

Research Article

A Kinetic and Mechanistic Study of Plasma-Induced Degradation of Monochloropropionic Acids in Water by Means of Anodic Contact Glow Discharge Electrolysis

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Decomposition of aqueous monochloropropionic acids (MCPAs) was investigated by means of anodic contact glow discharge electrolysis (CGDE). With the decay of MCPAs, the corresponding total organic carbon (TOC) also decreased smoothly. Furthermore, it was found that chlorine atoms in the MCPAs were released as chloride ions. As the main by-products, oxalic acid and formic acid were detected. The acetic acid (CA), monochloroacetic acid (MCA), and propanedioic acid (PDA) were also detected as the primary intermediates for decomposition of the corresponding MCPAs. The decay of both MCPAs and TOC obeyed the first-order kinetics, respectively. The apparent rate constant for the decay of MCPAs increased with the increase in pK_a values of MCPAs, while that for the decay of TOC was substantially unaffected. The reaction pathway involving the successive attack of hydroxyl radical and the carbon chain cleavage were discussed based on the products and kinetics.

1. Introduction

Chlorinated organic compounds (COCs), including chlorinated aromatic compounds (CACs) and chloroalkyl carboxylic acids (CACAs), have been used in polymer materials, herbicides, fungicides, insecticides, and solvents. As a result, COCs were constantly released into the environment [1–3]. Since COCs are toxic, carcinogenic, and persistent, the degradation of COCs compounds is unquestionably important [4, 5]. In the past decades, the degradation of CACs had been systemically studied by both bioprocesses and advanced oxidation processes (AOPs) [6–13], while, for CACAs, the studies were focusing on chloroacetic acids with two carbon atoms in the carbon chain [14, 15]. The treatments of monochloroacetic acid (MCA), dichloroacetic acid (DCA), and trichloroacetic acid (TCA) by bioprocesses have been reported [16, 17]. To exhaustive degradation of chloroacetic acid, AOPs such as photocatalysis has also been

employed [18–20]. Recently, the attraction has also been devoted to the other CACAs, which have more carbon atoms in the carbon chain. The decomposition of them has been done by bioprocesses [21, 22]; the decompositions kinetics of 2-MCPA and 3-MCPA have also been investigated [23], while few researches by AOPs have been reported yet [24].

One of the hopeful AOPs, named contact glow discharge electrolysis (CGDE) [25, 26], for exhaustive mineralization of organic pollutants has been developed these years [27–30]. In anodic CGDE, plasma is generated locally in the vicinity of an anode contacting the surface of electrolyte. The ionic species in the plasma are accelerated by the steep potential gradient and rush into the solution. Then, in the reaction zone the energized ionic species break several water molecules into hydrogen and hydroxyl radicals [26, 31]. Hydrogen peroxide is one of the main products of anodic CGDE in the solution of inert electrolytes and it should be formed by the recombination of OH radicals [26, 31, 32].

Based on a series of researches on decomposition of organic pollutants by anodic CGDE, it was assumed that hydroxyl radical would act as the most responsible key-species for the destruction of organic pollutants [27–30, 33–38]. Anodic CGDE has also been proved to be one of the hopeful AOPs to mineralize organic pollutants; thus it seems truly opportune to acquire practical knowledge of the aqueous degradation of CACAs induced by anodic CGDE.

Recently, Lei et al. studied the decomposition of trichloroacetic acid (TCA) in water by CGDE [39]; our group reported the degradation of MCA [40]. In the present paper, the comprehensive degradation of two monochloropropionic acids (MCPAs), typical CACAs with three carbon atoms in the carbon chain, by means of anodic CGDE is investigated. The study mainly focused on the cleavage of the carbon chain and kinetic aspects.

2. Experimental

The reaction apparatus and assembly for CGDE experiment are illustrated in Figure 1. A H-type glass cell separated with Nafion membrane N117 made by Techno-Sigma Co. (Okayama, Japan) was employed. And the capacity was 180 mL for each compartment. The anode, from which the discharge was emitted, was a pointed platinum wire (0.6 mm in diameter) sealed into a glass tube which was introduced into the cell. The cathode was a stainless steel plate and immersed into 180 mL of aqueous phosphate solution (8.7 mmol/L potassium dihydrogen phosphate and 30.4 mmol/L disodium hydrogen phosphate, pH = 7.4). Anode and cathode were separated by Nafion membrane N117, across which the electron still could path through. The voltage of 500 V from a DC power supply (ELEPOS PS-1510) was applied between both electrodes to start the run. The depth of discharge electrode (anode) dipped into solution was adjusted so that the average current might be 70 mA. The total electricity passing during the electrolysis was monitored with a digital coulomb meter (Hokuto Denko HF-201). In the course of the reaction, the cell was mounted on an ice-water bath to maintain the temperature of the bulk solution in the range of 10–20 °C. The solution was gently stirred with a magnet bar covered with Teflon.

Reagent-grade MCPAs were used without further purification. Identities of the products, as well as unreacted starting material, were determined by high-performance liquid chromatography (HPLC; Shimadzu LC10A). For the quantification of MCPAs, MCA, PDA, and CA, a Shodex Ionpak KC-811 column (8 ID × 300 mm) connected to an ultraviolet- (UV-) visible detector (Shimadzu SPD-M10A) was used; the eluent used was 50 mol/L perchloric acid. A Shodex Ionpak KC-811 column (8 × 300 mm) with an eluent composed of 0.5 mmol/L and 15 mmol/L perchloric acid + acetonitrile (85:15 V/V) was used together with a conductivity detector (Shimadzu CDD-6A) for the analysis of formic acid, and a Shodex Ionpak KC-811 column with an eluent composed of 0.5 mmol/L perchloric acid was used together with a conductivity detector (Shimadzu CDD-6A) for the analysis of oxalic acid. In addition, Shodex Ionpak IC

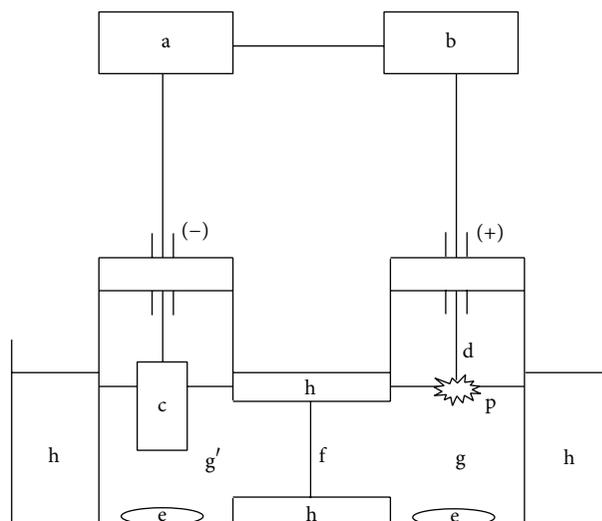


FIGURE 1: Apparatus for CGDE. a: digital coulomb meter; b: DC power supply; c: cathode; d: anode; e: Teflon-coated magnet bar; f: Nafion membrane N117; g: electrolytic solution; g': phosphate buffer solution; h: ice-water bath; p: plasma.

I-524A column with the eluent of 2.5 mmol/L phthalic acid (pH 2.5, flow rate 1.5 mL/min, 40 °C) was used together with a conductivity detector for the analysis of chloride ion. The amount of total organic carbon (TOC) in the solution was measured by a TOC analyser (Shimadzu TOC-V_E).

3. Results and Discussion

3.1. Decomposition of MCPAs. Two of MCPAs, including 2-MCPA and 3-MCPA, were subject to CGDE. Each of them smoothly degraded when CGDE was carried out between the platinum anode and the surface of the solution. With the decay of MCPAs, the amount of total organic carbon (TOC) in water also decreased. It should be noted that the decrease in TOC corresponds to the formation of inorganic carbon (IC). In addition, it was found that chlorine atoms in MCPAs were released as chloride ions.

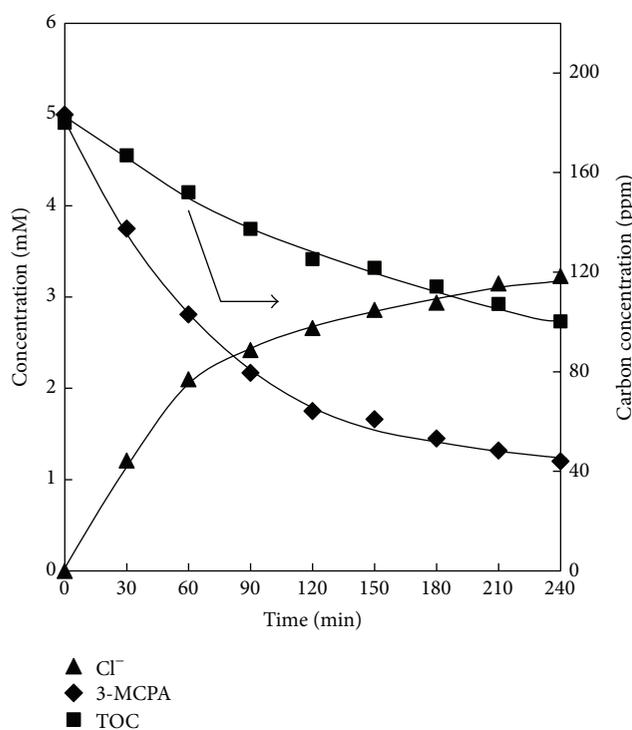
The outline of decomposition of 3-MCPA, as a typical example, is investigated in detail. In Figure 2, the variations of the concentrations of 3-MCPA, TOC, and chloride ion with the discharge time are displayed, where the initial concentration of 3-MCPA was 5.0 mmol/L, corresponding to the carbon concentration of 180 ppm. On starting the reaction, the concentrations of 3-MCPA and TOC decreased monotonously with the discharge time, respectively. After 240 min, 76% of 3-MCPA was consumed and 44% of TOC disappeared. This indicates that CGDE can convert carbon atoms in carbon chain of MCPA to IC, which might exist as hydrogen carbonate in the solution or carbon dioxide released to the gas phase. Furthermore, 65% of chlorine atoms in 3-MCPA were liberated as chloride ions.

The gap between the decay curves of 3-MCPA and TOC indicates the presence of organic intermediate products. In the reaction mixture, a variety of organic substances including some carboxylates were found but most of them

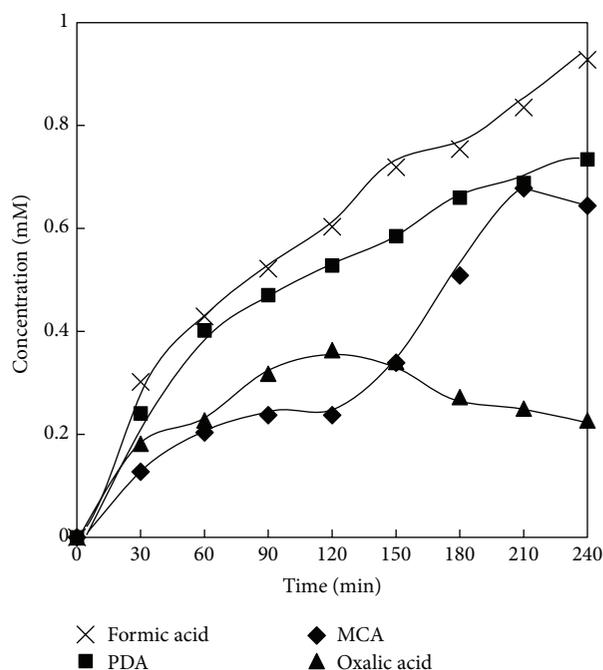
TABLE 1: Elemental yields of products from CGDE of 3-MCPA^a.

Time (min)	3-MCPA conversion (%)	Chlorine			Carbon					
		Cl ⁻	MCA	ND ^c	MCA	PDA	Oxalic acid	Formic acid	IC ^b	ND ^c
0	0	0	0	0	0	0	0	0	0	0
30	27	24	0	3	2	5	2	2	7	10
60	45	42	1	3	3	8	3	3	15	14
90	55	48	2	4	3	9	4	3	21	14
120	59	53	3	2	3	10	5	4	27	11
150	64	57	5	2	4	11	4	4	32	7
180	68	59	8	1	6	12	3	5	37	5
210	74	63	10	1	8	13	3	5	40	4
240	76	65	10	1	8	14	3	6	44	1

^aC₀: 5.0 mmol/L; ^binorganic carbon; ^cnot determined.

FIGURE 2: CGDE of 3-MCPA (C₀: 5.0 mmol/L).

have not been well determined yet, because of the minuteness and nonavailability. MCA, PDA, oxalic acid, and formic acid were only worth quantifying. In Figure 3, their variations of them with the discharge time were shown. Both PDA and MCA were considered as intermediate products in the early period of the 3-MCPA decompositions, and their concentrations increased for the 210 mins. According to the concentrations, PDA was formed more favorably than MCA, suggesting the degradation path resulting in PDA should be more important than that of MCA. Both formic acid and oxalic acid were intermediate products for the further decomposition of 3-MCPA. The concentration of formic acid was always the highest and increased through the reaction,

FIGURE 3: Carboxylates in CGDE of 3-MCPA (C₀: 5.0 mmol/L).

while the concentration of oxalic acid culminated at 120 min and then decreased obviously for the remaining time of the reaction. Formic acid was formed more favorably than oxalic acid, while oxalic acid appeared fairly reactive under the condition. However, it seems improbable that formic acid might result from the decomposition of oxalic acid, since formic acid was not produced in the reaction of oxalic acid as a starting material [35].

The elemental yields of the aforesaid products, namely, IC, PDA, MCA, oxalic acid, formic acid, and Cl⁻, are tabulated as well as the conversion of 3-MCPA in Table 1. The yield was defined as the percentage of moles of a certain element (C or Cl) in each product per initial moles of that in the starting material. The yields of undetermined intermediates, specified as ND, were estimated so as to balance the sum of yields

TABLE 2: Elemental yields of products at 240 min from CGDE of MCPAs^a.

MCPA	Conversion (%)	Chlorine			Carbon						
		Cl ⁻	MCA	ND ^c	MCA	CA	PDA	Oxalic acid	Formic acid	IC ^b	ND ^c
3-MCPA	76	65	10	1	8	*	14	3	6	44	1
2-MCPA	70	69	*	1	*	10	*	2	3	52	3

^aC₀: 5.0 mmol/L; ^binorganic carbon; ^cnot determined; * the corresponding intermediate products were not detected through the reaction time.

TABLE 3: Primary products in CGDE of MCPAs.

Starting material	Primary products			
ClCH ₂ CH ₂ COOH (3-MCPA)	HOOC-COOH (oxalic acid)	HCOOH (formic acid)	ClCH ₂ COOH (MCA)	HOOCCH ₂ COOH (PDA)
CH ₃ CHClCOOH (2-MCPA)	HOOC-COOH (oxalic acid)	HCOOH (formic acid)	CH ₃ COOH (CA)	

with the conversion. Especially in the earlier stage of run, ND was noticeable, but it steadily lowered as the conversion went up. It could be ascertained that the yields of Cl⁻ ion and IC reached 65% and 44%, respectively, at the discharge time of 240 min.

The decomposition of the 2-MCPA by means of CGDE was similarly examined. The behavior of the concentrations of starting materials, TOC, and chloride ion was substantially identical to those in the case of the other 2-MCPA. In Table 2, the elemental yields of products in CGDE of the 3-MCPA and 2-MCPA at the discharge time of 240 min are summarized. It was confirmed that about 70~76% of chlorine atoms and about 50% of carbon atoms could be mineralized by means of CGDE throughout all of MCPAs.

3.2. Products and Decomposition Mechanisms of MCPAs. The information of minor products is also important to elucidate the degradation of MCPAs. Hence, the primary products, which might be formed in the initial stage of decomposition, were searched in the reaction solutions of discharge by using IC, though there were still some intermediate products undetected and undefined due to the intermediate instability and absence of standard samples. The identified products are collected in Table 3.

For all the MCPAs subjected to CGDE, both oxalic acid and formic acid were found as intermediate products. And some other products were also detected. Based on the product survey, the possible formation pathways for the intermediate products were discussed as the following (Schemes 1 and 2).

For the decomposition of 3-MCPA, four intermediates including MCA, PDA, oxalic acid, and formic acid were detected (Table 3). The formation of PDA means the substitution reaction of hydrogen atom on β carbon by hydroxyl radical should take place during CGDE. Since the concentration of oxalic acid increased through CGDE as shown in Table 1, the substitution reaction of hydrogen atoms on α carbon by hydroxyl radical should also take place; subsequently the carbon bond between α carbon and carboxyl group cleavage would occur resulting in oxalic acid and formic acid (Scheme 1). Finally oxalic acid and formic acid were converted to IC and H₂O.

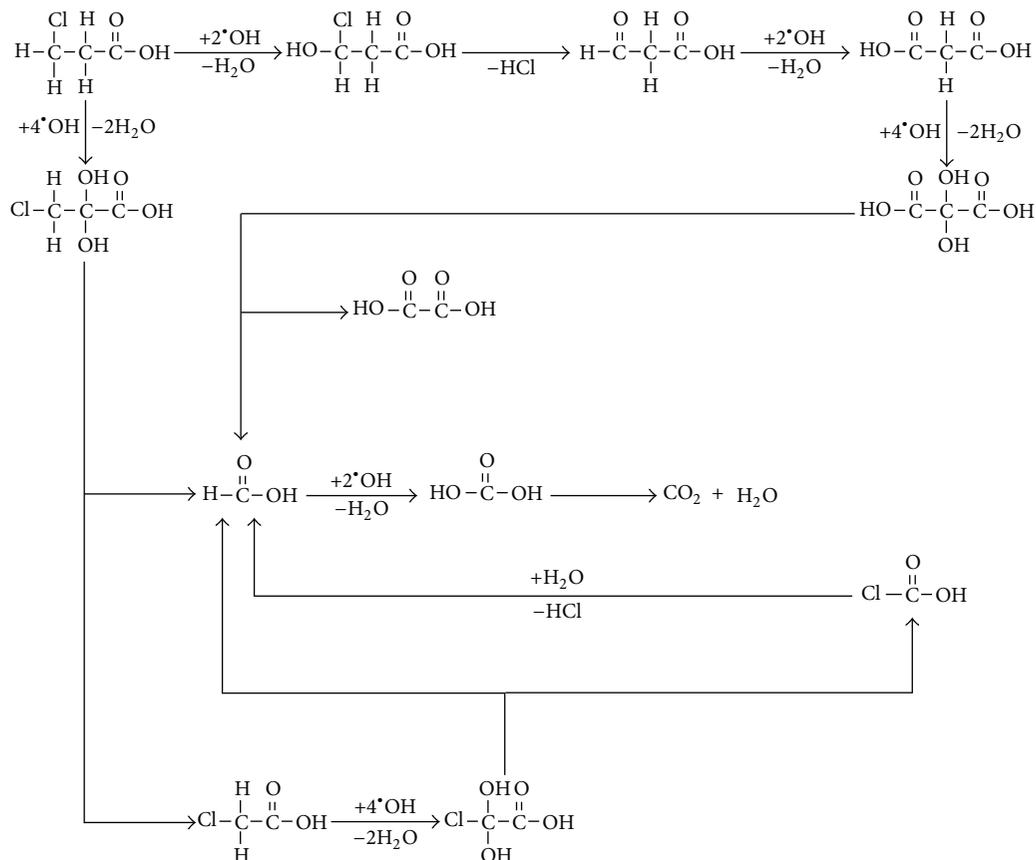
The formation of MCA indicted another reaction pathway; that is, the substitution reaction of hydrogen atoms on α carbon by hydroxyl radical should take place; subsequently the carbon chain cleavage between α carbon and carboxyl group would occur resulting in MCA and formic acid. In addition, during the decomposition of MCA by CGDE, oxalic acid was undetected. Comparing the yields of MCA and PDA in Table 1, one can notice that the formation of PDA was easier than the formation of MCA.

For the decomposition of 2-MCPA, three intermediates including CA, oxalic acid, and formic acid were detected (Table 3). The formation of CA indicted the carbon chain cleavage pathway: firstly substitution reaction of hydrogen atoms on α carbon by hydroxyl radical should take place; subsequently the carbon chain cleavage between α carbon and carboxyl group would occur resulting in CA and formic acid (Scheme 2).

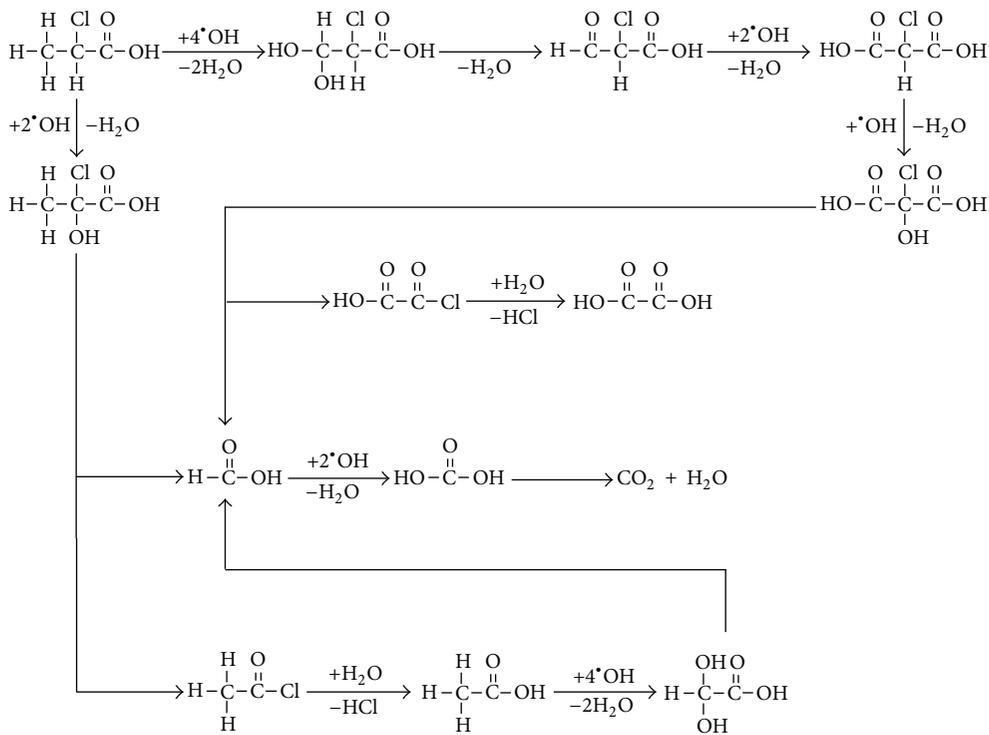
As there was no oxalic acid detected in CGDE of CA, for the formation of oxalic acid another reaction pathway was proposed: both the substitution reactions of hydrogen atom on α and β carbons should take place at the beginning; finally the carbon bond between α carbon and carboxyl group cleavage would occur resulting in oxalic acid and formic acid (Scheme 3).

For both 3-MCPA and 2-MCPA, in which hydrogen atoms bound at the α carbon, intermediate products such as MCA and CA formed were found. These results might be reasonably explained by the idea that the institution of the atoms bonded to carbon in MCPA by hydroxyl radical would occur at the hydrogen atom bonded with α carbon atom to the carboxyl group in MCPA resulting in the cleavage of the bond between α carbon atom and carboxyl group as shown in Schemes 1 and 2.

Based on the product survey, it can be assumed that degradation of aqueous MCPAs by CGDE proceeds in a successive manner as follows: (1) hydroxylation of MCPAs giving rise to substitution reaction of hydrogen or chlorine atom in the carbon chain, subsequently (2) oxidative carbon chain cleavage leading to the formation of carboxylates that includes oxalic acid and formic acid, and ultimately (3) mineralization to inorganic carbon (IC) and Cl⁻.



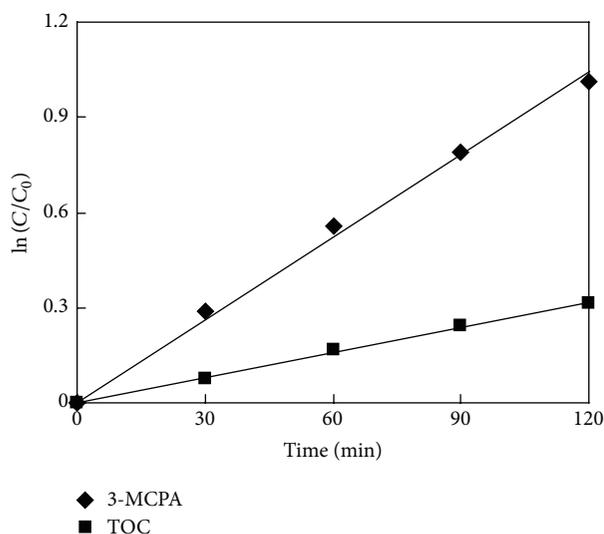
SCHEME 1: Possible decomposition pathway for 3-MCPA by CGDE.



SCHEME 2: Possible decomposition pathway for 2-MCPA by CGDE.



SCHEME 3: Ionization equilibrium for MCPAs.

FIGURE 4: Kinetical plots in CGDE of 3-MCPA (C_0 : 5.0 mmol/L).

3.3. *Kinetics of MCPAs Decomposition.* As the decay curves of the starting material and TOC in Figure 2 appeared to be exponential during the reaction, it was attempted to apply the data to the integral rate equation for the first-order reaction as:

$$\ln\left(\frac{C_0}{C}\right) = kt, \quad (1)$$

where C , C_0 , k , and t denote the concentration of 3-MCPA or TOC at the given reaction time, that is, at $t = 0$, the rate constant, and reaction time, respectively.

In consequence, for each set of data concerning the decay, a straight line with good correlation was obtained (Figure 4). This result indicates that both 3-MCPA and TOC were reacted following the first-order kinetics. The apparent rate constants, $k_{3\text{-MCPA}}$ and k_{TOC} , for the decay of 3-MCPA and TOC were calculated from the slope of each line to be $0.89 \times 10^{-2} \text{ min}^{-1}$ and $0.31 \times 10^{-2} \text{ min}^{-1}$, respectively.

In the reactions of 2-MCPA, the exponential curves for the decay of 2-MCPA and TOC were also obtained. Thus, the rate constants for 2-MCPA were determined similarly and are summarized together with the correlation coefficients in Table 4, where the pK_a data of the MCPAs are inserted. It might be noticed that k_{MCPA} depended on the pK_a ; that is, the higher pK_a , the bigger k_{MCPA} .

The pK_a value is one of the most fundamental physico-chemical properties of acids such as MCPAs and is defined as (2), where K_a is the equilibrium constant for the ionization process of acids:

$$pK_a = -\log K_a. \quad (2)$$

The ionization equilibrium for MCPAs is depicted in Scheme 3. Since the pH values of the reaction solutions are

TABLE 4: Apparent rate constants, k_{MCPA} and k_{TOC} , for the decay of MCPAs and TOC.

MCPA	pK_a	MCPAs		TOC	
		$k/10^{-2} \text{ min}^{-1}$	R^2	$k/10^{-2} \text{ min}^{-1}$	R^2
3-MCPA	4.00	0.89	0.995	0.31	0.996
2-MCPA	2.83	0.72	0.996	0.32	0.996

about 7.4, the ionizations of MCPAs in solution with pK_a value 2.83 to 4.00 are complete. The pK_a values are affected by the chlorine atom positions on the carbon chain of MCPAs: when the chlorine atom was far from the carboxyl group the value of pK_a was bigger. In the case above, the bigger pK_a was, the more electron pairs that the hydrogen atom connected to the carbon would share are. That means the electrophilic attack of hydroxyl radical at these positions resulting in substitution of hydrogen atom would be easier, and the decomposition rate of MCPAs, k_{MCPA} , was bigger for MCPAs with bigger pK_a . On the other hand, k_{TOC} was irrespective of pK_a . This seems reasonable because the rate of final oxidation to inorganic carbon would be supposed to be no more influenced by the characteristics of starting materials such as pK_a .

4. Conclusions

Each of the two MCPAs subject to CGDE was destructed and eventually converted to inorganic carbon. Most of the chlorines in the MCPAs were liberated as chloride ions. In the initial stage, the attack of hydroxyl radical occurred most favorably at the bond between carbon atom and hydrogen atom resulting in institution of hydrogen atom, to some extent, at the bond between carbon atom and chlorine atom resulting in institution of chlorine atom, but never at the bond between carbon and oxygen atom. The consumption of both MCPAs and TOC obeyed the first-order kinetics. The rate constant for the decay of MCPAs, k_{MCPA} , increased with the increase in pK_a , while that for the decay of TOC was irrespective of pK_a . This discrepancy could be rationalized by the decomposition pathway, where MCPAs might cascade through the sequence of hydrogen institution by hydroxyl radical giving rise to the chlorohydroxypropionic acid, subsequent formation of corresponding dicarboxylic acid and/or oxidative chain cleavage leading to the formation of carboxylates, and finally mineralization to inorganic carbon (IC) and Cl^- .

Competing Interests

The authors declare that they have no competing interests.

Acknowledgments

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