

Electrochemical Degradation of Diethyl Phthalate under Different Operating Conditions

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In this study, different operating conditions were tested for the electro-degradation of diethyl phthalate (DEP) (an emerging contaminant) in aqueous solutions. The results show that the DEP electro-degradation followed pseudo first-order kinetics. The degradation efficiencies and (apparent) rate constants of DEP increased with the increase of current density and solution temperature (activation energy = 8.0 kJ/mol), but decreased with initial DEP concentration. The boron-doped diamond (BDD) anode was significantly better than PbO₂ and Pt for DEP degradation and TOC removal. The apparent rate constants were 2.87×10^{-4} , 2.00×10^{-4} , and $5.00 \times 10^{-5} \text{ s}^{-1}$ for the degradation of 50 mg/L DEP at 200 mA/cm² and 25°C in 0.1 M Na₂SO₄ solution at BDD, PbO₂, and Pt anodes, respectively. After 210 min electrolysis, the BDD achieved total mineralization of 50 mg/L DEP in both 0.1 M Na₂SO₄ solution and municipal wastewater treatment plant secondary effluent. Hence, the electrochemical method was a promising process for the effective treatment of DEP and similar pollutants in municipal wastewater.

Keywords: electrochemical degradation; diethyl phthalate; boron-doped diamond (BDD) electrode; mineralization

1. INTRODUCTION

Phthalates (phthalic acid esters) are synthetic organic compounds used as plasticizers in a broad spectrum of industrial and commercial applications, so they are commonly found in a wide range of products, such as food packaging, paints or internal polyvinyl chloride (PVC) coatings, toys, shower curtains, vinyl flooring, personal care articles, and cosmetics [1–3]. The production of phthalates (PAEs) has reached 3.5 million tons per year [4]. Traditional biological treatments are not effective for

the degradation of PAEs [5–7]; hence, PAEs have been frequently detected in surface and groundwater (ng/L to mg/L level) [8–10]. Most of PAEs have been classified as endocrine disruption compounds and as priority pollutants by the European Environment Agency and US Environmental protection agency [11]. When PAEs enter into environment, adverse effects on organisms may occur even in low concentrations [12, 13]. Among PAEs, diethyl phthalate (DEP) is one of the most frequently detected compounds with high water solubility and short-chain structure in different environmental matrices (e.g., surface water, ground water, drinking water, sea water and sediment) [14]. This phenomenon poses an increasing potential risk to human health and ecosystem safety. Therefore, it is greatly in need to develop effective treatment processes for DEP removal in environmental matrices.

In recent years, the removal of DEP has been studied by different processes such as photochemically enhanced Fenton reaction (e.g. UV/H₂O₂/Fe²⁺) [15], ozonation [16], and sonophotocatalysis [17]. However, the complete removal of DEP cannot be obtained in UV/H₂O₂/Fe²⁺ treatment [15]; furthermore, low TOC removal or mineralization was observed in ozonation [16] or sonophotocatalysis [17]. On the other hand, electrochemical oxidation is regarded as an easy control, rapid, clean, and promising approach for the effective degradation and mineralization of persistent organic pollutants in water and wastewater through the aid of electro-generated hydroxyl radicals [18]. For example, Brinzila et al. [19] observed that the electrolysis of tetracycline (150 mg/L) using a BDD anode for 4 hr achieved the removals of COD and TOC by 93% and 87%, respectively. In another study, 94% TOC removal was attained at 50 mA/cm² and 4.5 hr electrolysis for 17β-estradiol (E2) degradation [20]. So far, little attention has been paid for the electrochemical degradation of DEP. Therefore, in this work we explored the electrochemical degradation of DEP under different current densities, temperatures, initial DEP concentrations, anode materials, and water matrices. Additionally, cyclic voltammetry was used to characterize the electrochemical behavior of DEP on BDD. The obtained parameters are useful to design the reactor for DEP electro- degradation and to evaluate the removal of DEP in real environmental samples.

2. MATERIALS AND METHODS

2.1. Chemicals and materials

Table 1. Characteristics of the municipal wastewater treatment plant secondary effluent (MWTPSE)

Parameter	TOC (mg/L)	BOD (mg/L)	COD (mg/L)	Conductivity (μs/cm)	ORP (mV)	pH
Value	37.8	10.4	21.0	2030	299	7.26

ORP: Oxidation-reduction potential (potential versus SCE).

The DEP (with an aromatic structure shown in Fig. 1) was purchased from Chem Service Inc. (USA). Na₂SO₄ (SHOWA Co. Ltd. (Japan)) was employed to prepare the supporting electrolyte. The BDD electrode (substrate: Niobium; BDD coating thickness: 2.0 mm) and Pt anode were supplied

from CONDIAS GmbH (Germany) and SPEMET Co. Ltd. (Taiwan), respectively. The preparation of PbO₂ electrode was provided elsewhere [21]. The separator of electrolytic cell was Nafion-212 (Dupont) (perfluorosulfonic acid cation exchange membrane).

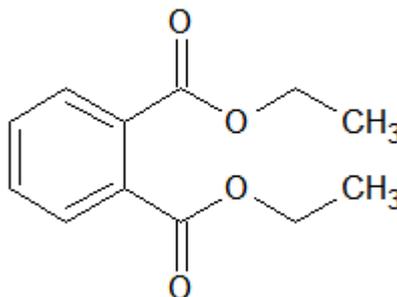


Figure 1. Molecular structure of DEP.

Before use, the separator was heated in 1 M (~3%) H₂O₂ at 65°C for 1 hr to remove organic impurities and then it was washed three times with deionized distilled water (DDW) and stored in DDW. The municipal wastewater treatment plant secondary effluent (MWTPSE) was collected in southern Taiwan. The composition of MWTPSE is provided in Table 1.

2.2. Cyclic voltammetry (CV) of DEP

The electrochemical characteristics of DEP in the prepared solutions were studied with cyclic voltammetry (CV). The potential scan range was -1.0↔2.0 V (starting/ending at -1.0 V vs Ag/AgCl and scan rate = 100 mV/s) for the CV measurements. A CHI 660B electrochemical work station connected with a computer was used to conduct the CV measurements. The working electrode was a BDD disk electrode (WINDSOR SCIENTIFIC Co. Ltd. (UK) with an area of 0.07 cm² and the counter electrode was a platinum wire. An Ag/AgCl electrode (3 M KCl) was used as the reference electrode.

2.3. Degradation experiments

The degradation reaction of DEP in aqueous solution was performed in a divided reactor. The anolyte (100 mL) was 0.1 M Na₂SO₄ or municipal wastewater treatment plant secondary effluent (MWTPSE) spiked with 50 mg/L DEP, whereas the catholyte was only 0.1 M Na₂SO₄ solution. BDD, PbO₂, and Pt were used as the anode while a Ti plate acted as the cathode for the electrolysis/degradation of DEP. These electrodes had the same geometric working surface areas (1 cm²). The experimental conditions were as follows: initial DEP concentration = 25–75 mg/L, current density = 50–200 mA/cm², temperature = 25–60°C, and Na₂SO₄ = 0.1 mol/L. All the experiments for the electro-oxidation of DEP were galvanostatically performed in a thermostatted divided cell with magnetic stirring. A DC power supply (Good Will Instrument Co. Ltd. GPS-2303) was used during the electrolytic experiments. The cell voltage and current were monitored with time based on the readings of DC power supply. During the experiments, samples were collected from the anolyte at regular

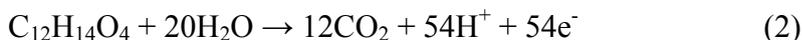
intervals. The concentrations of residual DEP in samples were detected by high-performance liquid chromatography (HPLC), while the total organic carbon (TOC) was analyzed by TOC instrument (see below).

2.4. Analysis

The concentration of DEP in each sample was monitored by HPLC (Hitachi chromaster 5420). A RP-C18 column (150 mm×4.6 mm, particle size, 5µm) was used for separation. The mobile phase was water/methanol (50:50, v/v) (ECHO Chemical Co), with a flow rate of 1.5mL/min. The injection volume was 20 µL and it displayed a well-defined peak for DEP at 224 nm at a retention time of 11.5 min. A personal computer equipped with a Hitachi chromaster system manager for HPLC was used to acquire and process chromatographic data. The linear fitting of calibration curve yielded an R² value of 0.999. Mineralization of DEP was evaluated based on the abatement of corresponding TOC determined using a Shimadzu 5000 Model TOC analyzer at 680°C of combustion temperature. The carrying gas was purified air with a flow rate of 150 mL/min. Calibration of the analyzer was done with potassium hydrogen phthalate standards. The mineralization current efficiency (MCE) for an electrolyzed solution was calculated by Eq. 1 [22]:

$$MCE(\%) = \frac{nFV_s \Delta(TOC)_{\text{exp}}}{4.32 \times 10^7 mIt} \times 100 \quad (1)$$

where n is the number of electrons consumed in the mineralization process, F is the Faraday constant (96,487 C/mol), V_s is the solution volume (L), Δ(TOC)_{exp} is the experimental TOC decay (mg/L), 4.32×10⁷ is a conversion factor (=3600 s/h × 12,000 mg of C/mol), m is the number of carbon atoms (12) in a DEP molecule, I is the applied current (A), and t is the electrolysis time (hour). The value of n is 54 assuming that the overall mineralization of the DEP involves its conversion into CO₂, and the mineralization of DEP is written as the following reaction



The specific energy consumption (SEC, in kWh/kg-DEP) is calculated as follows [23]:

$$SEC = \frac{IVt}{\Delta m_{\text{DEP}}} \quad (3)$$

where I and t are stated above, V is the average cell voltage (V), and Δm_{DEP} is the mass of degraded DEP (g).

3. RESULTS AND DISCUSSION

3.1. Cyclic voltammetric (CV) analysis of DEP on BDD electrode

CV experiments were performed to examine the electrochemical behavior of DEP on BDD in 0.1 M Na₂SO₄ solution (supporting electrolyte) (DEP concentration = 50 mg/L and scan rate = 100 mV/s). Fig. 2a shows the cyclic voltammograms of 0.1 M Na₂SO₄ aqueous solutions with/without 50 mg/L DEP on BDD, and those of anodic scan at 0.6–1.8 V are enlarged and presented in Fig. 2b. The

small oxidation peak at ~1.42 V vs. Ag/AgCl of 0.1 M Na₂SO₄ was associated with the oxidation of sulfate to persulfate [19]. When DEP was present in the 0.1 M Na₂SO₄, the clear anodic peak at ~1.34 V vs Ag/AgCl was attributed to the oxidation of DEP; however, no corresponding reduction peak could be identified, suggesting that the electrochemical oxidation of DEP at BDD was totally irreversible. Additionally, the anodic peak current decreased with the increase of the scanning cycle number. This result is related to the deposition of aromatic intermediates on BDD surface during the oxidation process, and then these intermediates deactivated the electrode. According to some earlier studies [24, 20], the intermediates-deactivated layer can be removed by anodic polarization in the potential region of water decomposition (>2.3 V).

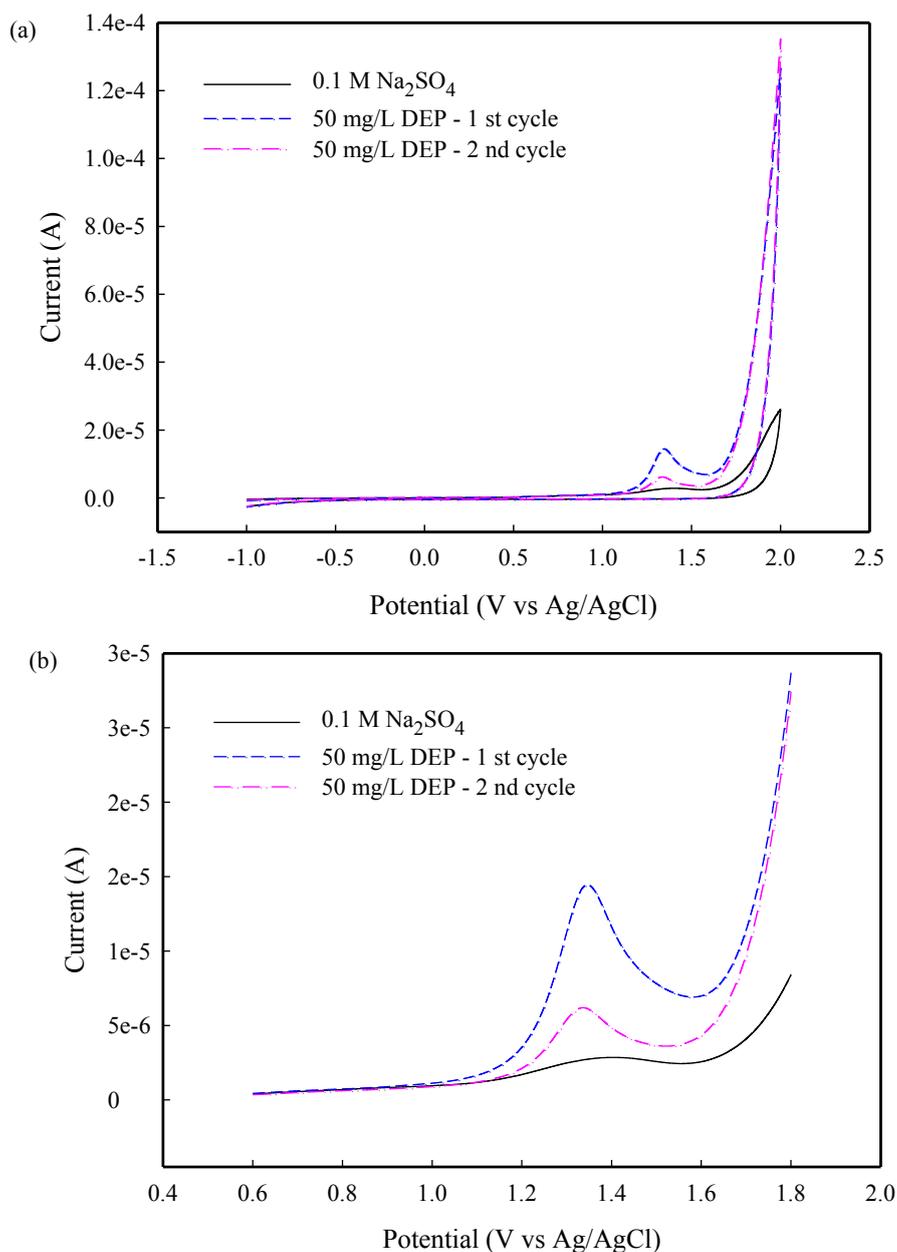


Figure 2. (a) Cyclic voltammograms of 0.1 M Na₂SO₄ aqueous solutions with/without 50 mg/L DEP on BDD at 25°C (scan rate = 100 mV/s); (b) positive scanning data within the potential range of 0.6–1.8 V taken from Fig. 2a.

3.2. Effect of current density on DEP degradation

Applied current density is an important operating factor affecting the electrolysis kinetics. The experiments of constant current density electrolysis at 50–200 mA/cm² and 25°C were performed to examine the degradation of DEP solutions on BDD. The degradation efficiency of DEP can be calculated by Eq. 4: Degradation efficiency = [1 – (C/C₀)]×100% (4)

where C is the residual concentration of DEP at a given electrolytic time and C₀ is the initial concentration of DEP.

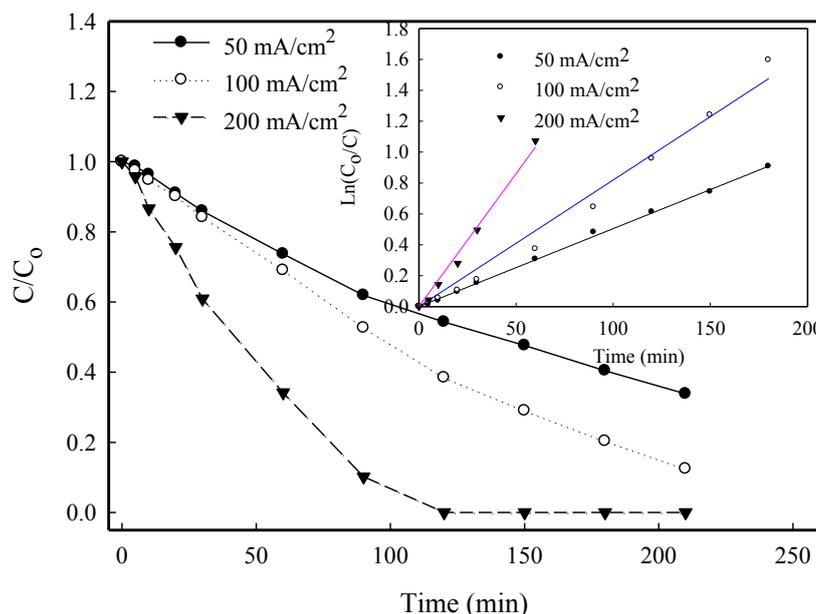


Figure 3. Effect of current density on DEP degradation on BDD; inset: ln(C₀/C) against time (DEP = 50 mg/L and T = 25°C).

Clearly, the degradation efficiency of DEP increased with the increase of current density (Fig. 3). In general, the increase of current density may improve the degradation of target compounds because of the increase of •OH production. Hence, for 120 min electrolysis, the DEP was completely degraded at 200 mA/cm², while the degradation efficiencies were 45% and 60% at 50 and 100 mA/cm², respectively. Moreover, increasing current density also increased the cell voltage (9.3–23.6 V) and SEC. For example, to obtain about 70% DEP degradation efficiency, the SEC value increased from 517 kWh/kg-DEP at 50 mA/cm² to 1430 kWh/kg-DEP at 200 mA/cm². Hence, the current density of 200 mA/cm² was used in the subsequent tests.

The plots of ln(C₀/C) against time of DEP abatement at 50–200 mA/cm² were all linear (R² = 0.983–0.999) (the inset in Fig. 3), indicating that the DEP degradation on BDD could be regarded as a pseudo-first order reaction and the apparent pseudo first-order rate constant (*k_{app}*) can be obtained through Eq. 5:

$$\ln(C_0 / C) = k_{app}C \tag{5}$$

The k_{app} increased with the increase of applied current density and ranged from 8.33×10^{-5} to $2.87 \times 10^{-4} \text{ s}^{-1}$ at 50–200 mA/cm^2 .

3.3. Effect of initial DEP concentration

Fig. 4 shows that the increase of DEP initial concentration (25–75 mg/L) resulted in the decrease of DEP removal efficiency at 200 mA/cm^2 and 25°C on BDD. At 30 min, the degradation efficiency was ~59% at DEP initial concentration = 25 mg/L , while the corresponding data were 39% and 28% at 50 and 75 mg/L , respectively.

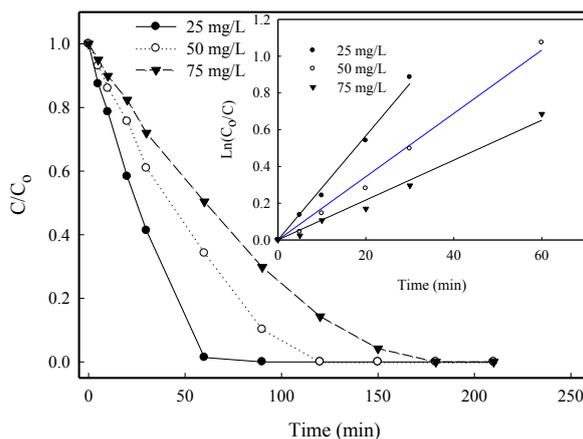


Figure 4. Effect of initial DEP concentration on DEP degradation on BDD; inset: $\ln(C_0/C)$ against time (current density = 200 mA/cm^2 and $T = 25^\circ\text{C}$).

Furthermore, the electrolysis time required for complete removal of DEP was proportional to the initial DEP concentration. As a result, the k_{app} values of DEP degradation also decreased ($(4.73\text{--}1.82) \times 10^{-4} \text{ s}^{-1}$) with DEP initial concentration (25–75 mg/L) (the inset in Fig. 4). Nevertheless, the total amount of degraded DEP increased with increasing DEP initial concentration. For 60 min electrolysis, the degraded DEP amounts were 2.5, 3.3, and 3.75 mg for the DEP initial concentrations of 25, 50, and 75 mg/L , respectively. This phenomenon can be attributed to the better mass transfer in the diffusion layer on BDD surface at higher DEP initial concentration.

3.4. Effect of temperature on DEP degradation

The effect of temperature (25, 40 and 60°C) on DEP electro-degradation is shown in Fig. 5. For 60 min electrolysis, the degradation efficiency of DEP increased from 66% to 78% when the temperature (T) increased from 25°C to 60°C. The result is associated with the fact that the increase of temperature decreases solution viscosity and increases mass transfer [25]. The complete DEP removal could be achieved in the tested temperature range within 120 min electrolysis. The k_{app} values of DEP degradation were 2.87×10^{-4} , 3.23×10^{-4} , and $4.02 \times 10^{-4} \text{ s}^{-1}$ at 25, 40 and 60°C, respectively.

Accordingly, the activation energy calculated using the Arrhenius's law was 8.0 kJ/mol (the R^2 of $\ln k_{app}$ versus $1/T$ plot = 0.99) for the DEP electro-degradation on BDD.

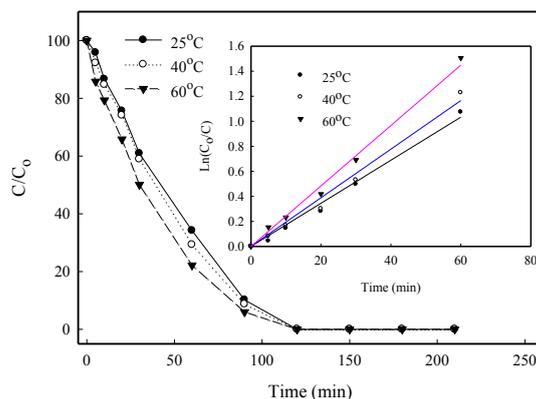


Figure 5. Effect of solution temperature on DEP degradation on BDD; inset: $\ln(C_0/C)$ against time (current density = 200 mA/cm² and DEP = 50 mg/L).

3.5. Effect of anode material on DEP degradation and mineralization

At 200 mA/cm² and 25°C, the complete DEP removal was achieved at 120 and 180 min on BDD and PbO₂, respectively, whereas it needed 210 min on Pt (Fig. 6a).

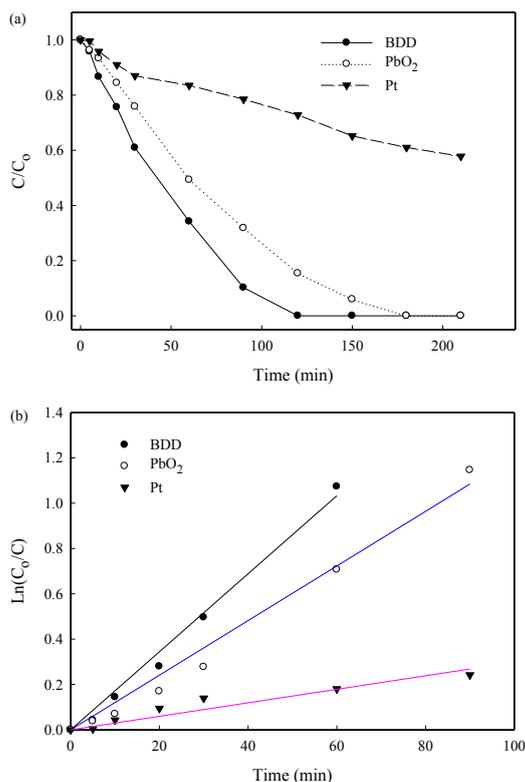


Figure 6. Effect of anode material on DEP degradation. (a) DEP degradation curves (C/C_0 against time) and (b) $\ln(C_0/C)$ against time plots (current density = 200 mA/cm²; DEP = 50 mg/L; T = 25°C).

Therefore, the k_{app} values were in the order $BDD > PbO_2 > Pt$ (2.87×10^{-4} , 2.00×10^{-4} , and $5.00 \times 10^{-5} \text{ s}^{-1}$ at, respectively) (Fig. 6b). The result is associated with the differences in $\bullet OH$ production and adsorption on the tested electrodes; in general, the $\bullet OH$ production on these electrodes varies with the order $BDD > PbO_2 > Pt$ but the order of $\bullet OH$ adsorption on these electrodes is opposite, so more hydroxyl radicals were available for the oxidation of target and intermediate compounds [20, 26].

The Eq. 6 can be used to calculate the removal efficiency of TOC.

$$\text{Degradation efficiency} = [1 - (\text{TOC}/\text{TOC}_0)] \times 100\% \quad (6)$$

where TOC is the residual TOC concentration at a given electrolytic time and TOC_0 is the initial TOC concentration. The performance of tested anodes for TOC removal also followed the order $BDD > PbO_2 > Pt$ (Fig. 7a). On BDD, the complete TOC removal was achieved at 210 min, while at the time only 70% and 36% of TOC were removed on PbO_2 and Pt, respectively. Some earlier studies also reported that BDD electrodes were superior to PbO_2 and Pt anodes for the electrochemical mineralization of organic compounds [27–30].

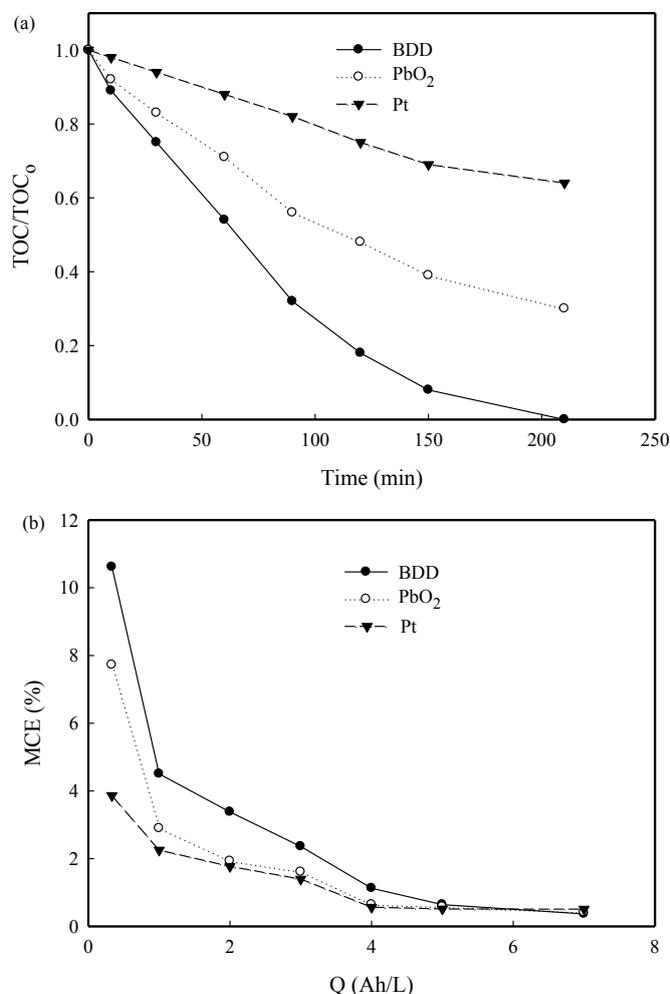


Figure 7. (a) TOC mineralization with time from DEP abatement at different anodes; (b) mineralization current efficiencies (MCE) calculated at specific electrical charges based on the TOC data in Fig. 7a (DEP = 50 mg/L, current density = 200 mA/cm², and T = 25°C).

Fig. 7b depicts the MCE values at three different anodes. As can be seen in this figure, the initial MCE was the highest for all cases, suggesting that the DEP and intermediates were easily degraded and mineralized by hydroxyl radicals. Moreover, the decrease in MCE with time was associated with the decrease of diffusion gradient of compounds of interest toward anode surface due to the gradual depletion of these compounds. In accordance with the result of DEP degradation, the maximum MCEs of the tested anodes were in the order BDD > PbO₂ > Pt.

3.6. Effect of water matrix on DEP degradation

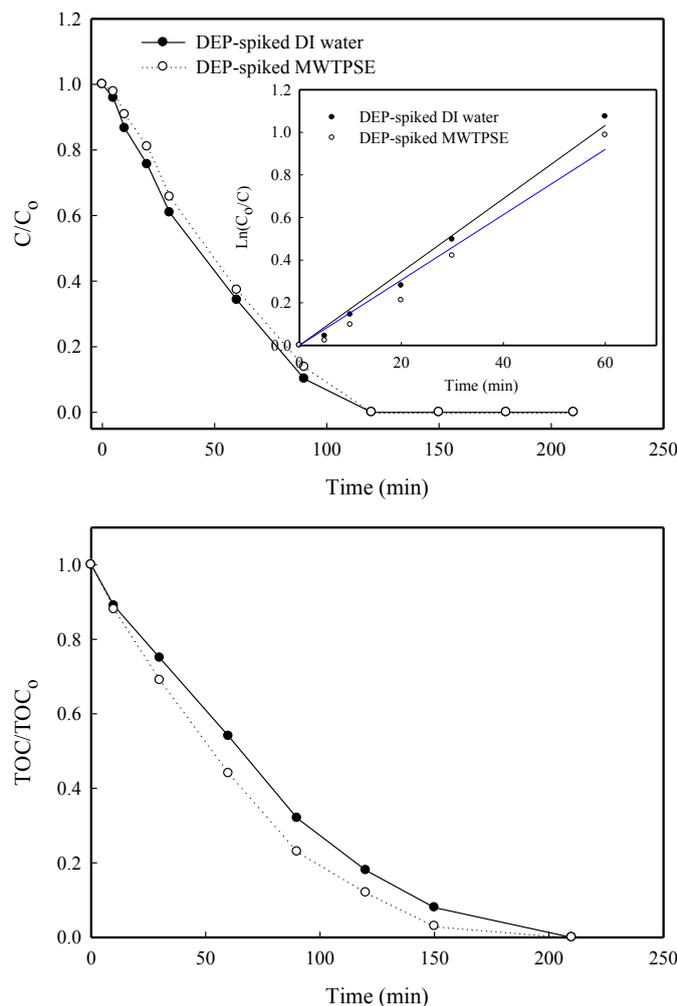


Figure 8. Effect of sample matrix on (a) DEP degradation (inset: $\ln(C_0/C)$ against time) and (b) TOC removal on BDD (DEP = 50 mg/L, current density = 200 mA/cm², and T = 25°C).

Two different types of water matrixes (0.1 M Na₂SO₄ and municipal wastewater treatment plant secondary effluent (MWTPSE) were tested in this work. The original MWTPSE had the values of TOC, BOD, and COD of 37.8, 10.4, and 21.0 mg/L, respectively (Table 1). Both the 0.1 M Na₂SO₄ and MWTPSE were spiked with DEP (50 mg/L) in the electrolysis tests. For comparison and conductivity, sodium sulfate (0.1 M) was also added into the MWTPSE. The degradation efficiency of DEP of MWTPSE was slightly lower than that of 0.1 M Na₂SO₄ solution within 120 min electrolysis

(Fig. 8a). The phenomenon can be attributed to the fact that the dissolved organics originally present in MWTPSE competed with DEP in the oxidation process. The complete DEP degradation was achieved in both water matrixes after 120 min electrolysis. The k_{app} values were 2.87×10^{-4} and $2.55 \times 10^{-4} \text{ s}^{-1}$ in the 0.1 M Na_2SO_4 solution and MWTPSE, respectively (the inset in Fig. 8a). Interestingly, the TOC removal efficiency in the MWTPSE was higher than that in 0.1 M Na_2SO_4 solution (Fig. 8b), revealing the presence of organic compounds which were easier to be mineralized than DEP and DEP-derived intermediates in the MWTPSE. Nevertheless, after 210 min electrolysis, the BDD anode achieved complete TOC removal in both water matrixes.

4. CONCLUSIONS

The electrochemical behavior of DEP was irreversible on the BDD anode and the degradation of DEP was a pseudo-first-order reaction. The degradation efficiency and apparent rate constant of DEP oxidation increased with increasing current density and temperature but decreased with the increase of initial DEP concentration. The performance of tested anodes was in the order $\text{BDD} > \text{PbO}_2 > \text{Pt}$ for DEP degradation and TOC removal. At 200 mA/cm^2 and 25°C , the apparent rate constants of DEP degradation were 2.87×10^{-4} and $2.55 \times 10^{-4} \text{ s}^{-1}$ in 0.1 M Na_2SO_4 solution and MWTPSE, respectively; furthermore, complete DEP degradation and TOC mineralization were achieved in these two solutions. Therefore, the electrochemical approach is a promising technology for the mineralization of DEP in environmental water matrices.

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