

Chapter 2

Remediation of Phenanthrene-Contaminated Soil Using Non-thermal Plasma Fluidized Bed

Abstract A non-thermal plasma fluidized bed (PFB) was developed for remediating phenanthrene (PHE)-contaminated soil. After a 25-min treatment, 95% of the PHE was removed with an energy density of 5960 J/g soil, air flow rate of 20 L/min, and soil moisture of 10%. The effects of the solid bed location, energy density, the flow rate, and the carrier gas were explored. It was found that decreasing the distance between the solid bed and electrodes properly and increasing the input energy density were favorable for PHE degradation. There existed appropriate flow rate and soil moisture for a higher efficiency. By means of various methods, the roles of the active species on the degradation efficiency and the products formed during discharge were analyzed. Possible mechanism of the PHE degradation by the PFB was proposed. Furthermore, a brief comparison with other competitive processes was performed, indicating that the proposed PFB was a promising alternative process for remediating PHE-contaminated soil [1].

Keywords Plasma • Fluidized bed • Contaminated soil • Phenanthrene • Remediation

2.1 Introduction

Due to the rapid advancements in the chemical industry, various persistent organic pollutants, such as chlorinated organics, polycyclic aromatic hydrocarbons (PAHs), and *p*-nitrophenol, have been extensively developed and accumulated in the industry sites. For instance, the concentration of the leftover PAHs in many coking industry lands in China exceeded 100 mg/kg [1]. The sites are contaminated by persistent organic pollutants (POPs) of high potential toxicity, mutagenicity, and carcinogenicity are posing greater threats to ecology, environment, and human health [2]. On the other hand, many contaminated sites still have economic value for reuse as commercial or residential areas. Therefore, remediating the soil contaminated by POPs is an essential task.

Various technologies, such as bioremediation [3], electrodynamic remediation [2], chemical remediation [4], solvent extraction [5], and thermal treatment [6],

have been proposed for remediating the soil contaminated by POPs. However, these traditional methods may not meet the demand of rapid utilization due to the long treatment period, the secondary pollution, or adding chemicals. Since the last decades, advanced oxidation progresses (AOPs), such as photochemical method [7], and Fenton progress [8] have drawn remarkable attention for remediating the soil contaminated by POPs. As one of the newly developed AOPs, non-thermal plasma is also increasing the awareness of the soil remediation for high efficiency and economic advantages. In the recent years, several studies were conducted to prove that a pulsed corona discharge plasma is capable of removing the pentachlorophenol and *p*-nitrophenol pollutants from the soil [9–12].

To date, however, there is still no report on remediating the soils contaminated by the persistent organic pollutants using the gliding arc process, a promising plasma technique. Previous studies indicated that gliding arc discharge process is very effective at many chemical processes, such as abatement of volatile organic compounds (VOCs), methane reformation, and water purification [13–16]. Furthermore, the gliding arc discharge has greater potential in an aspect of the scaling up of the application due its simple equipment, flexible and controllable operations, high non-equilibrium state, and high efficiency in organic pollutants removal [17]. The non-thermal plasma fluidized bed (PFB) combining gliding arc plasma and fluidized bed is the most notable proposal. The ideal PFB gives a lot of opportunities for varied heterogeneous processes, such as material process [18], fuel reformation [19], and plasma-catalyst reaction [20] due to excellent heat transfer and mass transfer inside a plasma reactor, and other original features, such as the microdischarge between the solid particles. It is believed that gliding arc discharge in a fluidized bed can be an alternative for remediating the soil polluted by organics.

The aim of this chapter is to investigate the potential for soil remediation through PFB. The gliding arc plasma presented in a previous study was chosen as plasma source [14]. The solid bed was separated from the discharge zone with a rotating parabolic structure to further improve the contact between discharge gas and solids, which is different from the former GAD fluidized bed that lack uniformity, dead zone, and particle attrition. PHE, a typical PAH, was used as the model pollutant. In this chapter, the performance of this novel PFB considering the input energy density, air flow rate, and soil moisture was evaluated. By means of various analysis methods, the degradation procedure was investigated and discussed. A possible pathway was proposed based on the experimental result.

2.2 Experimental Section

2.2.1 Experimental Setup

The schematic representation of the experimental setup for remediating PAH-contaminated soil is shown in Fig. 2.1. The system mainly consists of four parts: (1) the power supply, (2) the gas source, (3) the plasma reactor, and (4) an

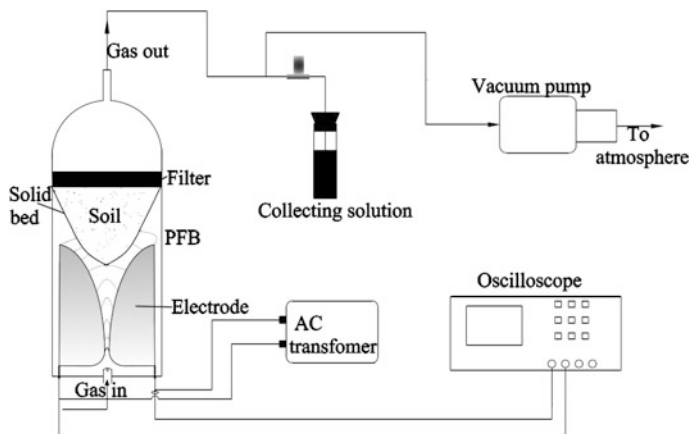


Fig. 2.1 Schematic diagram of the experimental setup

analysis section. The plasma reactor was connected to a high-voltage AC power supply with a neon transformer ranging from 7.5 to 15 kV. An oscilloscope with a high-voltage probe (Tektronix P6035) and a current probe (Tektronix TP301A) is used to measure the curves of discharge, and the input energy could be simultaneously calculated.

The plasma reactor was made up of a cylindrical quartz glass ($\Phi = 100$ mm, $H = 200$ mm). Two knife-shaped stainless steel electrodes were placed symmetrically on the opposite sides of the reactor. Above the electrodes was a stainless grid (300 mesh), which works as the solid bed supporter, and the soil samples were placed on the solid bed during the discharge. To prevent soil particles from escaping away with the carrier gas, a dust trap was also installed.

When applying a high voltage, the gas passing between the electrodes was broken down by the strong electric field, igniting the cycle of the gliding arc. Then, the length of the arc increased and moved with the gas flow until cooling down, and the new arc was generated. Carrier gas with active species (such as high energy electron, different radicals, and excited species) fluidized the soil particles and reacted with PHE in soil. The exhausted gas was directed through hexane to gather the intermediates and the residual PHE.

2.2.2 Materials and Analyses

Phenanthrene and other standards used were purchased from Sigma-Aldrich (Shanghai, China). Methanol with chromatographic purity was supplied by Honeywell (America). All other reagents were of analytical grade without any further purification.

The soil sample was prepared artificially. Clean soil was collected from the campus of Sun Yat-sen University in Guangzhou, China. The contaminated soil samples were prepared artificially. The features of the clean soil were as follows: the contents of sand, silt, clay, and organic matters were 3.0, 70.7, 26.3, and 5.44%, respectively; pH value is 6.6; and loss on ignition was 5.8%. To avoid the organic influence, the soil samples were extracted with methanol in ultrasonic bath repeatedly for five times.

The soil was polluted with PHE in the mixture solution (dichloromethane/acetone) with a ratio of 1:1 (v/v). The concentration was 200 mg/kg after the evaporation of solvent was completed.

The 15-g contaminated soil was treated every time. Prior to the treatment, the humidity of the soil was adjusted to 10%.

At the given intervals, 1 g of treated soil sample from the mixed-well solid bed (as the fly ashes were almost trapped in the solid bed by its filter) was extracted with 10 mL of methanol in ultrasonic bath repeatedly for three times. All the extracts were mixed and dehydrated by Na_2SO_4 before concentrated by rotary evaporation. Finally, the extracts were dried up by soft pure nitrogen of 99.99% purity, and the volume was re-set to 1 mL with methanol. The pre-treatment of the hexane solution was the same as the extracts.

Prior to detection, the solution was filtered through a 0.22- μm nylon filter. Quantification of PHE was performed with a high-performance liquid chromatography with a variable wavelength detector set at 254 nm (HPLC, Shimadzu, Japan). An 85:15 (v/v) mixture of methanol:water was employed as mobile phase with a flow rate of 1.0 mL/min.

The PHE efficiency (η_{PHE}) can be calculated as follows:

$$\eta_{\text{PHE}} = \frac{(c_0 - c)m - m_G}{c_0 m} \times 100\% \quad (2.1)$$

where c_0 and c represent the initial concentrations of the PHE in soil sample and treated soil, respectively, while m and m_G represent the mass of the treated soil and the mass of PHE absorbed in hexane, respectively. However, the mass of PHE absorbed in hexane was negligible, which is detailed in the following section. Therefore, the PHE degradation efficiency can be simplified as follows:

$$\eta_{\text{PHE}} = \frac{(c_0 - c)}{c_0} \times 100\% \quad (2.2)$$

The mineralization of PHE was measured by a total organic carbon analyzer (TOC; Shimadzu, Japan). Gas chromatography–mass spectrometry (GC–MS; Agilent Co., U.S.) was employed to identify the intermediates in the soil and exhausted gas. In the GC system, the GC column was a capillary column of 30 m long and 0.32 mm diameter. The temperature program was first set at 35 °C and heated up to 120 °C with a heating rate of 15 °C/min, held at 120 °C for 1 min followed by heating up to 260 °C with a heating rate of 4 °C/min, and held at a final temperature of 260 °C for 10 min.

The exhaust-gas ozone concentration is measured by the indigo dye reaction. Hydrogen peroxide is measured by a Hydrogen Peroxide ET8600 m.

The energy density is defined as follows:

$$ED = \frac{P \times T}{m} \quad (2.3)$$

where P is the power of plasma discharge (calculated by the voltage and discharge current measured by an oscilloscope) and, in this study, different input powers were offered by changing the voltage supplied. T is the treatment time (s), and m is the mass of soil treated (g).

The energy efficiency of the PFB system was defined as follows:

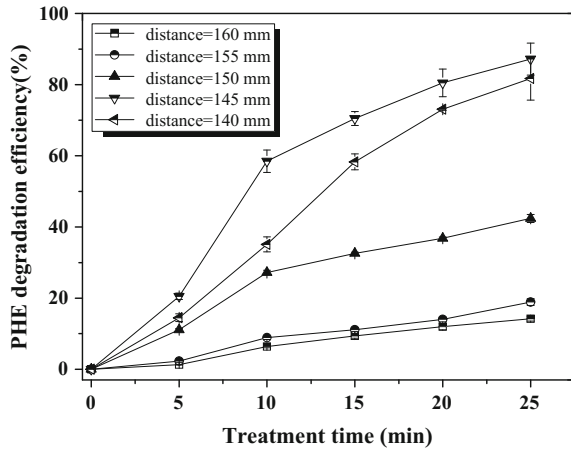
$$G = \frac{c_0 - c}{ED} \quad (2.4)$$

2.3 Results and Discussions

2.3.1 Effect of the Solid Bed Location

The potential of the PFB for PHE removal from the soil and the effect of the solid bed location, i.e., the distance between the bottom of the solid bed and the neck of the electrodes, were investigated first. Distances such as 140, 145, 150, 155, and 160 mm were taken into account. Air was used as carrier gas with a flow rate of 15 L/min. As shown in Fig. 2.2, the PFB performed very well in remediating the PHE-contaminated soil. To illustrate, after a 25-min treatment, the PHE degradation efficiency was up to 87.31% and the residual concentration was 25.36 mg/kg, while

Fig. 2.2 Effect of location of the solid bed on the PHE degradation (energy density = 3466 J/g soil, air flow rate = 20 L/min, and soil moisture = 10%)



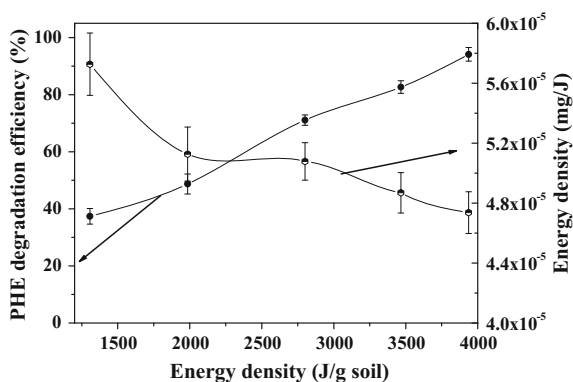
the Chinese environmental standard claims that the highest residual concentration of PHE for commercial and industrious reuse is 30 and 50 mg/kg, respectively [21], which confirmed that the proposed system had the potential for remediating the soil in such a shorter treatment period.

In Fig. 2.2, it can also be found that the distance was the smallest and the PHE degradation efficiency was the highest. This could be ascribed to the discharge characteristics between electrodes. When connected to a high voltage, energetic electrons were produced and then the ionization, excitation, and dissociation of the carrier gas were ignited to generate highly active radicals and molecules (such as OH, O₃, and H₂O₂), which might be responsible for the PHE degradation. However, the lifetime was very short for most of the active species produced, such as OH and O, leading to the inactivation of these species even before they work if the distance between the solid bed and the neck of electrodes is too large. Furthermore, there would also be discharge between the stainless solid bed and each electrode if they were close enough. Hence, a proper location might allow the full utilization of the short-lifetime active species together with a better transformation between the soil particle and the active species, whose lifetime was very short. Therefore, in the following experiment, the distance of 140 mm was chosen.

2.3.2 Effect of the Input Energy Density

Input energy density can characterize the energy demand of the proposed system for treating the contaminated soil, which is a very important parameter for the operation of plasma system. Hence, the energy density as function of the PHE degradation efficiency as well as the energy efficiency has been studied, as illustrated in Fig. 2.3. It can be seen that PHE degradation efficiency is partially depended on the energy density. When the energy density was set as 3973 J/g soil, about 94.1% of PHE was removed, while approximately 37.4, 48.7, 71.1, and 84.65% of PHE were degraded with an energy density of 1306, 1986, 2800, and 3466 J/g soil within the same time,

Fig. 2.3 Effect of the energy density on the PHE degradation efficiency and the energy efficiency (air flow rate = 20 L/min and soil moisture = 10%)



respectively. A higher energy density in the same time span means more energy input in the system, where a more number of available electrons initiate the reaction opportunities simultaneously. It is generally believed that more energetic electrons are produced with higher input energy, thus leading to the accelerated formation of active species, such as ozone, H_2O_2 , and OH [22]. These active species play important roles in PHE degradation, thus resulting in its enhancement. On the other hand, when the energy density was continuously increasing, the discharge was significantly more intense because of which more plasma channels and microdischarges occurred between the electrodes and the solid bed. In this way, the PHE-contaminated soil particle would directly collide with the energetic electrons and degrade rapidly, further increasing the performance.

Although being different from the degradation efficiency, energy efficiency of this PFB first decreased slightly as the input power increased, as shown in Fig. 2.3. Although more energy could produce more active species resulting in the enhancement of the degradation, during this procedure, more active species were inactivated before contacting with pollutants. Furthermore, some part of energy in discharge was not utilized and was consumed in other aspects. For instance, the more intense luminescence was the consumption of the energy.

2.3.3 *Effect of the Flow Rate of the Carrier Gas*

In a PFB system, the gas flow rate plays an important role. The fluidized bed requires proper velocity for better heat transfer and mass transfer. As for the gliding arc plasma, it also requires a reasonable velocity for gliding. Therefore, the PFB performance was investigated in function of air flow rate. Control experiment of PHE in soil was only performed by aeration, as illustrated in Fig. 2.4. Surprisingly, although the aeration did result in a small fraction of PHE entrained in the air flow, the residual PHE in air flow was negligible within 25-min discharge treatment. This denoted that PHE in air flow could also be degraded and that the PHE degradation by plasma be accounted for the PHE removal.

Figure 2.5 indicates that the flow rate of carrier gas has significant influence on the degradation performance. The increase in the gas flow rate was followed by the increase in the PHE degradation efficiency. This is due to a fast flow rate that was favorable for the gliding of the discharge arc, so the discharge zone was forced upstream to a larger area. In this way, more PHE-contaminated soil was immersed in a reactive zone. Meanwhile, since soil has a strong adsorption for the PHE, desorption of the PHE was very difficult; in order to remove the PHE strongly sorbed from the soil particle, better mass transfer is required. A higher flow rate improves the mass transfer between discharge and solid phases, so that PHE in the particles was more likely to react with the active species generated in discharge. However, the further increase in flow velocity may shorten the resident time of active species in the fluidized bed, which is not beneficial for fully utilizing the plasma species. Therefore, the optimal flow velocity chosen in this experiment was 20 L/min.

Fig. 2.4 Effect of air flow on the PHE removal (energy density = 3973 J/g soil and soil moisture = 10%)

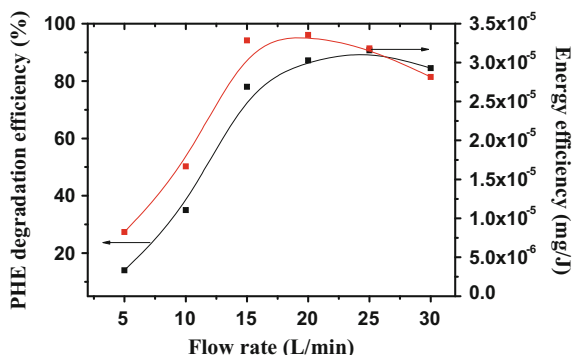
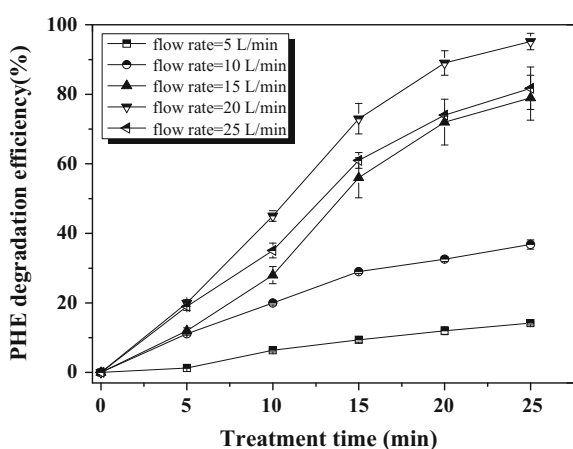


Fig. 2.5 Effect of the flow rate on the PHE degradation and the energy efficiency (energy density = 3973 J/g soil and soil moisture = 10%)

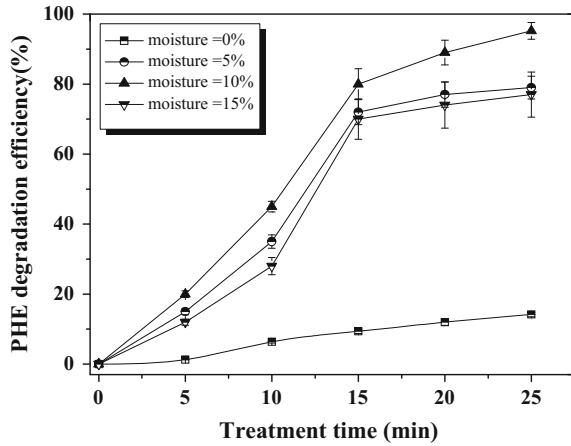


In addition, a similar energy variation of efficiency as function of the flow rate was obtained, which may be ascribed to the facts that the excellent mass transform enhanced the utilization of the active species and, in turn, the input power was fully used. With a too large flow rate, the short residence time was not beneficial for the interaction and reaction.

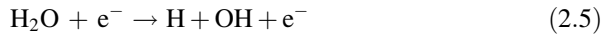
2.3.4 Effect of the Soil Moisture Content

Soil moisture content is one of the most important factors to consider in soil treatment. The experiments of PHE degradation in soil were conducted with various moisture contents. The results of the experiments are illustrated in Fig. 2.6. It was obvious that soil moisture was beneficial for PHE degradation in the proposed system. In dry soil, about 70.8% of PHE was degraded within 25 min of discharge treatment and PHE efficiency was up to 95.2% with the soil moisture content of

Fig. 2.6 Effect of soil moisture content on the PHE degradation (energy density = 3973 J/g soil and air flow rate = 20 L/min)



10% within the same discharge time; however, PNP degradation efficiency decreased up to 77.8% when the soil moisture continued to increase up to 15%. In air plasma, oxygen molecules could collide with energetic electrons generated in the discharge and then series of reactions took place. By these reactions, numerous active species, such as O_2^+ , O, and O_3 , were produced. Both energetic electrons and the produced oxygen-derived species could attack the H_2O molecules, thus leading to the following reactions [22, 23]:



Water content in soil could be evaporated into air with the dual role of the thermal effect of discharge together with the air flow to participate in the above-mentioned reactions. In addition, high-energy electrons could also react with water molecules in moist soil to form OH radicals and H_2O_2 , which have stronger oxidation potential than the oxygen-derived species. Therefore, higher PHE degradation efficiency in moist soil (moisture content, 10%) could be achieved, when compared with that in dry soil, implying that OH radical might work in PHE degradation.

Further increase in moisture content was hindered, which was unfavorable for PHE degradation, as it leads to the coalescence of soil particles and the mass transfer between discharge gas and soil particles.

2.3.5 Role of Active Species

To investigate the effect of various active species on the PHE degradation in soil, reactive species, such as H_2O_2 and O_3 , formed by the discharge were determined in the outlet gas. It was found that the concentrations of O_3 and H_2O_2 in the exhausted gas with clean soil were higher than that in polluted soil for both the reactive species. CH_2O_2 and CO_3 were up to 7.6 and 0.3 mg/L, respectively, with clean soil, while in polluted soil, CH_2O_2 and CO_3 were decreased up to 3.7 and 0.11 mg/L, respectively. The experimental results indicated that both H_2O_2 and O_3 play a significant role in the PHE degradation.

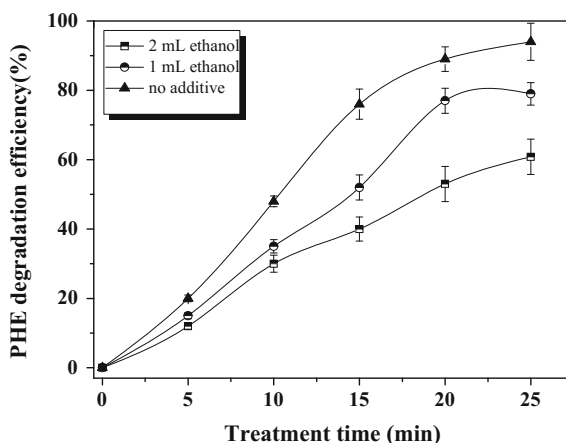
The role of OH radical was also studied by adding ethanol, an effective OH radical scavenger [24]. Different volumes of ethanol solutions were added to soil samples, as depicted in Fig. 2.7.

Obviously, the addition of radical scavenger could significantly influence PHE degradation. When ethanol was added, the reduction rate decreased rapidly and, with an increase in the amount of ethanol, the PHE degradation efficiency decreased. The experimental results indicated that OH radical played a very important role in PHE degradation and the decrease in OH radical could directly inhibit PHE degradation.

Furthermore, a previous study also demonstrated that N_2^* and N_2^+ generated in air plasma area can directly react with PAHs in gas and that energetic electrons can dissociate the C–H bonds of targets for further degradation [25].

Therefore, it is possible to clear that the reactions between PHE, oxygen-derived radicals, nitrogen-derived radicals, and electrons have an impact on PHE degradation.

Fig. 2.7 Effect of adding OH radical scavenger to PHE degradation (energy density = 3973 J/g soil, air flow rate = 20 L/min, and soil moisture = 10%)



2.3.6 TOC Analysis

Changes in the TOC value of the soil sample directly reflect the mineralization of PHE in the system. Figure 2.8 shows the TOC removal rate of the soil during the discharge. The decreased TOC value accompanies the discharge time in 40 min. It means that a fast and complete mineralization occurs under the plasma conditions. According to Fig. 2.8, in the first 10 min, the TOC removal occurred slowly and then accelerated gradually, indicating that the PHE oxidation could be divided into two stages. The PHE was first degraded into smaller organics and then mineralized to CO₂ or other gas products.

2.3.7 Proposed Pathway of the PHE Degradation

To further identify the products, GC–MS was employed. Less hazardous compound intermediates such as 9,10-phenanthrenequinone that has been detected as one of the main products in photodegradation process [26] were detected in Table 2.1.

Of note, trace product of NO₂ substituent was detected in exhausted gas, indicating that NO_x emitted by the nitrogen and oxygen discharge could also react with PHE or the unstable intermediates. However, the abovementioned intermediates represent only the portion of reaction intermediates, since some intermediates were trace products and could not be detected in the current GC–MS analysis. The degradation pathways are tentatively proposed in Fig. 2.9.

As shown in Fig. 2.9, the oxidation process was probably initiated by the attack of oxidizing species at positions 9 and 10 of the PHE to yield the theoretical product 9,10-phenanthrenequinone. Intermediate 9,10-phenanthrenequinone could be

Fig. 2.8 TOC removal efficiency as function of treatment time

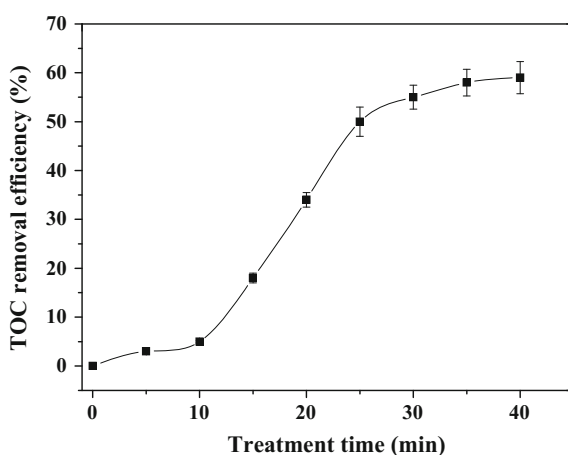
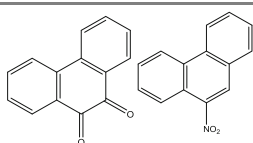
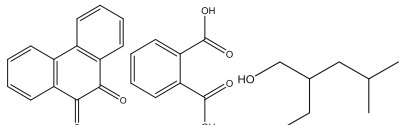
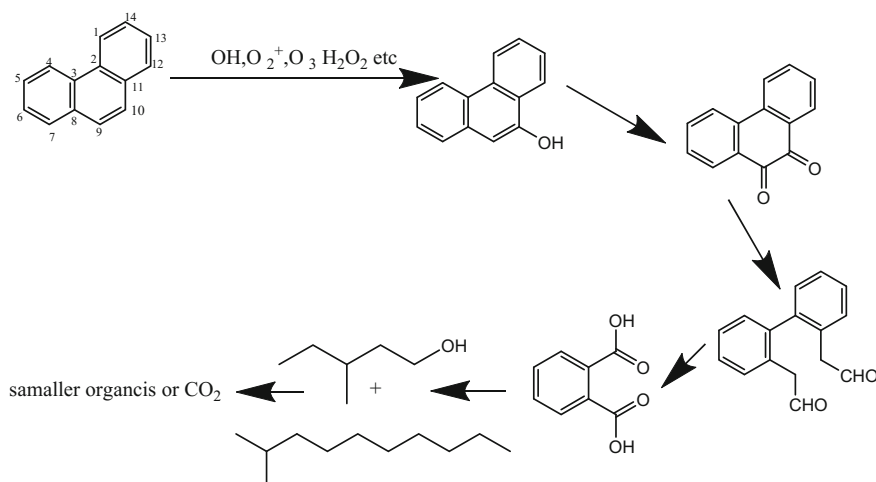


Table 2.1 Compounds identified in the products

	Products
Exhausted gas	
Treated soil	

**Fig. 2.9** Possible pathway of the degradation of phenanthrene by the PFB

cleaved and could yield phthalate acid [27]. After further cleavage over the rest benzene ring, various substances, such as esters, alkanolic acid, alkanol, and alkane, were yielded [28]. Given sufficient reaction time, all the intermediates could be completely mineralized.

A brief comparison of the PFB process with other technologies utilized for PHE degradation has been summarized in Table 2.2. It is observed that the ozone treatment showed great potential for PHE degradation due to a higher efficiency and a shorter period over other processes. However, the proposed PFB process was comparable to the previous ozone treatment, and this newly developed process showed higher efficiency in seriously polluted soil abatement. Hence, it is believed that PFB process is an alternative and efficient method to degrade PHE in soil, especially for those polluted sites that require quick remediation.

Table 2.2 Comparison between PFB process and other competitive technologies for remediating PHE-contaminated soil

Method	C ₀ (mg/kg)	Period	Capacity (g)	Degradation efficiency (%)	References
Bioremediation	200	28 day	–	100	[3]
Photodegradation/Fe (III)	100	8 h	10	100	[7]
Ultrasound/soil washing	101.04	30 min	10	70	[29]
Ozone treatment	10	20 min	10	90	[30]
PFB process	200	25 min	15	95	This study

2.4 Conclusions

In the present study, the remediation of PHE-contaminated soil by a novel plasma fluidized bed was investigated. The results showed that PHE in soil could effectively degrade after the discharge treatment. This new system promises high efficiency, for efficiency up to 95% could be attained in 25 min under the optimal conditions. The proposed reactor also has economic advantage compared with other remediation technologies with a much shorter treatment period without any other additional chemicals, except for energy input. Therefore, the plasma fluidized bed is a promising alternative technique for remediating the organics-polluted soil, especially for heavily polluted soil, to reuse.

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