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Degradation of monofluorophenols in water irradiated with gaseous plasma

Haiming Yang^{1,2,*}, Giya Mengen¹, Yuki Matsumoto¹, Meguru Tezuka^{1,*}

1. Graduate School of Engineering, Saitama Institute of Technology, 1690 Fusaiji, Fukaya, Saitama 3690293, Japan

2. School of Chemical Engineering, University of Science and Technology Liaoning, Anshan 114051, China

Abstract

Decomposition of aqueous monofluorophenols (MFPs) was investigated by contact glow discharge electrolysis (CGDE). During CGDE, both MFPs and the corresponding total organic carbon (TOC) in water were consumed smoothly, suggesting that carbon atoms of benzene nucleus could be eventually mineralized to inorganic carbon (IC). And all the fluorine atoms in the MFPs were equally converted to fluoride ions. Based on the primary intermediates from each starting materials, it showed that aromatic hydroxylation preferentially occurred at the para- or ortho- position to the phenolic OH group of each MFPs. The disappearance of both MFPs and TOC followed the first-order rate law. The apparent rate constants for the decay of MFPs were independent from the pK_a values of MFPs.

Key words: fluorophenol; decomposition; contact glow discharge electrolysis; first-order rate law; pK_a

Introduction

In recent years, much attention has been paid to the utilization of discharge plasma in and in contact with water generated by DC or AC voltage (Bruggeman and Leys, 2009). In a normal electrolysis using a thin anode wire with sufficient voltage, a stable sheath of solvent vapor can be seen around the tip of the anode, and the current flows as continuous discharge through the sheath. This phenomenon was termed as “Contact Glow Discharge Electrolysis (CGDE)” and has been systematically studied (Hickling and Ingram, 1964; Hickling, 1971; SenGupta and Singh, 1991; Gangal et al., 2009). Thus, it was presumed that the chemical changes in the solution could be caused by the charge species generated in the plasma and accelerated by potential gradient to bombard the solution surface. The resulting hydroxyl radical might combine to give rise to H_2O_2 in chemical yields exceeding Faraday’s law (Hickling, 1971; Mazzocchin et al., 1973; Almubarak and Wood, 1977; Bullock et al., 1980; Sengupta and Singh, 1994; Sengupta et al., 1998a, 1998b; Gangal et al., 2010). Considering the power of hydroxyl radical as an oxidant, a good number of studies have been reported on the water purification by CGDE (Tezuka and Iwasaki, 1997; Gao et al., 2003; Amano and Tezuka, 2006; Tomizawa and Tezuka, 2007; Wang and Jiang, 2008; Gong et al., 2008; Liu, 2009; Jin et al., 2011).

Monofluorophenols (MFPs), one kind of halophenols

like chlorophenols (CPs), have been widely used in industry and agriculture. They have been listed as priority pollutants by the U.S. Environmental Protection Agency (EPA) since 1976 (Luise et al., 2000). The destruction of MFPs has been studied by applying several advanced oxidation processes (AOPs) (Goskonda et al., 2002; Kimura et al., 2010). However, to our best knowledge, the degradation of MFPs by CGDE has not been attempted yet. Lately, we reported the destruction of CPs by CGDE (Yang et al., 2009; Yang and Tezuka, 2011a; Yang and Tezuka, 2011b), proposing that CGDE could be a convenient and useful method to remove CPs from wastewater. In the present work, we launched the degradation of MFPs by CGDE, focusing on the mechanistic and kinetic aspects.

1 Experimental methods

The reaction apparatus and assembly for CGDE experiment are illustrated in **Fig. 1**. A cylindrical glass cell was employed. 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 placed in another glass tube and immersed into 70 mL of aqueous phosphate solution (8.7 mmol/L potassium dihydrogenphosphate and 30.4 mmol/L disodium hydrogenphosphate, pH = 7.4). The voltage of 500 V from a DC power supply 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.

* Corresponding author. E-mail address: tezuka@sit.ac.jp (Meguru Tezuka); yanghaiming80@sina.com (Haiming Yang)

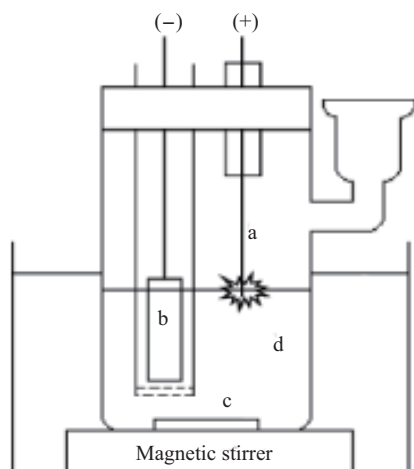


Fig. 1 Apparatus for CGDE. (a) anode; (b) cathode; (c) ice-water bath; (d) electrolytic solution; (e) Teflon-coated magnet bar.

The total electricity passing during the electrolysis was monitored with a digital coulomb-meter. In the course of the reaction, the cell was mounted on an ice-water bath to maintain the temperature of bulk solution at about 20°C. The solution was gently stirred with a magnet bar covered with Teflon. MFPs of reagent grade were purchased and used without further purification. The products, as well as unreacted starting material, were determined by the high performance liquid chromatography (HPLC). They were quantified on GL Sciences Inertsil ODS-4V column. In addition, Shodex Ionpak IC 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 analysis of fluoride ion. The amount of total organic carbon (TOC) in the solution was measured by a TOC analyzer (Shimadzu TOC-VE). The identification of products was based on GC-MS (Shimadzu QP 5050A, Japan) and HPLC (Shimadzu LC10A, Japan)

2 Results and discussion

2.1 Decomposition of MFPs

Figure 2 shows the variations of the concentrations of 4-MFP, TOC and fluoride ion with the discharge time, where the initial concentration of 4-MFP was 6.0 mmol/L, corresponding to the carbon concentration of 420 ppm. On starting the reaction, the concentrations of 4-MFP and TOC decreased monotonously with the discharge time, respectively. After 240 min, 4-MFP was completely removed and most of TOC disappeared, similarly to CGDE of CPs (Yang and Tezuka, 2009; Yang and Tezuka, 2011a; Yang and Tezuka, 2011b). The results indicate that CGDE can be used to effectively decompose 4-MFP and convert carbon atoms of benzene nucleus to inorganic carbon (IC). Furthermore, fluorine atoms in 4-MFP were quantitatively found as fluoride ions.

In the reaction mixture of MFPs, a variety of organic

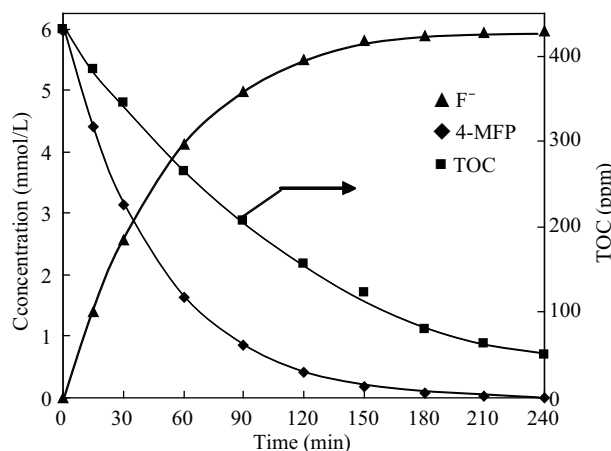


Fig. 2 CGDE of 4-MFP (C_0 : 6.0 mmol/L).

substances including some carboxylates were found but only oxalate and formate were worth quantifying. The variations of them with the discharge time are shown in **Fig. 3**. The concentration of oxalate culminated at 90 min, and then decreased slowly. Whereas, that of formate rose in the starting 60 min and then held on for the rest time of the reaction. The highest concentration of oxalate was several times higher than that of formate, meaning that oxalate was produced more favorably and relatively labile. In addition, it seems improbable that formate might be the product of degradation of oxalate (Amano and Tezuka, 2006).

Table 1 lists the elemental yields of the aforesaid products, namely IC, oxalate, formate, F⁻ and the conversion of 4-MFP. IC was evaluated from the reduction in TOC. The yields were defined as the percentage of moles of a certain element (C or F) in each product per initial moles of that in the starting material. In order to balance the sum of yields with the conversion, the yields of undetermined intermediates, specified as ND, were estimated, too. Although ND of both fluorine and carbon were somewhat substantial in the earlier stage of run, they steadily decreased as the conversion went up. The yields of F⁻ and IC reached 100% and 88%, respectively, at the discharge time of 240 min.

In order to obtain more information of the destruction

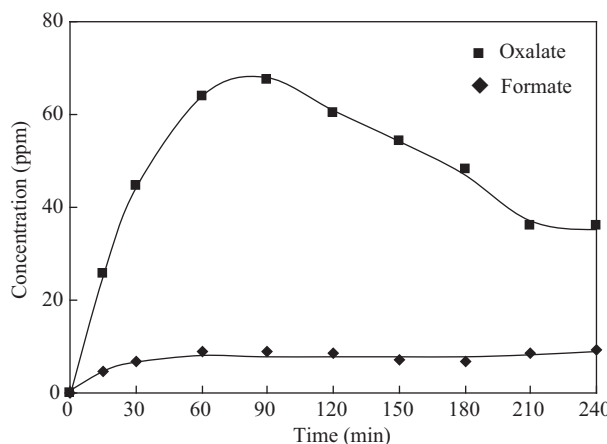


Fig. 3 Carboxylates in CGDE of 4-MFP (C_0 : 6.0 mmol/L).

Table 1 Elemental yields of products from CGDE of 4-MFP^a

Discharge time (min)	4-MFP conversion (%)	Product yield (%)					
		Fluorine		Carbon			
		F ⁻	ND ^c	Formate	Oxalate	IC ^b	ND ^c
15	26	24	2	1	6	11	8
30	47	43	4	2	10	20	15
60	73	69	4	2	15	39	17
90	86	83	3	2	16	52	16
120	93	92	1	2	14	64	13
150	97	97	0	2	13	71	11
180	99	99	0	2	11	82	4
210	99	99	0	2	8	86	3
240	100	100	0	2	8	88	2

^a C₀: 6.0 mmol/L; ^b inorganic carbon; ^c not determined.



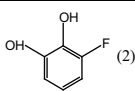

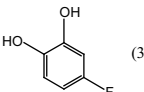
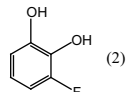
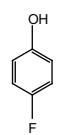
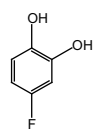
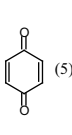
of MFPs by CGDE, the degradations of the other MFPs were also examined. The variations of the concentrations of starting materials, TOC and fluoride ion were essentially identical to those in the case of 4-MFP. The elemental yields of products in CGDE of three MFPs at the discharge time of 240 min were summarized in **Table 2**. It was confirmed that 100% of fluorine atoms were liberated as fluoride ions and almost 90% of organic carbon atoms were lost as IC, meaning the successful mineralization of MFPs by means of CGDE.

2.2 Primary products and degradation pathways of MFPs

During CGDE of MFPs, a diversity of primary products were brought about in the reaction solution especially in the discharge time of 10–30 min. Since the information of minor products is important to elucidate the degradation paths of MFPs. Hence, the primary products were searched in the reaction solutions by using GC-MS. However, they were so minute and susceptible under the present condition that only some of them could be detected. The results were summarized in **Table 3**. When 2-, 3- and 4-MFPs were subjected to CGDE, primary products: catechol (1) and 3-fluorocatechol (2), 4-fluorocatechol (3) and (2), and (3) were detected as the primary products, respectively. These products should be generated by attacking of hydroxyl radical at the ortho-position to the phenolic OH group. Furthermore, in CGDE of 4-MFP, hydroquinone (4) which might be generated by attacking of hydroxyl radical at the para-position to the phenolic OH group, and the corresponding benzoquinone (5) were concurrently found. On the other hand, no products resulting from the attack of hydroxyl radical at meta-position were detected. These results were similar to the previous works about degradation of CPs, and the orientational hydroxylation of MFPs could be explained by the idea that the degradation of MFPs were initiated by the attack of hydroxyl radical, a typical electrophilic species, to the benzene ring of MFPs with OH group, which is known as an ortho and para director.

The possible pathways for the formation of primary products (1)–(5) are depicted in **Schemes 1–3**. Primary

Table 3 Primary products in CGDE of MFPs.

Starting material	Primary products			
 2-MFP	 (1)	 (2)		
 3-MFP	 (3)	 (2)		
 4-MFP	 (4)	 (5)		

products (1)–(5) should subsequently undergo further oxidation leading to overall degradation. Defluorination of MFPs would take place by the attack of hydroxyl radical when fluorine binds at the para or ortho position to the phenolic OH group. Based on the results, one can assume that degradation of aqueous MFPs by CGDE proceeds in a successive manner as follows: (1) hydroxylation of benzene nucleus giving rise to the primary products, subsequently (2) oxidative ring cleavage leading to the formation of carboxylates, and ultimately (3) mineralization to inorganic carbon (IC) and F⁻.

2.3 Kinetics of MFPs decomposition

The variation curves of the starting material and TOC in **Fig. 2** seem to be exponential during CGDE, suggesting the starting material and TOC might disappear according to the first-order rate law. Therefore, the data were applied to integral rate equation for the first-order reaction as Eq. (1):

$$\ln(C_0/C) = kt \quad (1)$$

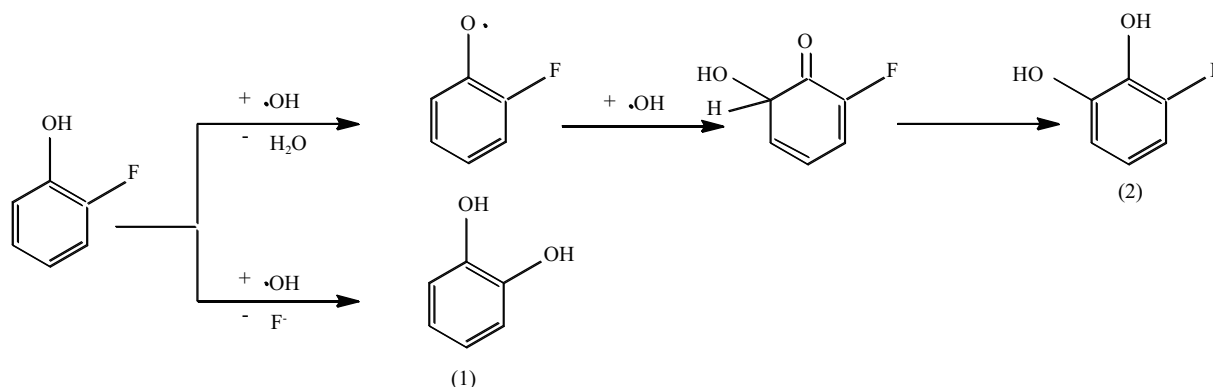
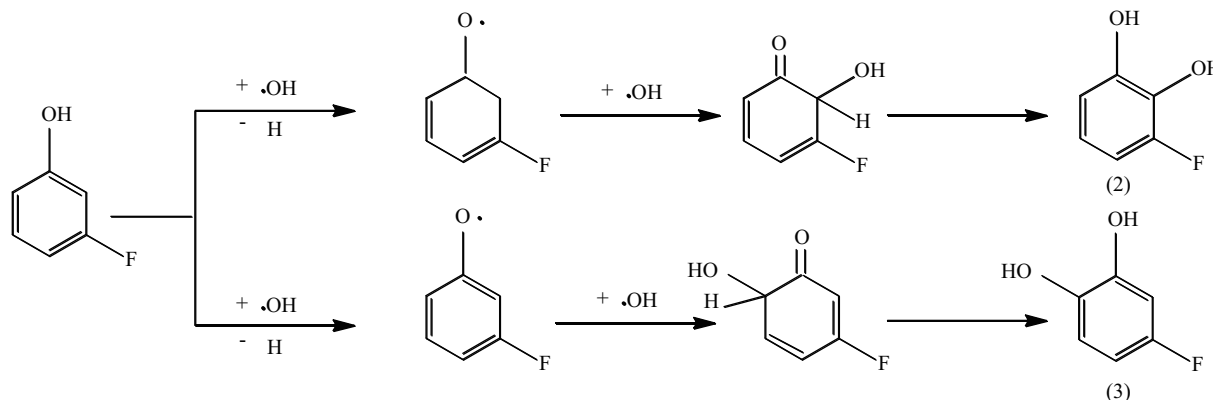
where, C , C_0 , k and t denote the concentration of 4-MFP or TOC at the given reaction time, that at $t = 0$, the rate constant and reaction time, respectively.

As expected, when each set of kinetic data was plotted

Table 2 Elemental yields of products at 240min in CGDE of MFPS^a

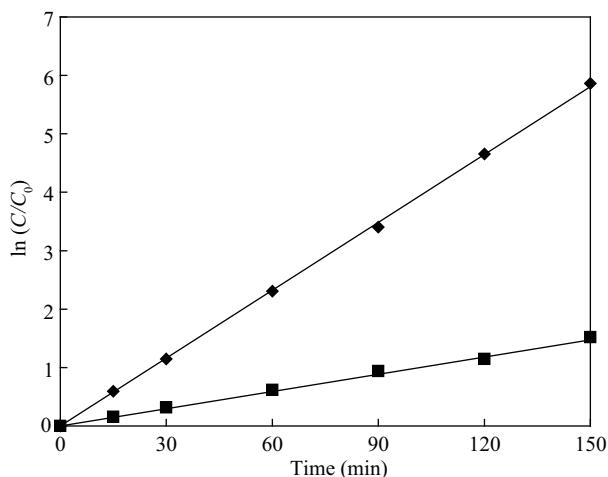
MFPS	Conversion (%)	Product yield (%)					
		Fluorine		Carbon			
		F ⁻	ND ^c	Formate	Oxalate	IC ^b	ND ^c
2-MFP	100	100	0	1	9	87	3
3-MFP	100	100	0	2	8	88	2
4-MFP	100	100	0	2	8	88	2

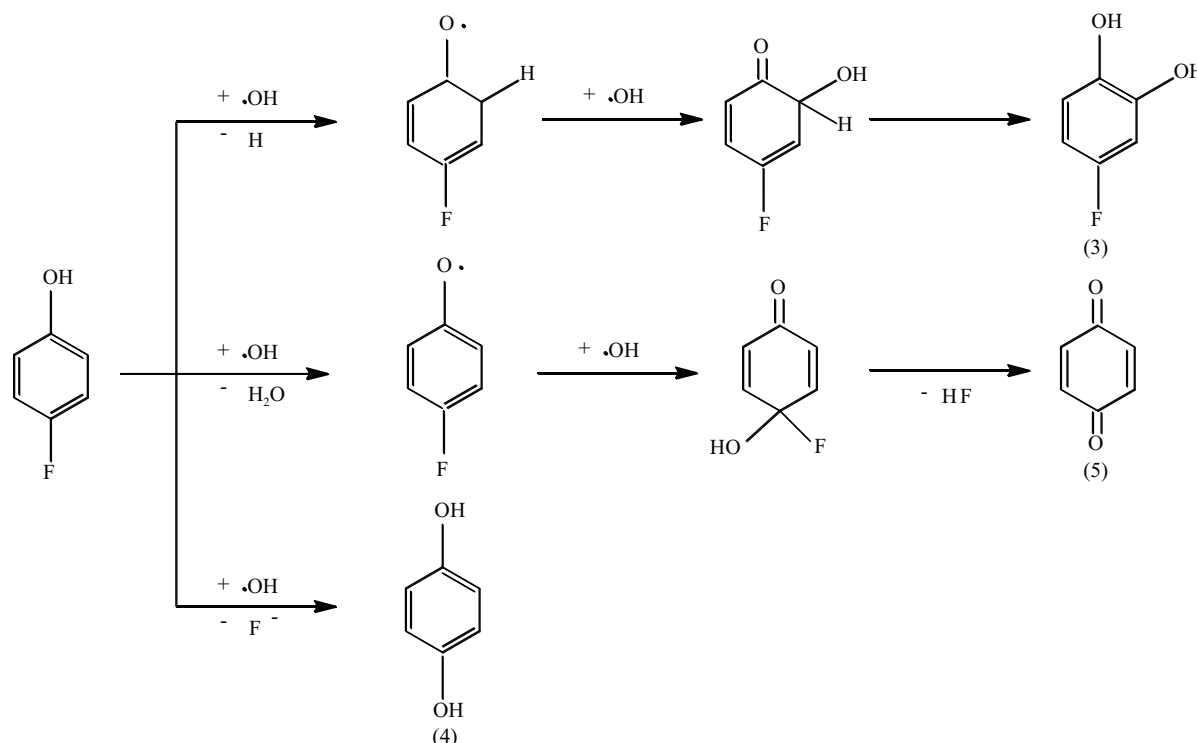
^a C₀: 6.0 mmol/L; ^b inorganic carbon.; ^c not determined.

**Scheme 1** A possible pathway for the formation of (1) and (2) from 2-MFP.**Scheme 2** A possible pathway for the formation of (2) and (3) from 3-MFP.

in conformity to Eq. (1), straight lines with satisfactory correlation were obtained (**Fig. 4**). This result manifests that the consumption of both 4-MFP and TOC obeyed the first-order rate law. The apparent rate constants, $k_{4\text{-MFP}}$ and k_{TOC} , for the decay of 4-MFP and TOC were calculated from the slopes of corresponding lines to be $2.27 \times 10^{-2} \text{ min}^{-1}$ and $0.88 \times 10^{-2} \text{ min}^{-1}$, respectively.

Also in CGDE of the other MFPS, it was demonstrated that the decay of MFPS and TOC followed the same kinetics. Thus, the rate constants for the decay of three isomeric MFPS and TOC were obtained and tabulated with the correlation coefficients in **Table 4**, where the $\text{p}K_{\text{a}}$ values of MFPS were also listed. Both the k_{MFP} and k_{TOC} appear independent from the $\text{p}K_{\text{a}}$, while the k_{CP} increased with decrease in $\text{p}K_{\text{a}}$ in CGDE of CPs (Yang et al., 2009; Yang and Tezuka, 2011a; Yang and Tezuka, 2011b). It suggests that the effects of fluorine atom in MFPS would be significantly different from that of chlorine atom in CPs.

**Fig. 4** Kinetic plots from CGDE of 4-MFP (C₀: 6.0 mmol/L).



Scheme 3 A possible pathway for the formation of (3), (4) and (5) from 4-MFP.

Table 4 Apparent rate constants, k_{MFP} and k_{TOC} , for the decay of MFPs and TOC.

MFPs	pK _a	MFPs		TOC	
		k (10^{-2} min^{-1})	R^2	k (10^{-2} min^{-1})	R^2
2-MFP	8.73	2.29	0.999	0.86	0.996
3-MFP	9.29	2.23	0.999	0.89	0.995
4-MFP	9.89	2.27	0.998	0.88	0.997

3 Conclusions

MFPs subjected to CGDE were completely destroyed and eventually converted to IC and fluoride ions. In the initial stage, the hydroxylation of MFPs occurred preferentially at the para and ortho positions to phenolic OH group due to the fact that phenolic OH group is a para, ortho director and hydroxyl radical is an electrophilic species. The degradation of MFPs might cascade through the sequential pathway as follows; the hydroxylation of benzene nucleus, the subsequent oxidative ring cleavage leading to the formation of carboxylates, and ultimately the mineralization to IC. Both MFPs and TOC were degraded according to the first-order rate law. The effects of the orientation of fluorine atoms on the apparent rate constants, k_{MFP} and k_{TOC} were not so obvious in contrast with the results in CGDE of CPs.

Acknowledgments

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