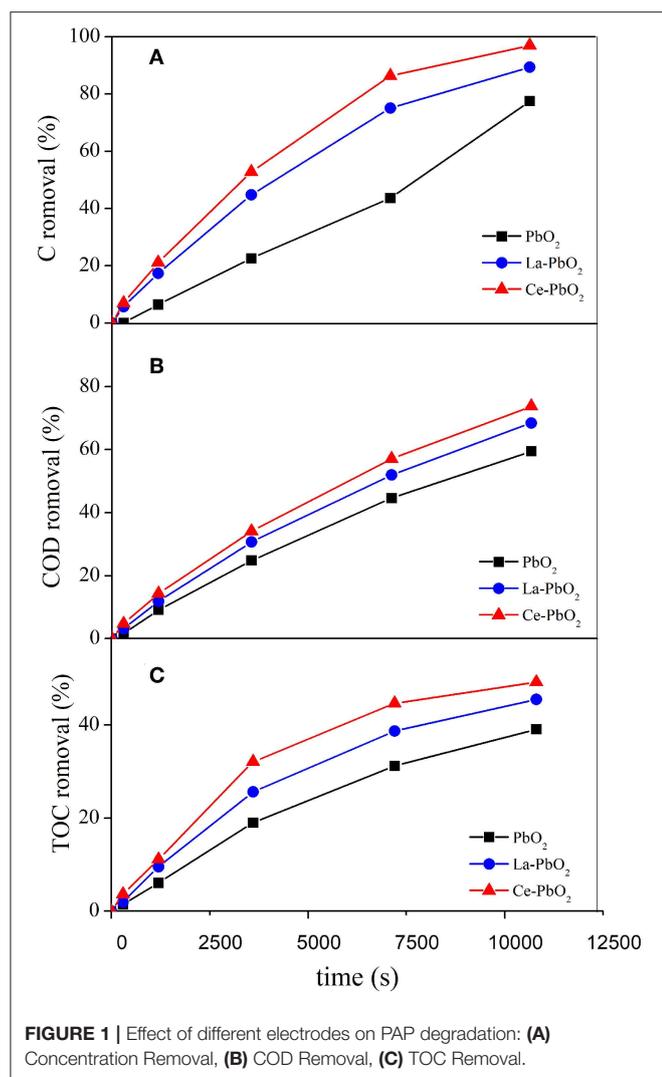
PAP Degradation With Different Electrodes

PbO₂ electrode, La-PbO₂ electrode and Ce-PbO₂ electrode were applied for the degradation of PAP wastewater on the condition of electrolyte concentration Na₂SO₄ 0.1 mol·L⁻¹, PAP concentration 500 mg·L⁻¹ and current density 70 mA·cm⁻². The degradation results were shown in **Figure 1**.

The PAP removal with Ce-PbO₂ electrode was 96.96%, which is higher than the removal with La-PbO₂ (89.34%) and PbO₂ electrodes (77.55%) after 3 h treatment. The COD and TOC removal after 3 h for these three electrodes was 73.79, 68.49,

TABLE 1 | General characteristics of PAP.

Name	4-Aminophenol
Chemical structure	
Water solubility	1.5 g/100 mL (20°C)
CAS number	123-30-8



59.49%, and 49.23, 45.47, 39.08%, respectively. And these results were showed in **Figures 1A–C**, respectively. Under the same conditions, the ACE of the La-PbO₂ and Ce-PbO₂ electrode were higher 8.35 and 11.69% than that of undoped electrode (42.05%). The results demonstrated that the catalytic activity of La-PbO₂ and Ce-PbO₂ were improved degradation of PAP effectively. Also, the Ce-PbO₂ electrode displayed a higher removal efficiency and faster mineralization than La-PbO₂ and PbO₂ electrodes for PAP degradation.

Materials Characterization

Figure 2 showed the XRD patterns of different electrodes. Compared with PbO₂, La and Ce doping would cause the change of crystal orientation of β-PbO₂. After doped, the lattice plane of (110) was enhanced, which would influence the catalytic activity for organics degradation. The SEM results showed that after doped, the electrode surface had a tinier crystal structure and higher degree of crystallinity (Weng et al., 2013), which means that the addition of Ce could effectively improve the crystallinity

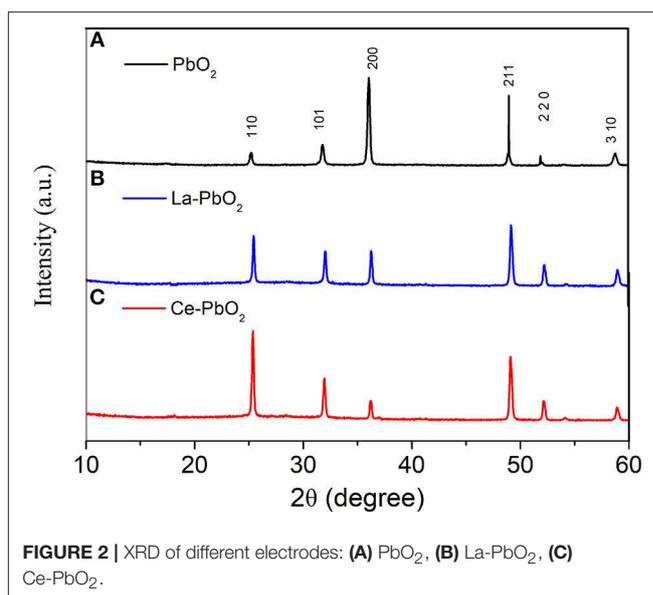


TABLE 2 | Mainly elementary reactions involved in electrochemical oxidation system.

No	Reactions	Rate Constants, mol L ⁻¹ s ⁻¹
1	$\text{H}_2\text{O} + \text{PbO}_2 \rightarrow \text{PbO}_2(\cdot\text{OH}) + \text{H}^+ + \text{e}^-$	k_1
2	$\text{PbO}_2(\cdot\text{OH}) \rightarrow \text{PbO}_2(\cdot\text{O}) + \text{H}^+ + \text{e}^-$	k_2
3	$\text{PbO}_2(\cdot\text{O}) \rightarrow \text{PbO}_2 + 0.5\text{O}_2$	k_3
4	$\text{PbO}_2(\cdot\text{O}) + \text{PAP} \rightarrow \text{PAP}(\text{O}) + \text{PbO}_2$	$k_{\cdot\text{O}}$
5	$\text{PbO}_2(\cdot\text{OH}) + \text{PAP} \rightarrow \text{oxidation products}$	$k_{\cdot\text{OH}}$
6	$\text{PbO}_2(\cdot\text{OH}) + \text{PbO}_2(\cdot\text{OH}) \rightarrow 2\text{PbO}_2 + 0.5\text{O}_2 + \text{H}_2\text{O}$	k_4
7	$\cdot\text{OH} + \text{HCO}_3^- \rightarrow \text{CO}_3^{\cdot-} + \text{H}_2\text{O}$	$k_{\text{HCO}_3^{2-}} = 8.5 \times 10^6$
8	$\cdot\text{OH} + \text{CO}_3^{2-} \rightarrow \text{CO}_3^{\cdot-} + \text{OH}^-$	$k_{\text{CO}_3^{2-}} = 3.9 \times 10^8$
9	$\text{H}_2\text{CO}_3 \rightleftharpoons \text{H}^+ + \text{HCO}_3^-$	$\text{pKa}_1 = 6.36$
10	$\text{HCO}_3^- \rightleftharpoons \text{H}^+ + \text{CO}_3^{2-}$	$\text{pKa}_2 = 10.25$
11	$\text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{OH}^-$	$\text{pKa}_3 = 15.7$

of PbO₂ crystal on electrode surface, and thus could increase the efficiency of PAP degradation.

Kinetics Model Study

The Key Reaction in Electrochemical Oxidation System

A generalized scheme of the electrochemical degradation of PAP with PbO₂ electrode was shown in **Figure 3**. Firstly (No. 1, **Table 2**), H₂O was decomposed on the electrode surface to yield adsorbed hydroxyl radical PbO₂ (•OH). Secondly (No. 2, **Table 2**), hydroxyl radical on the oxide electrode could both react with PAP and form the lattice oxide of PbO₂ (•O). Thus, there are two kinds of “active oxygen” could be existed on the PbO₂ electrode surface: physisorption “active oxygen” PbO₂ (•OH) and chemisorbed “active oxygen” PbO₂ (•O) (Comminellis, 1994).

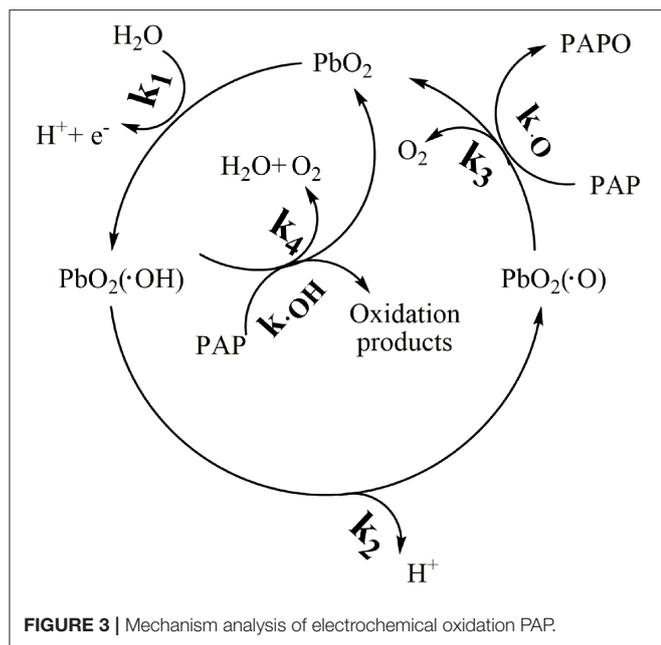


FIGURE 3 | Mechanism analysis of electrochemical oxidation PAP.

Due to the instability, the chemisorbed “active oxygen” $\text{PbO}_2(\bullet\text{O})$ would decompose according to the equation (No. 3, Table 2). The $\text{PbO}_2(\bullet\text{O})$ would participate in the reaction of oxidation PAP, as was listed in the equation (No. 4, Table 2), while $\text{PbO}_2(\bullet\text{OH})$ complete fast reaction of oxidation PAP and mineralization them completely, according to No. 5 in Table 2 (Buxton et al., 1988; Simond et al., 1996). The hydroxyl radicals also might interact and the reaction constant of k_4 means the interaction of hydroxyl radicals (No. 6, Table 2). The ionization balances and carbonate quenching equation were considered in our system list in Table 2.

From the No. 4 and No. 5 in Table 2, the ordinary differential equation (ODE) of PAP degradation rate with the time was deduced as follows, which included PAP reaction with $\text{PbO}_2(\bullet\text{OH})$ and $\text{PbO}_2(\bullet\text{O})$:

$$\frac{d[\text{PAP}]}{dt} = -k_{\bullet\text{OH}}[\text{PbO}_2(\bullet\text{OH})][\text{PAP}] - k_{\bullet\text{O}}[\text{Pb}(\bullet\text{O})][\text{PAP}] \quad (3)$$

From the No. 1, 2, 5, 6, 7, and 8 in Table 2, the ODE of $\text{PbO}_2(\bullet\text{OH})$ reaction rate variation with the time was studied, which included both yielding and decaying of $\text{PbO}_2(\bullet\text{OH})$:

$$\begin{aligned} \frac{d[\text{Pb}(\text{HO}\bullet)]}{dt} = & k_1[\text{H}_2\text{O}] - (k_2+k_4)[\text{PbO}_2(\bullet\text{OH})] \\ & - k_{\bullet\text{OH}}[\text{PbO}_2(\bullet\text{OH})][\text{PAP}] \\ & - k_{\text{HCO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{HCO}_3^-] \\ & - k_{\text{CO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{CO}_3^{2-}] \end{aligned} \quad (4)$$

From the No. 2, No. 3 and No. 4 in Table 2, the ODE of $\text{PbO}_2(\bullet\text{O})$ reaction rate variation with the time was deduced, which included yielding and decaying of $\text{PbO}_2(\bullet\text{O})$:

$$\begin{aligned} \frac{d[\text{Pb}(\bullet\text{O})]}{dt} = & k_2[\text{PbO}_2(\bullet\text{OH})] - k_0.[\text{PbO}_2(\bullet\text{O})][\text{PAP}] \\ & - k_3[\text{PbO}_2(\bullet\text{O})] \end{aligned} \quad (5)$$

The ODE of HCO_3^- and CO_3^{2-} termination the reaction with the time was deduced, including the key reaction No. 7 and No. 8 in Table 2:

$$\frac{d[\text{HCO}_3^-]}{dt} = -k_{\text{HCO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{HCO}_3^-] \quad (6)$$

$$\frac{d[\text{CO}_3^{2-}]}{dt} = -k_{\text{CO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{CO}_3^{2-}] \quad (7)$$

These ODEs were calculated, as were listed in Table 3. The reaction constants of k_1 , k_2 , and k_3 represent the speed of $\text{PbO}_2(\bullet\text{OH})$, $\text{PbO}_2(\bullet\text{O})$ formation and $\text{PbO}_2(\bullet\text{O})$ decomposition, which were determined by the properties of electrode materials on a certain external condition. The larger value of k_1 means that the production of $\text{HO}\bullet$ and degradation PAP efficient would be enhanced. The larger of k_2 means that the larger probability of physisorbed $\bullet\text{OH}$ could transfer to $\text{PbO}_2(\bullet\text{O})$, which would react with PAP relatively slowly and competed with oxygen evolution from $\text{PbO}_2(\bullet\text{O})$ (represented by k_3). As the concentration of hydroxyl radicals is very little, so the interaction of hydroxyl radicals (k_4) could be ignored. The k_0 and k_{HO} represent the speed of PAP reaction with “active oxygen.” These reaction constants were calculated by least square method and GA.

Rate Constants Calculated and Modeling

The model assumption as follows:

- (1) Solution in reaction system mixed completely, where species concentration uniform.
- (2) Reaction temperature was constant.
- (3) The O_2 evolution of physisorbed “active oxygen” $\text{PbO}_2(\bullet\text{OH})$ was ignored; The O_2 evolution of chemisorbed “active oxygen” $\text{PbO}_2(\bullet\text{O})$ were considered. The $\text{PbO}_2(\bullet\text{OH})$ and $\text{PbO}_2(\bullet\text{O})$ were the main radical reaction with PAP in the solution.

Figure 4 showed the comparison between the predicted and experimental decomposition of PAP under condition of PAP initial concentration $500 \text{ mg}\cdot\text{L}^{-1}$, Na_2SO_4 $0.1 \text{ mol}\cdot\text{L}^{-1}$ and current density $70 \text{ mA}\cdot\text{cm}^{-2}$. The kinetic model was established by least square method and GA. The pseudo reaction constants k_0 was much less than k_{OH} , which indicated that the activity of physisorbed “active oxygen” is much stronger than chemisorbed “active oxygen.”

Kinetics and Mechanism Analysis

Compared with PbO_2 electrode (a), the La doped PbO_2 electrode (b) and Ce doped PbO_2 electrode(c) would lead to increase active

TABLE 3 | The ODEs of the reaction kinetic model.

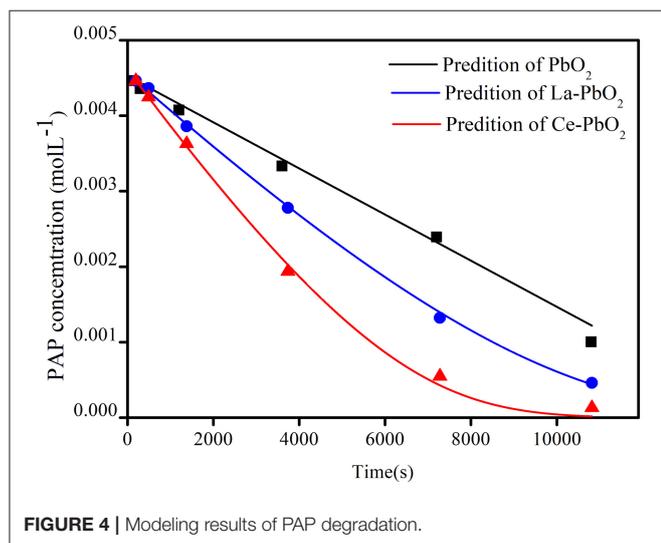
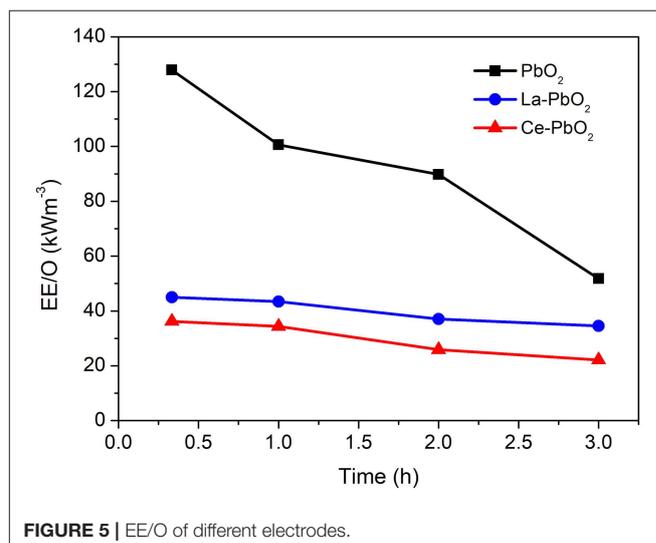
$$\frac{d[\text{PAP}]}{dt} = -k_{\bullet\text{OH}}[\text{PbO}_2(\bullet\text{OH})][\text{PAP}] - k_{\text{O}\bullet}[\text{Pb}(\bullet\text{O})][\text{PAP}] \quad (1)$$

$$\frac{d[\text{Pb}(\bullet\text{OH})]}{dt} = k_1[\text{H}_2\text{O}] - k_{\bullet\text{OH}}[\text{PbO}_2(\bullet\text{OH})][\text{PAP}] - (k_2+k_4)[\text{PbO}_2(\bullet\text{OH})] - k_{\text{HCO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{HCO}_3^-] - k_{\text{CO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{CO}_3^{2-}] \quad (2)$$

$$\frac{d[\text{Pb}(\bullet\text{O})]}{dt} = k_2[\text{PbO}_2(\bullet\text{OH})] - k_{\text{O}\bullet}[\text{PbO}_2(\bullet\text{O})][\text{PAP}] - k_3[\text{PbO}_2(\bullet\text{O})] \quad (3)$$

$$\frac{d[\text{HCO}_3^-]}{dt} = -k_{\text{HCO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{HCO}_3^-] \quad (4)$$

$$\frac{d[\text{CO}_3^{2-}]}{dt} = -k_{\text{CO}_3^{2-}}[\text{PbO}_2(\bullet\text{OH})][\text{CO}_3^{2-}] \quad (5)$$

**FIGURE 4** | Modeling results of PAP degradation.**FIGURE 5** | EE/O of different electrodes.

points on crystal plane (Feng et al., 2008), which would lead to the increase of k_1 and decrease of k_2 and k_3 . After comparing k_1 of undoped and doped PbO_2 electrodes, it could be found that Ce- PbO_2 had a greater k_1 value, which indicated $\text{PbO}_2(\bullet\text{OH})$ was easier produced from electrode material of Ce- PbO_2 . Comparing k_2 and k_3 of doped PbO_2 electrodes, it could be found that Ce- PbO_2 had a smaller k_2 and k_3 value, which indicated the speed of physisorbed “active oxygen” transfer to crystals and lattice oxygen releasing of superoxide was weaker. Therefore, the catalytic efficiency of the three electrodes is Ce- $\text{PbO}_2 > \text{La-PbO}_2 > \text{PbO}_2$.

The intermediates during the PAP degradation were analyzed. Kinds of intermediates were detected at time intervals, including maleic acid, acetic acid, formic acid, NO_2^- and NO_3^- . The results showed that the degradation of PAP would open loop into small molecule acid, such as maleic acid, acetic acid, formic acid on the condition of $\text{PbO}_2(\bullet\text{OH})$ effective. Then kinds of small molecule acids would be degraded into to CO_2 and H_2O in the process of electrochemical oxidation.

EE/O Calculation

The electrical efficiency per log order (EE/O) is one of the best conceptions to describe energy utilization, which could be calculated by the established model. The EE/O calculation using equation (Crittenden et al., 2012; Butcher, 2016):

$$\text{EE/O} = \frac{P \times t}{V \times \lg(C_0/C_t)} \quad (8)$$

Where: EE/O= the electrical efficiency per log order reduction (kWh m^{-3});

P = the average power output (kW), t is the electrolysis time (h);

V = the wastewater volume (m^3);

C_0 = the initial PAP concentration (mg L^{-1});

C_t = the PAP concentration at different time (mg L^{-1});

The results of EE/O at time intervals were showed in Figure 5. The final EE/O of PbO_2 , La- PbO_2 , and Ce- PbO_2 were 51.79 kWh m^{-3} , 34.56 kWh m^{-3} , 22.22 kWh m^{-3} . The EE/O of Ce- PbO_2 was <34% of La- PbO_2 and 55% of PbO_2 .

CONCLUSIONS

The Ce- PbO_2 electrode was successful prepared and used for PAP wastewater treatment. It was found that the novel Ce doped electrode is much more energy saving than La- PbO_2 and PbO_2 . The EE/O of Ce- PbO_2 was 34% less than that of La- PbO_2 and 55% of PbO_2 . A kinetic model was established base on GA calculation, which could help us evaluate the catalytic efficiency of doped PbO_2 electrode and predicted the reaction process on the surface of the electrode. The main advantage of this paper is to give a method for the energy consumption analysis based on pseudo element reaction calculation. This paper could provide basic data and technique reference of the prediction the oxidation reaction process of different electrodes for the electrochemical oxidation application in wastewater treatment.

AUTHOR CONTRIBUTIONS

MW designed and co-carried the experiment, performed the analysis and wrote the manuscript. XY co-carried the experiment.

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Conflict of Interest Statement: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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