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Photocatalytic degradation of phenanthrene on soil surfaces in the presence of nanometer anatase TiO₂ under UV-light

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Abstract

The effect of nanometer anatase TiO₂ was investigated on the photocatalytic degradation of phenanthrene on soil surfaces under a variety of conditions. After being spiked with phenanthrene, soil samples loaded with different amounts of TiO₂ (0 wt.%, 1 wt.%, 2 wt.%, 3 wt.%, and 4 wt.%) were exposed to UV-light irradiation for 25 hr. The results indicated that the photocatalytic degradation of phenanthrene followed the pseudo first-order kinetics. TiO₂ significantly accelerated the degradation of phenanthrene with the half-life reduced from 45.90 to 31.36 hr for TiO₂ loading of 0 wt.% and 4 wt.%, respectively. In addition, the effects of H₂O₂, light intensity and humic acid on the degradation of phenanthrene were investigated. The degradation of phenanthrene increased with the concentration of H₂O₂, light intensity and the concentration of humic acids. It has been demonstrated that the photocatalytic method in the presence of nanometer anatase TiO₂ was a very promising technology for the treatments of soil polluted with organic substances in the future.

Key words: nanometer anatase TiO₂; photocatalytic degradation; phenanthrene; soil surfaces; UV-light

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Introduction

Phenanthrene is a compound of polycyclic aromatic hydrocarbons (PAHs) consisting of two or more fused benzene rings. PAHs are hazardous materials because of their mutagenic and carcinogenic properties, and contaminate environment normally through combustion, discharge of fossil fuels, automobile emissions, and subsequent atmospheric transport and deposition (Mantis et al., 2005; Lönnermark and Blomqvist, 2006; Pekey et al., 2007; He et al., 2009; Zhao et al., 2010; Ma and Zhou, 2011; Li et al., 2011; Yan et al., 2012). Because they are hazardous to ecosystems and human beings, it is important to develop effective and economical means to eliminate them (Sabljic, 2001; Ding et al., 2008; Sun et al., 2010; Arulazhagan and Vasudevan, 2011).

Heterogeneous photocatalysis of organic pollutants using TiO₂ under UV-irradiation and/or solar light has been successfully demonstrated in various systems for remediation of polluted soil. The addition of small amounts of TiO₂ enhanced the photodegradation of *p*, *p'*-DDT on soil surfaces significantly (Zhao et al., 2004; Quan et al., 2005). Photocatalytic treatment using TiO₂ combined with solar light was very efficient in destroying pesticide Diuron in

the top 4 cm of contaminated soils (Higarashi and Jardim, 2002). The organic contaminants were destroyed in a relatively short time when the contaminated soils containing atrazine, 2-chlorophenol, and 2,7-dichlorodibenzodioxin were mixed with TiO₂ and exposed to simulated solar radiation (Pelizzetti et al., 1990, 1992).

Photocatalytic degradation might be also an efficient way to eliminate PAHs. It is extensively reported that PAHs can be photocatalytically degraded in aqueous suspensions (García-Martínez et al., 2005; Pal and Sharon, 2000; Kefi et al., 2011) by using pure solid phase catalysts such as silica, alumina, Fe₂O₃ and TiO₂ (David and Boule, 1993; Reyes et al., 1998, 2000; Wen et al., 2002, 2003; Wang et al., 2009). However, few studies investigated the photocatalytic degradation of PAHs in the presence of nanometer anatase TiO₂ on soil surfaces.

In this article, the photocatalytic degradation of phenanthrene on soil surfaces with the addition of nanometer anatase TiO₂ under UV-irradiation is studied and the possibility of heterogeneous photocatalysis using nanoparticulate anatase TiO₂ in remediation of phenanthrene contaminated soils is discussed. The influence of important factors, such as H₂O₂, the light intensity and humic acids is examined.

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1 Materials and methods

1.1 Reagents and materials

Phenanthrene was purchased from Fluka, Germany. Methanol (HPLC grade) was purchased from Shandong Yuwang Company, China. Hexane and dichloromethane (analytical grade) were purchased from Tianjin Concord Technical Company, China. NaN₃ was purchased from Zhejiang Hailan Chemical Co., Ltd., China. Nanometer anatase TiO₂ (chemical purity) was obtained from the Hangzhou Wang Jing New Material Co., Ltd., China. H₂O₂ (AR, 30% content), was purchased from Shenyang Chemistry Reagent Corporation, China. Humic acid was purchased from Tianjin Jinke Company, China.

Surface soil samples (0–10 cm) were collected from the Ecological Station of the Shenyang, Institute of Applied Ecology, Chinese Academy of Sciences. The soil samples were sterilized by soaking in NaN₃ solution for 48 hr and then dried in an oven at 105°C. After drying, the soil samples were passed through a 1 mm sieve. The sieved soil samples were then stored in a dark chamber before use. Its characteristics are as follows: the contents of sand, silt, and clay are 21.4 wt.%, 46.5 wt.%, and 32.1 wt.%, respectively; organic matter 1.78 wt.%; density 2.53 g/cm³; pH 6.8.

For the individual experiments, the soil samples were treated with a methanol solution of phenanthrene, mixed thoroughly and then air-dried to evaporate the methanol. The concentration of phenanthrene in the resulting soil samples was 40 mg/kg.

1.2 Photolysis experiments

Obtained soil samples were spiked with nanometer anatase TiO₂ powder in a similar way as the method described by Higarashi and Jardim (2002).

The photolysis experiments were conducted in a photodegradation chamber with UV lamps fixed at the top and separated by 60 mm as shown in Fig. 1. The UV lamps (Phillips ATLD 20 W, Model UVA, Phillips, Holland) had a wavelength of 253.7 nm. In all photolysis experiments, three replicates of 5.00 g of soil samples were evenly spread on Petri dishes and randomly located in the anaerobic photodegradation chamber under the UV lamps. Light proof Petri dishes also containing 5.00 g of uniformly spread soil samples were used as controls for the measurement of non-photolytic phenanthrene loss for all experimental treatments. Petri dishes containing soil samples were placed on the shelves for UV irradiation. The height of the lamps above the soil samples was 100 mm. Temperature within the chamber was held constant at 25°C using a heater and fans throughout all experiments. Soil samples were taken at 5 hr intervals from the Petri dishes.

The effect of H₂O₂ on the photocatalytic degradation was investigated by loading the desired amount of H₂O₂ to soil samples in the presence of 2 wt.% TiO₂. The number of UV lamps was adjusted to change the light intensity accordingly. The concentrations of humic acids in soil

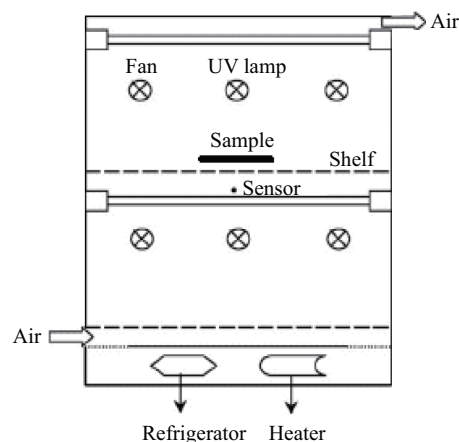


Fig. 1 Schematic experimental device for photocatalytic degradation of pyrene on soil surfaces.

samples were 0, 10, 20, 30 and 40 mg/kg, respectively.

1.3 Phenanthrene extraction procedure

Concentrations of phenanthrene in the irradiated and non-irradiated soil samples were obtained by the following method: the soil samples were transferred into 100 mL Teflon tubes and mixed with 20 mL dichloromethane. Each sample was extracted for 2 hr in an ultrasonic bath in which the water temperature was lower than 35°C. The mixtures were then centrifuged at 4000 r/min for 5 min to separate the supernatant from the soil samples. An aliquot of 0.5 mL of extraction was passed through a glass column containing 0.9 g of silica gel wetted with hexane. The extraction was eluted with 1 mL hexane:dichloromethane (1:1, V/V) four times. The elutant was completely dried under a gentle stream of nitrogen. The solid residue was re-dissolved in 1 mL of methanol for HPLC analysis.

1.4 Analytical methods

Quantification of phenanthrene in methanol solutions were conducted using an Agilent model 1200 HPLC (USA) with a variable wavelength detector set at 240 nm. The mobile phase used was methanol: water (90:10, V/V) at a flow rate of 0.7 mL/min.

The crystalline of TiO₂ is determined by X-ray diffractometer (RINT2200, Rigaku, Japan). Transmission electron microscopy (TEM) images were taken with JEM 2010 electron microscope (JEOL Company, Japan) at 200 kV to identify the size of TiO₂ particles.

2 Results and discussion

2.1 Description of nanometer anatase TiO₂ particles after heat-treatment

The heat-treated nanometer anatase TiO₂ is predominantly anatase phase (100% anatase as determined from XRD) and its particle size is about 15–25 nm (as determined from TEM) (Fig. 2).

2.2 Effect of TiO₂

The amount of photocatalyst added to the sample is a major parameter affecting the photocatalytic oxidation rate. The

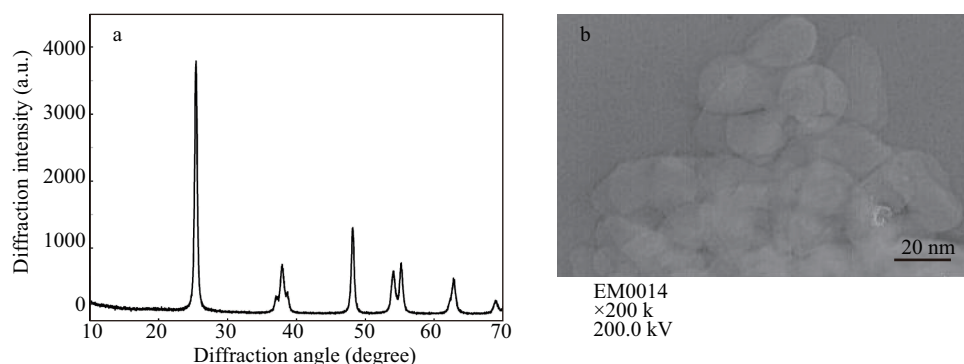


Fig. 2 XRD pattern (a) and TEM (b) of nanometer anatase TiO₂ after heat-treatment.

photocatalytic degradation of phenanthrene in soils spiked with given amounts of TiO₂ was carried out to investigate the photocatalytic activity of TiO₂ toward degrading the phenanthrene on soil surfaces. Experimental results indicated that photocatalytic degradation of phenanthrene fitted the pseudo first-order kinetics.

The pseudo first-order rate constants in the presence of 0 wt.%, 1 wt.%, 2 wt.%, 3 wt.% and 4 wt.% TiO₂ in soils are listed in Table 1. It is suggested that the photocatalytic degradation rate of phenanthrene increases with the TiO₂ additional amount.

It has been well established that the conduction band electrons (e⁻) and valence band holes (h⁺) are generated when TiO₂ is irradiated with light energy greater than its band gap energy (3.2 eV). The photogenerated electrons could reduce the organic compounds or react with electron acceptors such as O₂, forming superoxide radical anion O₂^{•-}. The photogenerated holes can oxidize the organic molecule directly. These O₂^{•-} and photogenerated holes may be responsible for the TiO₂ photocatalytic degradation of phenanthrene on soil surfaces (Dong et al., 2010).

2.3 Effect of H₂O₂

Hydrogen peroxide has been shown to accelerate the degradation of organic matters, depending on its concentration and the nature of reductants (So et al., 2002). The effect of H₂O₂ in the range of 0–40% (W/W) in the presence of 2 wt.% TiO₂ on the degradation of phenanthrene was examined over 25 hr. As shown in Fig. 3, the degradation of phenanthrene increases with increasing H₂O₂ concentration. The observed enhancement by aqueous H₂O₂ of phenanthrene degradation could be due to both the increase in soil moisture and the oxidative effect of hydroxyl radicals formed from the H₂O₂.

Frank et al. (2002) and Graebing et al. (2003) have reported that the photodegradation of organic pollution can increase with increasing soil moisture. There are

Table 1 Kinetic parameters for the photocatalytic degradation of phenanthrene on soil surfaces at different TiO₂ additional amount

TiO ₂ addition amount	k_T (hr ⁻¹)	$t_{1/2}$ (hr)	R^2
0 wt.%	0.013	52.90	0.976
1 wt.%	0.014	49.50	0.970
2 wt.%	0.015	46.20	0.966
3 wt.%	0.017	41.50	0.939
4 wt.%	0.018	37.87	0.903

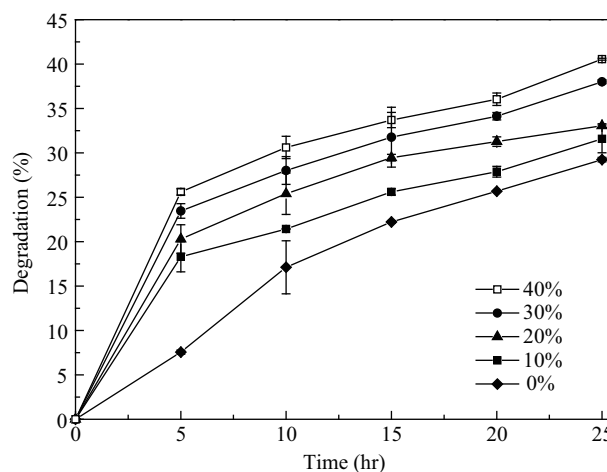


Fig. 3 Effect of H₂O₂ on phenanthrene degradation in the presence of 2 wt.% TiO₂.

three probable reasons contributing to this. Firstly, the superoxide radical anion (O₂^{•-}) can react with water to form hydroxyl radicals (OH[•]) which further enhance the degradation of phenanthrene on soil surfaces. Secondly, in the case of direct radiation effects, water greatly increases the amount of radiation absorbed in the soil. Thirdly, owing to less water available, the mobility of the chemical is affected, which results in less phenanthrene sorption and more diffusion in moist soils.

H₂O₂ could also increase the formation rate of OH[•] in two ways. Firstly, H₂O₂ could enhance the degradation by providing additional OH[•] through trapping of photogenerated electrons (Eq. (1)) (Coleman et al., 2007). Secondly, the self-decomposition by illumination would also produce OH[•] (Eq. (2)) (Lee et al., 2003; Coleman et al., 2007):



2.4 Effect of light intensity

UV light intensity is an important factor in the process of photocatalytic degradation and the effect of the UV light intensity on the degradation of phenanthrene on soil surfaces is illustrated in Fig. 4. The pseudo first-order rate constants with the increase of UV light intensity 119, 238, and 357 μW/cm² were 0.011 hr⁻¹ ($R^2 = 0.966$), 0.015 hr⁻¹ ($R^2 = 0.971$) and 0.0183 hr⁻¹ ($R^2 = 0.9428$) hr⁻¹, respectively. The results indicated that the degradation

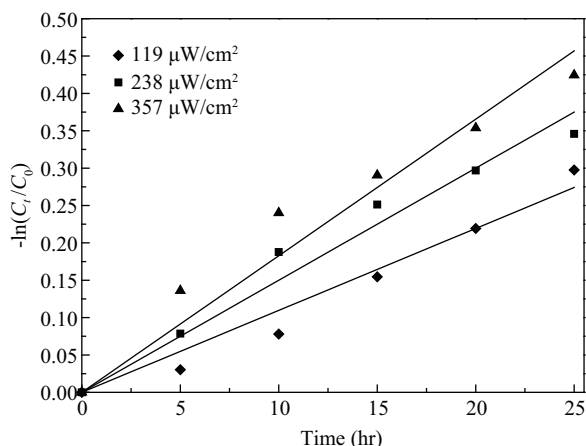


Fig. 4 Degradation kinetics of phenanthrene on soil surfaces under different light intensity.

increased with UV light intensity. This phenomenon may be explained as following: under the higher light intensity, the enhancement was considerably higher because that the electron-hole formation is predominant and, hence, electron-hole recombination is negligible. However, at lower light intensity, electron-hole pair separation competes with recombination which in turn decreases the formation of free radicals, thereby, causing less effect on the degradation of the phenanthrene on soil surfaces (Bahnemann and Boule, 1999).

2.5 Effect of humic acids

Humic acids are complex organic molecules produced by the decomposition of plant and animals remained in soil which can either enhance (Wang et al., 1995; Si et al., 2004; Chan and Chu, 2005) or inhibit photolysis (Bachman and Patterson, 1999; Gong et al., 2001). The effect of the concentration of humic acids on photocatalytic degradation of phenanthrene on soil surface was discussed when the concentration increased from 0 to 40 mg/kg. The pseudo first-order rate constants are listed in Table 2.

It can be seen in Table 2 that humic acids have accelerated effects on the photocatalytic degradation of phenanthrene. Humic acids can enhance the degradation of phenanthrene in several ways. On one hand, the absorption of light by humic acids can lead to rapid photosensitized reactions of phenanthrene via energy transfer from molecules in its triplet state. Similar result was reported by Wang et al. (1995). On the other hand, ultraviolet irradiation also induces a variety of photochemical changes in humic acids and leads to production of reactive oxygen species (e.g., singlet oxygen, peroxy radicals) (Garbin et al., 2007; Chen et al., 2008). These reactive oxygen species

Table 2 Effect of humic acid on photocatalytic degradation of phenanthrene on soil surfaces with 2 wt.% TiO₂

Humic acid (mg/kg)	k_T (hr ⁻¹)	$t_{1/2}$ (hr)	R^2
0	0.015	46.20	0.966
10	0.016	43.86	0.933
20	0.018	38.72	0.950
30	0.020	34.14	0.927
40	0.022	30.94	0.955

could oxidize the phenanthrene on soil surfaces.

3 Conclusions

A detailed feasibility study has been carried out on photocatalytic degradation of phenanthrene on soil surfaces in the presence of nanometer anatase TiO₂ powder as a photocatalyst under UV radiation. It was observed that catalyst concentration, H₂O₂, light intensity and humic acids concentration all significantly affected the photocatalytic degradation of phenanthrene on soil surfaces. The results of the study indicate that the photocatalytic degradation of phenanthrene in the presence of nanometer anatase TiO₂ on soil surfaces under UV light irradiation followed pseudo first-order kinetics.

The obtained results demonstrate that the phenanthrene on soil surfaces can be decomposed by photocatalytic degradation in the presence of nanometer anatase TiO₂. Meanwhile, some important parameters such as catalyst addition amount, the addition of H₂O₂, the concentration of humic acids and light intensity needed to be monitored in order to control the degradation. The surface soils are exposed to solar light, which consists of UV fraction. Therefore, catalytic technique in the presence of nanometer TiO₂ under UV light will be far attractive in remediation of surface soils contaminated with persistent organic pollutants.

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