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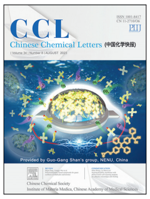

Degradation and detoxification mechanisms of organophosphorus flame retardant tris(1,3-dichloro-2-propyl) phosphate (TDCPP) during electrochemical oxidation process

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ABSTRACT

Electrochemical oxidation of aqueous tris(1,3-dichloro-2-propyl) phosphate (TDCPP) by using Ti/SnO_2-Sb/La-PbO_2 as anode was investigated for the first time, and the degradation mechanisms and toxicity changes of the degradation intermediates were further determined. Results suggested that electrochemical degradation of TDCPP followed *pseudo*-first-order kinetics, and the reaction rate constant (k) was 0.0332 min^{-1} at the applied current density of 10 mA/cm^2 and Na_2SO_4 concentration of 10 mmol/L. There was better TDCPP degradation performance at higher current density. Free hydroxy radical ($^{\bullet}OH$) was proved to play dominant role in TDCPP oxidation *via* quenching experiment, with a relative contribution rate of 60.1%. A total of five intermediates (M1, $C_6H_{11}Cl_4O_4P$; M2, $C_3H_7Cl_2O_4P$; M3, $C_9H_{16}Cl_5O_5P$; M4, $C_9H_{14}Cl_5O_6P$; M5, $C_6H_{10}Cl_3O_6P$) were identified, and the intermediates were further degraded prolonging with the reaction time. Flow cytometer results suggested that the toxicity of TDCPP and degradation intermediates significantly reduced, and the detoxification efficiency was achieved at 78.1% at 180 min. ECOSAR predictive model was used to assess the relative toxicity of TDCPP and the degradation intermediates. The EC_{50} to green algae was 3.59 mg/L for TDCPP, and the values raised to 84, 574, 54.6, 391, and 8920 mg/L for M1, M2, M3, M4, and M5, respectively, indicating that the degradation intermediates are less toxic or not toxic. Electrochemical advanced oxidation process is a valid technology to degrade TDCPP and pose a good detoxification effect.

Organophosphorus flame retardants (OPFRs) are substituted for brominated flame retardant (BFRs), which can improve the flammability, self-extinguishing and smoke-extinguishing of products, the global usage of which has increased significantly in recent years [1,2]. OPFRs have been proven to be an excellent additive to prevent inflammable and combustible materials from igniting and controlling fires, owing to efficient flame retardant properties, environment friendly, high stability and other excellent characteristics [3,4]. However, OPFRs are physically added into the polymer materials such as electronic casings, furniture, household materials without forming a chemical bond in products [5]. Therefore, it is easy to trigger an environment leakage issue *via* wearing, evaporation and leaching. The concentrations of OPFRs detected in global water, soil, dust, and biotic environment are increasing year by year [6,7]. OPFRs can enter the human body from environment through inhalation, breathing, diet, *etc.*, and they have been proven to be neurotoxicity, reproductive toxicity, carcinogenicity and genotoxicity [8,9]. Thus, it is imperative to develop effective elimination technologies to degrade OPFRs.

Advanced oxidation processes (AOPs) are promising techniques that have been universally applied to remove recalcitrant organic compounds in recent years [10,11]. Contaminants are degraded by free radicals with high redox potentials generated from AOPs, such as hydroxyl radical ($^{\bullet}OH$), sulfate radical, and superoxide radical. Researches demonstrated that OPFRs can be degraded by photolysis, however, it has some disadvantages including secondary contamination, low degradation efficiency and harsh reaction environment [12]. Electrochemical advanced oxidation process (EAOPs) has the advantages of simple operation, mild reaction conditions and no secondary pollution, which are considered to a promising method [13,14]. However, there are few studies on degradation of OPFRs by EAOPs, and it is not clear about the degradation mechanisms and toxicity changes of the intermediates produced in the reaction process.

OPFRs can be divided into alkyl, halogenated and aromatic ones depending on different structures, and halogenated ones are the most difficult to be biodegraded [15]. Tris(1,3-dichloro-2-

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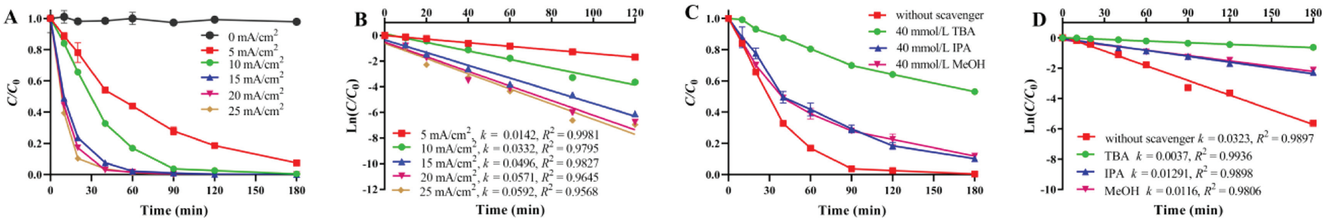

Fig. 1. Degradation efficiency and mechanism of TDCPP in electrochemical oxidation process. (A) Degradation performance at different current densities. (B) Kinetics at different current densities. (C) Effect of quenching agents on the degradation performance. (D) Effect of quenching agents on the degradation kinetics. Experimental conditions: reaction system temperature 25 ± 2 °C, pH 6.6–7.1, initial Na_2SO_4 concentration was 10 mmol/L, and the initial TDCPP concentration was 1 mg/L.

propyl)phosphate (TDCPP), a repeatedly exposed chlorinated OPFR, is demonstrated to be one of the most toxic OFPRs in aquatic system. TDCPP was detected as the highest concentration among all the OPFRs, with a maximum concentration of 218.8 µg/L in wastewater in Suzhou, China [16,17]. TDCPP was chosen as the representative OPFR contaminant to be degraded by electrochemical oxidation process in the current study. The electrochemical degradation of TDCPP was carried out in an electrolytic cell under the galvanostatic mode and performed on a magnetic stirrer (IKA-RCT, Germany) at rotating speed of 600 rpm to enhance the mass transfer efficiency. The PbO_2 electrode has the advantages of simple preparation, strong activity and high stability, and is widely used in the effective degradation of organic pollutants, such as antibiotic drugs, anticancer drugs and even perfluorochemicals [18,19]. Thus, Ti/SnO_2-Sb/La-PbO_2 (50 × 50 mm) was prepared as an anode and two titanium plates (50 × 50 mm) were used as the cathodes in our reaction system. The initial concentration of TDCPP was 1 mg/L, and 10 mmol/L Na_2SO_4 of used as the electrolyte in the electrochemical oxidation process unless otherwise stated, and the working volume was 100 mL. Ti/SnO_2-Sb/La-PbO_2 was fabricated from titanium plate with a dimension of 50 mm × 50 mm × 2 mm (thickness) by means of sol-gel and electrodeposition methods as described in our previous studies [20,21]. The detailed preparation method was provided in Text S1 (Supporting information). The SEM image and XRD pattern of the Ti/SnO_2-Sb/La-PbO_2 anode are shown in Fig. S1 (Supporting information), and results demonstrated its similar physical characterization with other literatures [22,23]. According to our previous report, the service life of Ti/SnO_2-Sb/La-PbO_2 electrode is 68 h under extreme working conditions. The degradation efficiency of Ti/SnO_2-Sb/La-PbO_2 electrode on TDCPP remained above 99.0% after undergoing 30 times of consecutive electrochemical oxidation cycles (Fig. S2 in Supporting information). Degradation mechanisms were determined by investigating the degradation efficiency and identifying the degradation intermediates formed. The actual toxicity changes on *Escherichia coli* (*E. coli*) during electrochemical oxidation of TDCPP were assessed using flow cytometer. Moreover, the acute and chronic toxicity of TDCPP and intermediate products to fish, daphnid, and green algae were calculated using the ECOSAR model (EPA, USA). The effects of applied current densities on TDCPP degradation performance were studied, and the results were illustrated in Figs. 1A and B. TDCPP concentration was analyzed using UPLC-MS/MS (XEVO TQD, Waters, USA). The mobile phase consisted of 80% methanol and 20% water (with 0.1% formic acid). The injection volume was 5 µL and the column temperature was maintained at 30 °C. Results showed that TDCPP was barely degraded at open circuit, demonstrating that there was not any adsorption in the system. After 20 min of reaction, the degradation efficiencies of TDCPP were 21.9%, 34.4%, 76.3%, 82.8%, and 89.7% at the current density of 5, 10, 15, 20 and 25 mA/cm^2, respectively. The degradation efficiency of TDCPP significantly enlarged since the current density increased. This result could be attributed to that the generation rate of $^{\bullet}$OH radicals would be faster under the condition of higher current densities [23]. A previous study applied UV/H_2O_2 oxidation process demonstrated that the degradation efficiency of TDCPP was 84% after 60 min of reaction, while the degradation efficiency of electrochemical oxidation was as high as 98.7% at the current density of 25 mA/cm^2 [24]. Based on the kinetics fitting calculation, the electrochemical oxidation of TDCPP followed *pseudo*-first-order kinetics. The reaction rate constants (k) were 0.0142 min^{-1} (5 mA/cm^2), 0.0332 min^{-1} (10 mA/cm^2), 0.0496 min^{-1} (15 mA/cm^2), 0.0571 min^{-1} (20 mA/cm^2), and 0.0592 min^{-1} (25 mA/cm^2). The highest k value (0.0592 min^{-1}) was lower than that of 0.140 min^{-1} in UV/TiO_2 oxidation process with 50 mg/L of TiO_2 [25]. Correspondingly, the estimated time of TDCPP removal efficiency obtained at 90% were 162.7, 73.1, 39.5, 31.3, and 28.5 min, respectively. Based on the equation for energy consumption calculation, the energy per order (E_{EO}) were 38.6, 42.0, 42.5, 53.2, and 70.1 Wh/L, respectively. The detailed calculation method was displayed in Text S2 (Supporting information). The total organic carbon (TOC) removal of TDCPP by electrochemical oxidation at 10 mA/cm^2 were studied using TOC trace analyzer (VarioTOC, Elementar, Germany), and the results were shown in Fig. S3 (Supporting information). It was seen that TOC continued to reduce prolong with time, and the k value was 0.0044 min^{-1}. The degradation efficiency of TDCPP obtained at 99.6% after 180 min of reaction while the simultaneously removal efficiency of TOC was only achieved at 45.0%, indicating that refractory intermediates were produced in the electrochemical oxidation process. The effect of co-existed humic acid (HA), NO_3^-, Cl^-, and HCO_3^- on the degradation performance of TDCPP were investigated, and results were displayed in Fig. S4 (Supporting information). NO_3^- had no significant effect on the degradation of TDCPP. However, Cl^- and HA showed significantly inhibitory effects at high concentrations. The reaction rate constants decreased from 0.0332 min^{-1} to 0.0297 min^{-1} and 0.0044 min^{-1} in the presence of 10 mg/L and 50 mg/L HA. Besides, it should be noted that HCO_3^- had a slight promoting effect when the HCO_3^- concentration was lower than 100 mg/L.

In order to further infer the electrochemical degradation mechanisms of TDCPP, the quenching experiments were investigated. Isopropyl alcohol (IPA, 40 mmol/L) was used as the quenching agent of free $^{\bullet}$OH, and *tert*–butyl alcohol (TBA, 40 mmol/L) was used as the quenching agent of both adsorbed and free $^{\bullet}$OH. Methanol (MeOH, 40 mmol/L) was used as the quenching agent of $^{\bullet}$OH and $SO_4^{\bullet -}$. The changes of degradation efficiency and kinetics caused by the scavenger are proposed in Figs. 1C and D. Results suggested that the degradation efficiency was greatly inhibited by the quenchers, and the inhibition effect increased with the order of TBA > MeOH > IPA. The degradation efficiency of TDCPP was 96.3% after 90 min of electrochemical oxidation, while only 30.1%, 72.1%, and 70.7% of TDCPP were eliminated in the presence of TBA, IPA, MeOH, respectively. Meanwhile, the k value decreased from 0.0323 min^{-1} to 0.0037 min^{-1}, 0.0129 min^{-1}, 0.0116 min^{-1} when TBA, IPA, MeOH were present in the reaction system. The relative contribution of free radicals were calculated to be 60.1% (free $^{\bullet}$OH) and 28.5% (adsorbed $^{\bullet}$OH) based on the equations given in Text S3

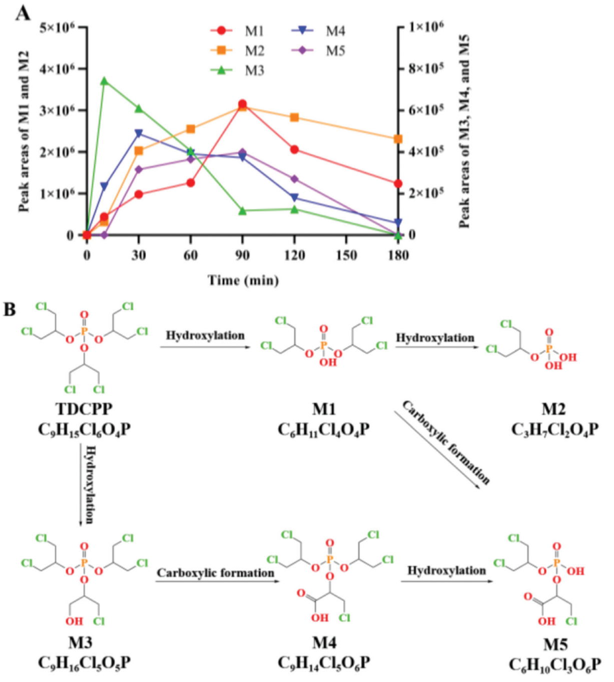

Fig. 2. Degradation intermediates of TDCPP identified in the electrochemical oxidation process. (A) Changes of relative abundance of five intermediate products with reaction time. (B) Proposed degradation pathways of TDCPP by electrochemical oxidation.

(Supporting information), while $SO_4^{\bullet-}$ did not contribute significantly to the degradation of TDCPP. Thus, it can be concluded that free $^{\bullet}OH$ played a dominant role in TDCPP oxidation. The mechanism of the generation of $^{\bullet}OH$ in electrochemical oxidation process is supposed to react with the following reactions (Eqs. 1 and 2):

$$H_2O \rightarrow {}^{\bullet}OH + H^+ + e^- \tag{1}$$

$${}^{\bullet}OH + \text{TDCPP} \rightarrow \text{intermediate products} \tag{2}$$

In order to explore the direct oxidation of TDCPP on Ti/SnO_2-Sb/La-PbO_2, cyclic voltammetry (CV) curves and linear sweep voltammetry (LSV) curves were measured in 10 mmol/L Na_2SO_4 solution with and without TDCPP, and the results were shown in Figs. S5 and S6 (Supporting information). Results illustrated that the oxidation peak and oxidation current of TDCPP were not obviously observed, indicating that there was not any obvious direct oxidation of TDCPP, or indicating that the direct oxidation potential might be higher than oxygen evolution potential where there was much higher oxygen evolution current than TDCPP oxidation current.

Degradation intermediates of TDCPP generated during electrochemical oxidation process were analyzed by LC-Orbitrap-MS/MS (Dionex Ultimate 3000 and Q Exactive Foucus, Thermo Fisher Scientific Inc., Germany). A total of five metabolites including M1 ($C_6H_{11}Cl_4O_4P$, 317.9147), M2 ($C_3H_7Cl_2O_4P$, 207.9457), M3 ($C_9H_{16}Cl_5O_5P$, 409.9169), M4 ($C_9H_{14}Cl_5O_6P$, 423.8968), M5 ($C_6H_{10}Cl_3O_6P$, 313.9278) were identified. MS information for TDCPP and five degradation intermediates was present in Fig. S7 (Supporting information). The formation and re-degradation of intermediate products of TDCPP with reaction time are illustrated in Fig. 2A. Results illustrated that the five intermediates all showed trends of first increase then decrease, indicating that the five intermediates were produced during the electrochemical oxidation process would be further degraded prolonging with reaction time. To be noted, M3 and M5 were completely degraded after 180 min of electrochemical oxidation. However, degradation intermediates M1, M2, and M4 were incompletely degraded, which might be attributed to the recalcitrant TOC in the system. According to the changes of relative abundance, we proposed the possible degradation pathways, as shown in Fig. 2B. Firstly, $^{\bullet}OH$ attacked the $C_3H_5Cl_2{-}O$ bond of TDCPP, forming intermediates M1. Subsequently, M2 was formed following the similar way same as TDCPP transformed to M1 *via* hydroxylation. Besides, $^{\bullet}OH$ could also attacked the C−Cl bond of TDCPP to generate M3 *via* dechlorination. M3 was further oxidized to generate M4 by carboxylic acid formation after the following oxidation reaction. Eventually, $^{\bullet}OH$ attacked the $C_3H_5Cl_2{-}O$ bond of M4, resulting in the formation of M5. Meanwhile, M5 could also be produced after the dechlorination process and oxidation reaction occurred on M1. In general, the degradation pathways of TDCPP mainly includes hydroxylation and dichlorination, which is similar with the previous studies on OPFRs degradation in the AOPs, *i.e.*, electrochemical degradation of tris(2-chloroethyl) phosphate (TCEP) [23], degradation of TCEP by UV/PMS [26], degradation of tris-(2-chloroisopropyl) phosphate (TCPP) by UV/H_2O_2 [27].

Electrochemical oxidation process has been proven to degrade TDCPP efficiently, and some degradation intermediate products would be generated during the process. However, the toxicity of the intermediate products is inconclusive. In order to further study the toxicity changes during electrochemical oxidation of TDCPP, cell death and apoptosis of *E. coli* induced by TDCPP and its intermediates. *E. coli* ATCC 25,922 used for toxicity assessment was purchased from Guangdong Institute of Microbiology, and the accession number was GIM1.173. *E. coli* was firstly added into beef extract peptone medium for 12–18 h, and strains were harvest after washes three times with phosphate buffered solution (PBS, 0.5 mol/L, pH 7.4). Stains of 0.5 g/L (wet weight) were added to the toxicity evaluation medium containing TDCPP and its degradation intermediates products. The composition of beef extract peptone medium and toxicity evaluation medium was present in Text S4 (Supporting information). After 24 h of incubation and exposure, strains were collected after centrifuge. Subsequently, strains were stained with 5 µL Annexin V-FITC and 5 µL propidium iodide (PI) for 15 min in darkness. The dead cells of *E. coli* would be stained with propidium iodide, while the apoptosis cells of *E. coli* would be stained with Annexin V-FITC. Eventually, flow cytometer (FCM) (Beckman Coulter Gallios, USA) was used to measure the fluorescence intensities of two dyes to distinguish live cells, dead cells, and apoptotic cells caused by TDCPP and its intermediates. FCM images demonstrate four types of cells, *i.e.*, dead cells (part of Q1), late apoptotic cells (part of Q2), live cells (part of Q3) and early apoptotic cells (part of Q4). The results of FCM shown in Fig. 3 demonstrated the proportions of active, dead and apoptotic cells, as well as the relative toxicity. The detailed calculation method for relative toxicity was displayed in Text S5 (Supporting information). Compared with the control sample, the proportion of live cells was sharply decreased from 98% to 49.3% after exposed to TDCPP. The results suggested that TDCPP was toxic to *E. coli*, and the live cells increased gradually along with the reaction of electrochemical oxidation. The proportion of live cells increased from 49.3% to 62.4%, 71.2%, 84.6% and 87.3% after 30 min, 60 min, 120 min, and 180 min of reaction, respectively. This indicated that electrochemical oxidation was effective to detoxify TDCPP, and the intermediates are relatively lower toxic or harmful to *E. coli* than its parent compound. The detoxification efficiency of TDCPP was 78.1% in 180 min, while the detoxification efficiency of TCEP was reported to be 94.1% after 90 min of electrochemical oxidation [23]. This indicated that TDCPP was more difficult to detoxify than TCEP in electrochemical oxidation process. The degradation efficiency of TDCPP almost reached 100% after 180 min oxidation reaction. However, 12.0% of apoptotic cells were observed, suggesting that degradation inter-

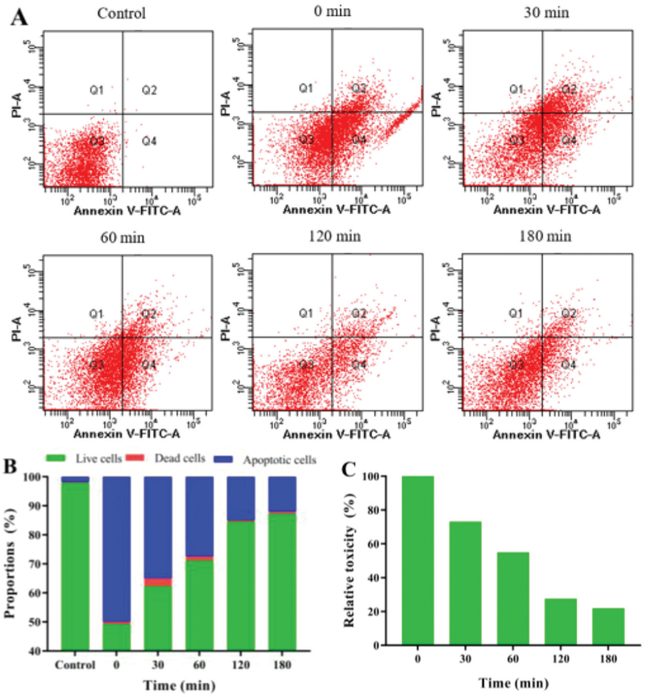

Fig. 3. Toxicity changes of TDCPP and its degradation products. (A) Representative flow cytometry images for electrochemical oxidation of TDCPP. (B) Proportions of active, dead and apoptotic cells exposure to TDCPP and its degradation products. (C) Relative toxicity. The four sections in flow cytometry images represent as below: Q1 dead cells; Q2 late apoptotic cells; Q3 live cells; Q4 early apoptotic cells. Experimental conditions: initial TDCPP concentration was 10 mg/L; dosage of Na_2SO_4 was 10 mmol/L, and the current density was 10 mA/cm^2.

mediate products such as M1, M2 and M4 and other unidentified products could be toxic or harmful.

Based on the ECOSAR predictive program, structural information of TDCPP and its degradation products was used to further evaluate the toxicity by comparing with the toxic chemicals that have been reported, including acute toxicity and chronic toxicity to fish, daphnid, and green algae [28,29]. EC_{50} represents the concentrations of TDCPP and its degradation intermediate products can lead to 50% death of green algae after 96 h exposure, and LC_{50} represents the concentrations of TDCPP and its degradation intermediate products resulted in 50% death of daphnid and fish after 48 h and 96 h exposure. Chronic toxicity values (ChV) represent long-term cumulative toxic effects of chemicals *in vivo*. The toxicity of the chemicals was divided in four stages, *i.e.*, very toxic (red label), toxic (red label), very toxic (orange label), harmful (yellow label), and not harmful (green label), and the result was shown in Table 1. It can be seen that TDCPP possessed most acute and chronic toxic to fish, daphnid, and green algae. M1 and M3 are predicted to be more toxic than other intermediates products. Based on the relative concentration of degradation products showed in Fig. 2A, M1 was degraded incompletely after 180 min of electrochemical oxidation. This could also prove that the residual toxicity of the products was attributed to M1. The EC_{50} to green algae was 3.59 mg/L for TDCPP, and the concentrations raised to 84, 574, 54.6, 391, and 8920 mg/L for M1, M2, M3, M4, and M5, respectively. The toxicity of all the metabolites was lower than TDCPP, resulting in the rapidly detoxification in the electrochemical oxidation process. The acute LC_{50} to fish was 6.28 mg/L for TDCPP, while it increased to 183 mg/L for M1 with a $C_3H_5Cl_2-O$ bond substituted by $-OH$. Similarly, the acute LC_{50} to fish further increased to 2510 mg/L for M2 with two $C_3H_5Cl_2-O$ bonds substituted by $-OH$. This indicated that the $C_3H_5Cl_2-O$ bond substituted by $-OH$ bond would significantly reduce the toxicity. M4 was transformed by carboxylic formation of M3, and its LC_{50} to fish was 461 mg/L, significantly lower than 60.6 mg/L of M3. This evidence suggested that carboxylic formation was conducive to reduce toxicity as well. M5 formed by simultaneously dechlorination and carboxylation, showed the lowest toxicity. Therefore, TDCPP could be detoxified effectively by electrochemical oxidation process, which was consistent with the results of actual toxicity shown by flow cytometry.

Table 1
Estimation of acute and chronic toxicity of TDCPP and its degradation products by using ECOSAR predictive model.

	Acute toxicity (mg/L)			Chronic toxicity (mg/L)		
Chemicals	Fish (LC_{50})	Daphnid (LC_{50})	Green Algae (EC_{50})	Fish (ChV)	Daphnid (ChV)	Green Algae (ChV)
TDCPP	6.28	10.9	3.59	0.33	4.65	1.57
M1	183	105	84	18.2	10.8	22.8
M2	2510	1260	574	213	88.3	115
M3	60.6	127	54.6	4.63	88.6	13.7
M4	461	943	391	33.6	616	106
M5	39300	19800	8920	3330	1380	1780

The toxicity values are classified four grades: very toxic, $LC_{50}/EC_{50}/ChV \leq 1$, red label; toxic, $1 \langle LC_{50}/EC_{50}/ChV \leq 10$, orange label; harmful, $10 < LC_{50}/EC_{50}/ChV \leq 100$, yellow label; not harmful, $LC_{50}/EC_{50}/ChV \rangle 100$, green label.

In summary, TDCPP can be efficiently degraded by EAOPs using Ti/SnO_2-Sb/La-PbO_2 as anode. The degradation efficiencies of TDCPP significantly increased as increasing current densities. $^{\bullet}OH$ oxidation was proved to be the main degradation mechanism of TDCPP in electrochemical oxidation by conducting quenching experiment. TDCPP was almost completely degraded in 180 min, while TOC removal efficiency only achieved 45.0%. The five transformation products of TDCPP were identified by LC-Orbitrap-MS/MS. All of intermediates showed trends of first increase and then decrease, indicating that the five intermediates produced during the electrochemical oxidation process would be further degraded prolonging with reaction time. The actual toxicity of TDCPP and its degradation intermediate products sharply decreased *via* prolonging reaction duration. ECOSAR prediction results reveal that the products are less toxic or not toxic. Electrochemical advanced oxidation process is a promising way to degrade TDCPP and pose a good detoxification effect.

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.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.108090.

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