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Remediation of soils contaminated with polychlorinated biphenyls by microwave-irradiated manganese dioxide

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ABSTRACT

The removal of polychlorinated biphenyls (PCBs) using microwave-irradiated manganese dioxide (MnO_2) in PCB-contaminated soils under different conditions is investigated. The removal of PCB77 in two actual soil samples exhibits strong pH-dependent behavior, and the removal efficiency is higher in acidic soil (Ali-Perudic Ferrosols) than that in neutral soil (Udic Argosols). The removal kinetics of PCB77 using microwave-irradiated MnO_2 under different experimental conditions fits a pseudo-first-order kinetic model well. Both the removal efficiency and the kinetic constant (k) values of PCB77 in Ali-Perudic Ferrosols considerably increase, although in a nonlinear fashion, as the initial amount of MnO_2 is increased, as the treated soil mass is increased, and as the microwave power is increased. The reactivity of three PCBs (PCB28, PCB77, and PCB118) did not present as a function of the degree of chlorination in the reaction with microwave-irradiated MnO_2. The pronounced removal of three PCBs in contaminated soil (all above 95%) indicates that MnO_2 in combination with microwave irradiation is promising for technological applications that seek to remediate sites critically polluted with PCBs.

1. Introduction

Since 1930, polychlorinated biphenyls (PCBs) have been widely used in many countries for industrial purposes, such as dielectric liquids in electrical transformers, capacitors, hydraulic and heat transfer fluids, and plasticizers, because of their excellent stability and thermal properties [1]. In China, the production of PCBs was discontinued in 1974 [2], but the improper disposal of a large amount of disused capacitors resulted in serious leakage of PCBs into the soil; PCB concentrations and distribution in soil have been reported in recent years [3–5]. Li et al. [4] discovered that a total of 57 PCB congeners was identified in Dalian and the mean concentration of total PCBs among all the sites was 2.8 $\mu g\,kg^{-1}$ dry weight (dw) with a range of 1.3 (rural sites) to 4.8 $\mu g\,kg^{-1}$ dw (urban sites) in surface soils. Dong et al. [5] detected and reported similar concentration levels of PCBs in the topsoils of the Tai Lake region. Five dioxin-like PCBs (PCB77, PCB118, PCB126, PCB167, and PCB180) were also detected at a total concentration of 0.352 $\mu g\,kg^{-1}$ dw, or about 33% of $\sum$PCBs [5]. Because of the highly persistent, toxic and bioaccumulative effects of PCBs in both humans and wildlife [6], their levels and distributions have aroused immense concerns especially with regard to environmental health.

PCB-contaminated soils at Superfund sites are generally treated by incineration, but this process often emits dioxins via combustion of the gas stream [7]; dioxins are more toxic than their parent compounds [8]. As a novel and efficient approach, microwave irradiation was used to treat soils contaminated with hazardous wastes [9–11], and this method yielded promising results. Abramovitch et al. [12–14] used microwave energy to decompose polychlorinated aromatic compounds in soil with the assistance of microwave absorbents (Cu_2O, graphite, or pencil lead) and NaOH. They reported that most of the chlorinated aromatics are decomposed, but a minute amount of decomposition product mixtures can be extracted from the soil during remediation because the majority of the dechlorinated products are possibly either mineralized or very tightly bound to the soil. Abramovitch et al. [12] also revealed that higher chlorine-substituted biphenyl contents resulted in lower decomposition efficiency by microwave heating. Liu and Yu [15] investigated the combined effects of microwave and granular activated carbon (GAC) on the removal of PCBs from soils, and indicated that the addition of GAC to soil effectively increases the ability of soil to absorb microwave energy and to obtain high temperatures, resulting in the enhanced degradation of PCBs in soil. Some oxidants, such as manganese dioxide (MnO_2), have been developed for the removal of PCBs in soils. Pizzigallo et al. [16] investigated the mechanochemical removal of five PCBs from soils using MnO_2.

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Table 1
Physicochemical properties of the two tested soils.

Soil type	Organic matter (OM) (g kg^{-1})	pH in $CaCl_2$	CEC (cmol kg^{-1})	Texture (v/v) (%)			Composition of minerals
				Clay	Silt	Sand	
Udic Argosols	5.36	7.38	17.89	22.1	41.6	36.3	Illite, Vermiculite, Montmorillonite
Ali-Perudic Ferrosols	8.88	4.65	24.08	28.2	37.8	34.0	Kaolinite, Hematite

They reported that only 2,2′-dichlorobiphenyl (2,2′-DCB) could be completely removed in 10 days, and the partial removal of 3,3′-DCB (84%), 4,4′-DCB (40%), 2′,3,4-trichlorobiphenyl (30%), and 3,3′,4,4′-tetrachlorobiphenyl (20%) was detected in 15, 27, 90 days, respectively, indicating the low removal efficiency of PCBs by MnO_2 alone.

To the best of our knowledge, studies on the remediation of PCB-contaminated soils by microwave irradiated MnO_2 have not been carried out, although MnO_2 can absorb microwave energy. Therefore, the purpose of this study is to investigate the removal efficiency and kinetics of three PCBs (PCB28, PCB77, and PCB118) in PCB-contaminated soils by microwave-irradiated MnO_2 and examine the effects of the amount of MnO_2, type of contaminated soils, microwave power, and degree of chlorination on the removal of PCBs.

2. Materials and methods

2.1. Experimental materials

2,4,4′-Trichlorobiphenyl (PCB28), 3,3′,4,4′-tetrachlorobiphenyl (PCB77), and 2,3′,4,4′,5-pentachlorobiphenyl (PCB118) were purchased from AccuStandard, Inc. (New Haven, CT). High-performance liquid chromatographic (HPLC)-grade *n*-hexane and acetone were obtained from Merck (Darmstadt, Germany). Ultra pure water was obtained using a Milli-Q Advantage A10 Water Purification System from Millipore (Bedford, MA). MnO_2 was synthesized according to the method by Murray [17]. The synthesized MnO_2 was characterized as δ-MnO_2 according to X-ray diffraction analysis. The Brunauer–Emmentt–Teller-specific surface area of δ-MnO_2 was determined by a Micromeritics Tristar 3000 analyzer to be 250 m^2 g^{-1} at −195 °C over a wide relative pressure range from 0 to 1.0. Scanning electron microscopy images indicated that the synthesized δ-MnO_2 was composed of both granular and rod-shaped particles.

Ali-Perudic Ferrosols were collected from Yingtan of Jiangxi, and Udic Argosols were collected from Nanjing of Jiangsu. They were air-dried in a fume hood, and then ground and sieved through a 20 mesh sieve to remove debris and stones. The soils were sealed in a glass jar for later use. The physicochemical properties of the two soils are listed in Table 1.

2.2. Microwave irradiation

A sample (200 g soil) was weighed and placed in a glass beaker. A total of 5 mL of 200 mg L^{-1} (1 mg) PCB28 (or PCB77 or PCB118) in acetone was added to the soil. Each spiked sample was stored in a brown glass vial after acetone was added, and then completely evaporated in a fume hood with occasional stirring. For all the experiments, the initial additional concentration of each PCB was 5 mg kg^{-1} in soils. The same soil samples that were not spiked were treated as control reactors.

A microwave oven with a frequency of 2450 MHz was used. The microwave apparatus had an adjustable power setting, which could be set to a fixed power output, such as 200, 500 and 800 W, based on the requirements of the experiment. Four 15-mL glass bottles capped with Teflon®-lined septa were placed on the rotary table in the microwave oven as a batch treatment, and each batch treatment was set up as four replicates. An aliquot of PCB-spiked soil was placed into the glass bottle with the addition of δ-MnO_2 as an oxidant. The glass bottle was then mixed using a mini vortex mixer for 5 min to ensure the homogeneity of the PCB-contaminated soil and δ-MnO_2 mixture. The samples were treated by microwave heating under different conditions. A detailed experimental scheme, including five sets of different reaction conditions is described in Table 2. At the designated time, microwave irradiation was stopped, and four bottles were taken out, cooled, and then extracted with 5 mL *n*-hexane in an ultrasonic bath. The slurry was ultrasonically extracted for 30 min and centrifuged for 10 min at 4000 rpm. An aliquot (1 mL) of the supernatant was transferred from each reactor to 2 mL glass vials for chemical analysis via gas chromatography (GC). All the recoveries for PCBs in soil under different treatments were above 70%, satisfying the necessity of quantifying PCBs in this study.

Table 2
The experimental scheme including different reaction conditions.

Set no.	Soil type	PCB type	MnO_2 dosage (g g^{-1} soil)	Microwave power (W)	Soil dosage (g)
1	UA[a]	PCB77	0	800	1.0
			0.1		
	AF[b]		0		
			0.1		
2	AF	PCB77	0	800	1.0
			0.01		
			0.05		
			0.1		
3	AF	PCB77	0.1	200	1.0
				500	
				800	
4	AF	PCB77	0.1	800	1.0
			0.2		2.0
			0.4		4.0
5	AF	PCB28	0.1	800	1.0
		PCB77			
		PCB118			

[a] Udic argosols.
[b] Ali-Perudic Ferrosols.

2.3. Analysis methods

PCBs were analyzed with a Hewlett-Packard 6890N gas chromatograph equipped with a micro-electron capture detector (μ-ECD), using external standards and the peak area calculation method. A BD-5 column (J&W scientific Inc.) with 30.0 m × 0.32 mm × 0.25 μm was used. The injection volume was 1 μl. The injection temperature was set to 250 °C, and the detector temperature was set to 300 °C. Helium was used as the carrier gas, and its flow rate was set to 1 mL min^{-1}. The make-up gas was nitrogen and its flow rate was 60 mL min^{-1}. The column temperature was programmed as follows: 150 °C held for 2 min, adjustment by 10 °C min^{-1} until 280 °C was reached, and finally 280 °C held for 2 min.

3. Results and discussion

3.1. Effect of soil type

The removal profiles of PCB77 in acid soil (Ali-Perudic Ferrosols, pH 4.65) and neutral soil (Udic Argosols, pH 7.38) treated by microwave-irradiated MnO_2 are shown in Fig. 1. The removal efficiencies of PCB77 in two contaminated soils were observed to be approximately 10% in the absence of MnO_2. However, the removal efficiencies of PCB77 in the presence of MnO_2 under microwave irradiation were found to be approximately 90% and 40% in Ali-Perudic Ferrosols and Udic Argosols, respectively. This result shows that the addition of MnO_2 can significantly increase the removal efficiency of PCB77 in two different types of actual soil samples. The removal of PCB77 in the two actual soil samples also exhibited strong pH dependence. The removal efficiency by microwave-irradiated MnO_2 was considerably higher in Ali-Perudic Ferrosols than in Udic Argosols because Ali-Perudic Ferrosols has a lower pH than Udic Argosols (Table 1). This result is in good agreement with a previous study on the removal of PCB77 in diatomite, where acidic conditions were more suitable for the removal of PCB77 by microwave-irradiated MnO_2 [18]. Microwave heating can cause "hotspot" formation, an important feature that arises because of the nonlinear dependence of the electromagnetic and thermal properties of a material on temperature [19,20]. Guan et al. [21] reported that MnO_2 could be used as a microwave absorbent

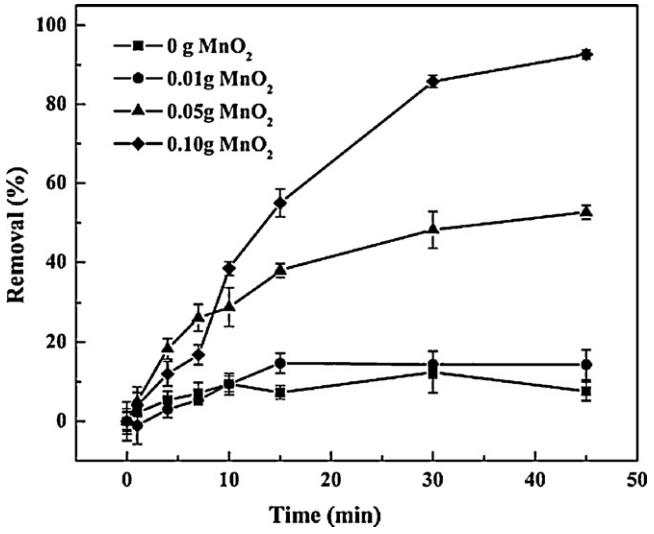

Fig. 1. Removal profiles of 5 mg kg^{-1} PCB77 in two tested soil samples using 800 W microwave irradiation with and without addition of 10% MnO_2 (AF: Ali-Perudic Ferrosols, UA: Udic Argosols).

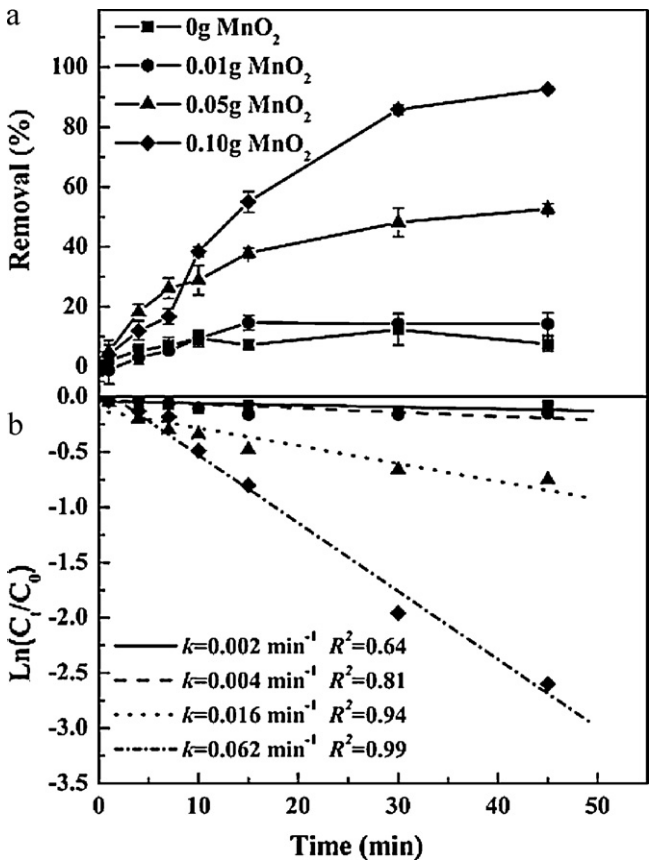

Fig. 2. (a) Removal profiles and (b) kinetics of 5 mg kg^{-1} PCB77 in Ali-Perudic Ferrosols using 800 W microwave irradiation with addition of different amounts of MnO_2.

with a very broad bandwidth, and its temperature could increase from 298 to 1378 K in 100 s at a speed of 10.80 K/s when subjected to electromagnetic wave irradiation [22]. These previous studies indicate that the surface of MnO_2 particles could have many such hotspots at temperatures of approximately 1378 K, resulting in the ability to accelerate the oxidative removal of PCB77 under microwave irradiation. The effects of additional amounts of MnO_2, microwave power, treated soil mass, and a degree of chlorination on the removal of PCBs in Ali-Perudic Ferrosols were also investigated in this study, and the details are described in the following sections.

3.2. Effect of MnO_2 amount

The removal efficiencies and kinetics of PCB77 in Ali-Perudic Ferrosols with varying dosages of MnO_2 are shown in Fig. 2. The removal efficiencies of PCB77 with the addition of 0.01, 0.05, and 0.10 g MnO_2 in 1.0 g contaminated soil after microwave irradiation for 45 min were enhanced by approximately 14.3%, 52.7% and 92.6%, respectively, compared to the removal efficiency of PCB77 (approximately 7.6%) without the addition of MnO_2 (Fig. 2a), indicating that the removal of PCB77 increased along with increasing amounts of MnO_2. Fig. 2b shows that the removal kinetics of PCB77 fits a pseudo-first-order kinetic model well. The apparent kinetic constant (k) increased from 0.002 min^{-1} (without the addition of MnO_2) to 0.004, 0.016, and 0.062 min^{-1} in the presence of 0.01, 0.05, and 0.10 g MnO_2, respectively. The enhancement of removal efficiency and the k value became more pronounced as the amount of MnO_2 was increased, but such a correlation was not linear. In this study, MnO_2 played both the roles of an oxidant and a microwave

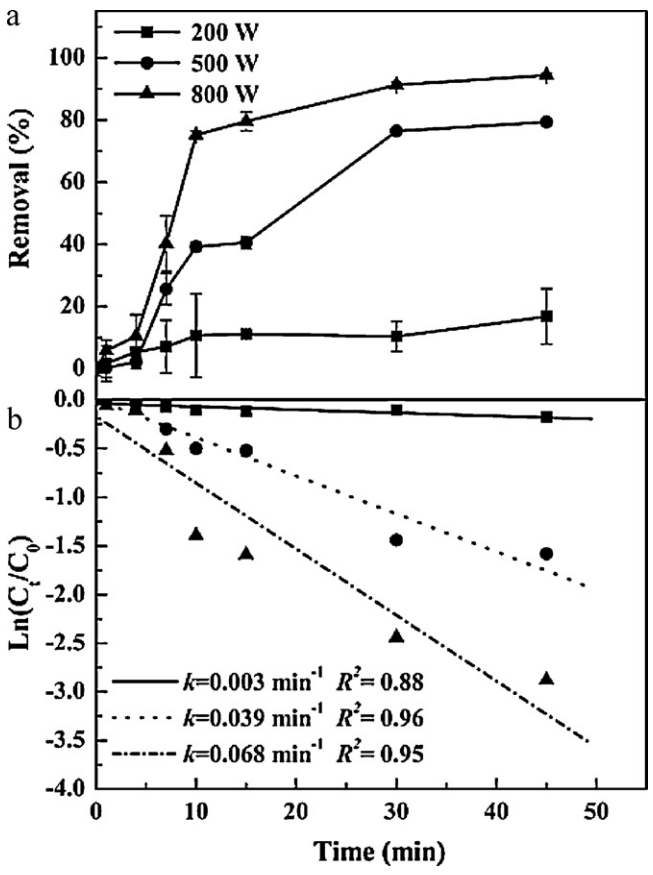

Fig. 3. (a) Removal profiles and (b) kinetics of 5 mg kg^{-1} PCB77 in Ali-Perudic Ferrosols using microwave irradiation at different levels of microwave power.

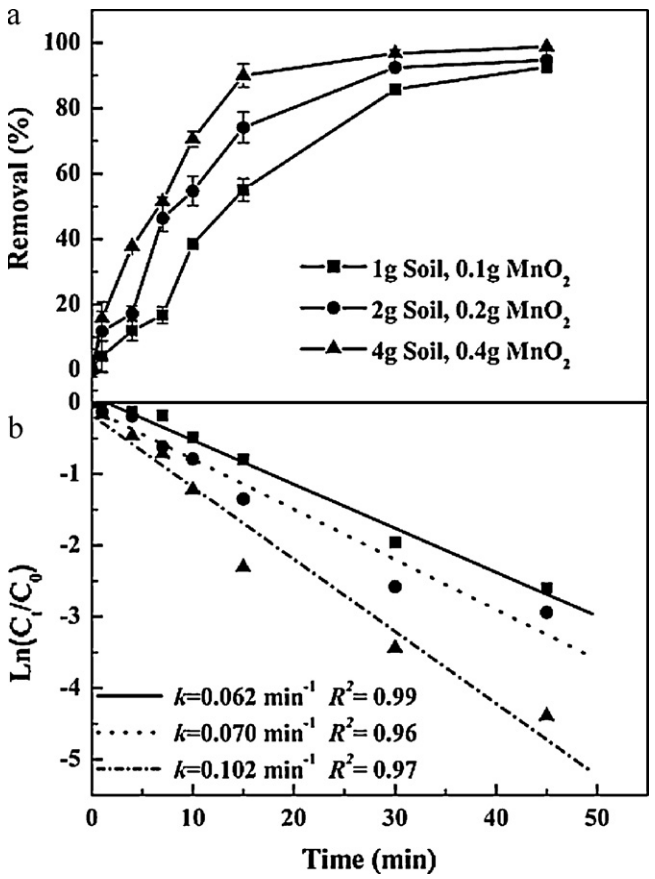

Fig. 4. (a) Removal profiles and (b) kinetics of 5 mg kg^{-1} PCB77 in Ali-Perudic Ferrosols using 800 W microwave irradiation at different masses of treated soil.

absorbent; thus, increase in the initial amount of MnO_2 added can lead to the absorption of more microwave energy, effectively enhancing MnO_2 reactivity and oxidizing more PCB77.

3.3. Effect of microwave power

Microwave power is a crucial factor because it is the sole energy source in the reaction system. Three levels of microwave power (200, 500, and 800 W) were used to test the effect of power outputs on the removal efficiencies and kinetics of PCB77 in the presence of 0.10 g MnO_2 in 1 g contaminated soil. The results show that increases in microwave power can enhance the removal efficiencies and kinetics of PCB77 in soil. Fig. 3a shows that higher removal efficiency of PCB77 occurred at higher microwave powers, and the removal efficiencies were approximately 16.8%, 79.4% and 94.4% after irradiation for 45 min at microwave powers of 200, 500, and 800 W, respectively. The removal kinetics fits a pseudo-first-order kinetic model well in Fig. 3b. The k values were 0.003, 0.039, and 0.068 min^{-1} at microwave power levels of 200, 500, and 800 W, respectively. The removal efficiency and k value of PCB77 increased along with increasing microwave power, although again in a nonlinear fashion. Higher microwave powers can supply more energy to MnO_2 to react with PCB77, which consequently results in the improved removal efficiencies and k values observed in this study.

3.4. Effect of treated soil mass

Fig. 4 presents the removal profiles and kinetics of PCB77 in Ali-Perudic Ferrosols at three different treated soil masses (1, 2 and 4 g) using 800 W microwave power with the addition of 10% MnO_2 to the treated soil mass. PCB77 was removed more rapidly with larger treated soil masses, especially when the microwave irradiation time was less than 15 min (Fig. 4a), which was also proven by the k values in Fig. 4b, in which the removal kinetics of PCB77 fits a pseudo-first-order kinetic model. The k values were 0.062, 0.070 and 0.102 min^{-1} when the treated soil masses were set to 1, 2 and 4 g, respectively. Based on the result where approximately 10% PCB77 could be removed in Ali-Perudic Ferrosols without MnO_2 by microwave irradiation, as shown in Fig. 1, it was clear that PCB-contaminated soil could absorb microwave energy to remove PCBs. Thus, larger masses of treated soil can absorb more microwaves and remove more PCB77, resulting in an increase in the removal efficiency and kinetics of PCB77 with increasing treated soil mass, although in a nonlinear fashion. The results in Fig. 4 also indicate that microwave irradiation is an excellent energy field for removing hazardous pollutants from soil, in agreement with many previous studies [23–26]. The enhancing effect of treated soil mass on the removal efficiency and rate of PCB77 demonstrates that the remediation of PCB-contaminated soil by microwave-irradiated MnO_2 is much more practical and efficient compared to mechanochemical treatments [16].

3.5. Effect of chloro-substituents

The position and number of chloro-substituents on the PCB structure controls the chemical reactivity, toxicity, and bioavailability of PCB [16,27,28]. The so-called coplanar congeners, such as PCB77, are the most toxic PCBs, and are hardly biodegraded in soil. Abiotic transformation of PCB77 also exhibited remarkably low removal efficiency in the presence of MnO_2 compared to dichlorobiphenyls [16]. PCB28, PCB77, and PCB118 were chosen to evaluate the effect of the degree of chlorination on the removal efficiency

Table 3
Removal of three PCBs using 800 W microwave irradiation with addition of 0.1 g MnO_2 in 1.0 g Ali-Perudic Ferrosols soil.

Irradiation time (min)	Removal (%)		
	PCB28	PCB77	PCB118
4	51.1 ± 7.4	38.7 ± 6.0	30.6 ± 3.3
10	85.3 ± 2.2	78.9 ± 3.3	75.6 ± 2.0
15	92.1 ± 3.2	88.1 ± 2.7	86.1 ± 5.9
30	98.5 ± 1.1	99.8 ± 0.1	96.9 ± 2.3
45	97.9 ± 0.2	97.2 ± 1.8	96.2 ± 0.4

using 800 W microwave irradiation with the addition of 0.1 g MnO_2 in 1.0 g Ali-Perudic Ferrosols. Table 3 lists the removal efficiencies of the three PCBs, all of which increased with the increasing irradiation time. The removal efficiency of PCB28 was slightly higher than those of PCB77 and PCB118 when the microwave irradiation time was 5, 10 and 15 min; no significant difference was observed in the removal of the three PCBs after irradiation for 30 and 45 min. In previous studies, a PCB congener with a lower degree of chlorination will exhibit higher reactivity in the process of mechanochemical degree of dechlorination by MnO_2 [16] and oxidative biodegradation by aerobic bacteria [29]. However, the reactivity of PCBs did not present as a function of the degree of chlorination in the reaction with microwave-irradiated MnO_2. No considerable difference in the removal efficiency was found among PCB28, PCB77, and PCB118 at the end of the reaction. Although PCB77 has a comparatively stable coplanar structure, 97.2% of the PCB77 was easily removed by microwave-irradiated MnO_2 after 45 min of irradiation compared to the removal of 97.9% of PCB28 and 96.2% of PCB118. The results obtained in this work suggest that the use of MnO_2 in combination with microwave irradiation is promising for the complete removal of nonbiodegradable PCBs from soil.

4. Conclusion

The removal of PCB77 using microwave-irradiated MnO_2 in Ali-Perudic Ferrosols was significantly higher than that in Udic Argosols due to the higher acidity of the former compared to the latter. The removal kinetics of PCB77 using microwave-irradiated MnO_2 under different experimental conditions fits a pseudo-first-order kinetic model well. Both the removal efficiency and k values of PCB77 in Ali-Perudic Ferrosols considerably increased, although in a non linear fashion, as the initial amount of MnO_2 was increased, as the soil mass was treated, and as the microwave power increased. The reactivity of three PCBs (PCB28, PCB77, and PCB118) did not present as a function of the degree of chlorination in the reaction with microwave-irradiated MnO_2. No considerable difference in the removal efficiency was found among PCB28, PCB77, and PCB118 at the end of the reaction. The pronounced removal of three PCBs in contaminated soil (all above 95%) indicated that the use of MnO_2 in combination with microwave irradiation is promising for technological applications to remediate sites critically polluted with PCBs.

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