

Photocatalytic degradation of pentachlorophenol in aqueous solution employing immobilized TiO₂ supported on titanium metal

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Abstract

A study on photocatalytic degradation of pentachlorophenol (PCP) in aqueous solution employing immobilized TiO₂ on titanium metal has been conducted. TiO₂ film was prepared via a deep coating in a sol–gel system of titanium (IV) bis (ethyl acetoacetato)-diisopropoxide precursor, followed by calcinations at 525 °C. Two catalysts sheets (5 cm × 5 cm, each) were put in a batch reactor containing 10 ppm PCP and irradiated by a UV black light. The occurrence of PCP degradation was indicated by decrease in pH, increase of conductivity, and formation of chloride ion. Moreover, the UV spectra observation indicate that the degradation of PCP solely by UV light (photolysis) was observed due to in part dechlorination of PCP molecules, while aromatic moiety remained. The aromatic intermediate could be further degraded in the presence of TiO₂ and UV light (photocatalysis). The determination of intermediate degradation products by HPLC revealed that oxalic acid was detected consistently.

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Keywords: PCP; Immobilized TiO₂; Photocatalytic degradation; Aromatic moiety; Chloride ion

1. Introduction

Pentachlorophenol, PCP, has been extensively used for wood preservation and biocide. This compound is carcinogenic and toxic to plants, animals, and human even at low concentrations [1]. PCP is also known as an environmental precursor for the formation of polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), which are more harmful [2].

Recent studies indicate that advance oxidation processes such as sonication, ionising radiation, combination of UV and ozone or hydrogen peroxide are able to destroy environmentally persistent pollutants, due to their ability to generate the highly oxidizing species, •OH radical. Interestingly, •OH radical is also generated at the surface of TiO₂ when being irradiated with UV light [3–8].

Titanium dioxide (TiO₂) when being irradiated with light of wavelength (λ) ≤ 415 nm produce excited-state electron and hole pairs, which are able to initiate various chemical reactions. Titanium dioxide as photocatalyst has been proposed to solve various environmental problems. It has been shown to be useful for elimination of microorganisms such as bacteria and viruses, for odour control, and degrading organic pollutants such as pesticides and phenol [9–13]. The degradation of PCP under UV light employing TiO₂ slurry to mineralize PCP producing formic and acetic acid as intermediate compounds has been examined successfully [14]. The use of slurry catalysts, however, creates a great technical problem in retrieving the catalyst from the treated water. In response to the retrieving problem many researchers have introduced an immobilized catalyst. In this paper, we report our investigation of photocatalytic degradation of PCP in aqueous solution using TiO₂ immobilized on titanium metal and under black light irradiation in a batch system.

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2. Material and methods

2.1. Materials

TiO₂ catalyst was prepared by calcinations of sol-gel film produced from titanium (IV) bis (ethyl acetoacetato)-diisopropoxide (TAA) precursor at 525 °C. Based on X-ray diffraction data, the crystal structures was determined and predominantly was found to be in the anatase form. Scanning electron microscopy data revealed that the thickness of the film was 24.7 μm, and seem to be porous. The TAA and pentachlorophenol (PCP) 86% were purchased from Aldrich. The PCP was used after purification. Other chemicals such as ethanol, isopropyl alcohol, acetonitrile, petroleum benzene (60–80 °C), acetone, phosphoric acid, nitric acid, and oxalic acid were obtained from Merck, methanol and acetonitrile were HPLC grade.

2.2. Photocatalytic reactor

Photocatalytic reactor was constructed from a Pyrex vessel (25 cm × 11 cm × 7 cm), and two catalysts sheets (5 cm × 5 cm), were placed in it. Two of 10 W UV black light lamps (TOKI, Japan), were positioned 8 cm above Pyrex vessel (Fig. 1). The intensity of UV light was measured by UV ray radiometer. At the initial experiment the intensity were 1.9, 2.3 and 1.9 mW/cm² at the left, centre and right of reactor respectively and at the last experiment the intensity were 1.4, 1.8 and 1.5 mW/cm² at the left, centre and right of reactor respectively.

2.3. Chemical analysis

Determination of PCP residue was performed by HPLC, Shimadzu model LC-9A equipped with UV detector at 215 nm, and C-18 phenomenex column. The mobile phase was 90% methanol in phosphate buffer (pH 3.4) with the flow rate 1 mL/min. On the other hand, intermediate organic acids were determined by same instrument, but the UV detector was set at 210 nm, applying shodex KC-811 column,

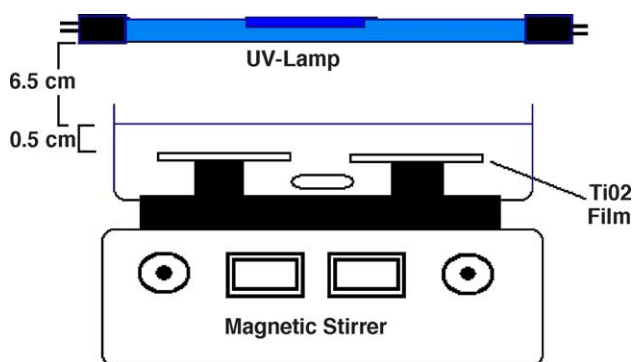


Fig. 1. Schematic of the photocatalytic reactor, comprised of a 2 × 10 W black light UV lamp (Toki, Japan), a Pyrex glass vessel equipped with magnetic bar and two catalyst sheets, and a magnetic stirrer.

and 0.005% phosphoric acids as the mobile phase with the flow rate were 1 mL/min.

The UV spectra of PCP solutions before and after irradiation were measured on Hewlett Packard Model 8453 Diode Array Spectrophotometer. Chloride ion concentrations were monitored by ferric thiocyanate methods, which was measured by UV-vis Spectrophotometer at 460 nm. Digital pH meter and conductometer were used to measure the pH and conductivity of the solution.

2.4. Methods of irradiation

Irradiation of 10 ppm PCP in aqueous solution were conducted at pH 6 for 15 min, 30 min, 1, 2, 4, 6, 8, 10, 13, and 16 h. During irradiation the solution of PCP was homogenized with magnetic stirrer.

3. Results and discussions

3.1. The pH, conductivity, and chloride ion concentration of the treated water

It is widely accepted that photocatalytic oxidation of organic compounds may produce carbon dioxide, mineral acid, and water. It can be inferred that pH will decrease during the course of reaction while chloride ion will evolve. It is apparent that before complete mineralization occurs, some intermediate may be produced.

The change of pH and conductivity during the photocatalytic of PCP in aqueous solution are displayed in Figs. 2 and 3. Fig. 2 shows that under the dark experiment, pH and conductivity of the solution was not changed during 16 h observation, even in the presence of TiO₂. When UV black light without TiO₂ is applied, the pH of the solution was decreased as the irradiation time increased, due to photolysis.

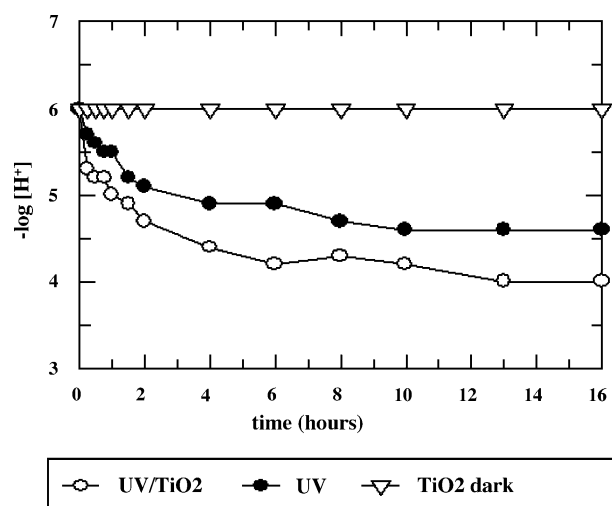


Fig. 2. The change in pH value during the photocatalysis, photolysis, and control experiment of 10 ppm pentachlorophenol.

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