

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Synthetic wastewater

Phenol (99.9%) used as a model of wastewater pollutant for treatment in this work was purchased from Polskie Odczynniki Chemiczne S.A.. The phenol solution was used as received without any purification. The test solution was made from demineralized water (conductivity = 2 microziemen).

#### 3.2 Analytical instruments

The instrument used to characterize the characteristics of fibers of HEPA filter and the deposition of TiO<sub>2</sub> nanoparticles formed on the surface of HEPA filter was Scanning Electron Microscopy (SEM) (JEOL, JSM-5800 LV). The instrument used to measure the concentration of aqueous phenol was the High Performance Liquid Chromatography (Shimadzu column, Class VP), while the total organic carbon (TOC) in the phenol solution was measured using total organic carbon analyzer (Shimadzu TOC V<sub>CPH</sub>, 2005). Before the residual phenol solution was analyzed, it was filtered with 0.45 μm Millipore filter membrane.

### *Scanning Electron Microscopy (SEM)*

The HEPA filter fibers and the deposition of TiO<sub>2</sub> cake formed on the surface of HEPA filter were observed using SEM images. SEM specimens were prepared by cutting out a small piece of HEPA filter or TiO<sub>2</sub> formed on the surface of HEPA filter, and then directly placing the piece onto a conductive gold coated microscope grid. The specimens were loaded into a sample chamber, and observations were immediately started using image catcher scanner for taking the photos. A photo of the Scanning Electron Microscopy (SEM) machine is shown in Fig. 3.1.

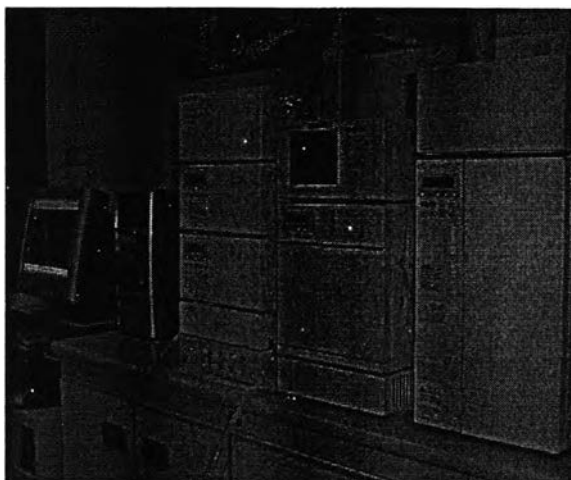


**Figure 3.1.** Scanning Electron Microscopy (SEM) (JEOL, JSM-5800 LV).

### *High Performance Liquid Chromatography (HPLC)*

In the experiments involving the present rotary drum filtering reactor, the residual concentration of phenol in the supernatant liquid was analyzed by the above-mentioned HPLC with 25% acetonitrile: 75% water as the mobile phase and a UV-Vis detector using a wavelength of 254 nm. The column temperature was 30 °C. The

flow rate of the mobile phase was  $0.6 \text{ cm}^3 \text{ min}^{-1}$  and the injection volume was equal to  $10 \text{ }\mu\text{L}$ . Fig. 3.2 shows the HPLC used in this work.



**Figure 3.2.** High Performance Liquid Chromatography (HPLC).

*TOC (Total Organic Carbon) analyzer*

The total photomineralization of phenol could be obtained as indicated by the total organic carbon (TOC) disappearance. Fig. 3.3 shows the TOC analyzer used in this work.



**Figure 3.3.** TOC analyzer.

### 3.3 Photocatalysts

The TiO<sub>2</sub> powders used as photocatalysts in this work were provided by Prof. Yasushige Mori of Doshisha University, Kyoto, Japan and Nonami Science (Thailand) Co., Ltd. Their properties given were shown in Table 1.1.

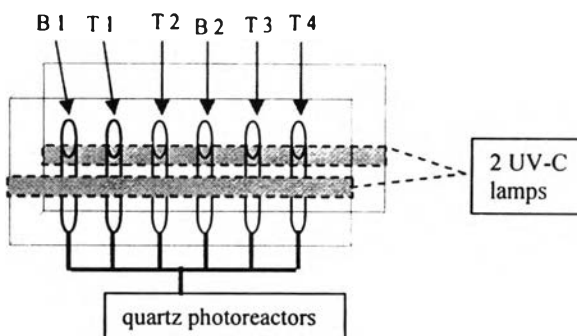
### 3.4 Photocatalytic reactor

#### 3.4.1 Shaker

In order to find out the initial rate of phenol decomposition by the titania nanoparticles, a preliminary study was first carried out. The experimental unit for the photocatalytic reaction using titania is illustrated in Fig. 3.4. First, approximately 2 mg of the titania sample of interest was suspended in 20 cm<sup>3</sup> of 10 ppm phenol solution. Then the samples were irradiated with two 30-Watt UV-C lamps (Philips G30T8) while they were mechanically shaken to ensure good dispersion of the titania in each sample. All the experiments were carried out at room temperature. For comparative purposes, the titania was also suspended in a phenol solution under shaking condition but without UV-C light irradiation. Details of the experimental results on phenol adsorption and photocatalytic decomposition were as follows:

Type 1. Tests for the adsorption efficiency of titania in the phenol solution without exposure to light for either 10 or 60 min.

Type 2. Tests for the combined efficiency of photocatalytic decomposition plus adsorption for titania in the phenol solution after exposure to UV light for either 10 or 60 min.



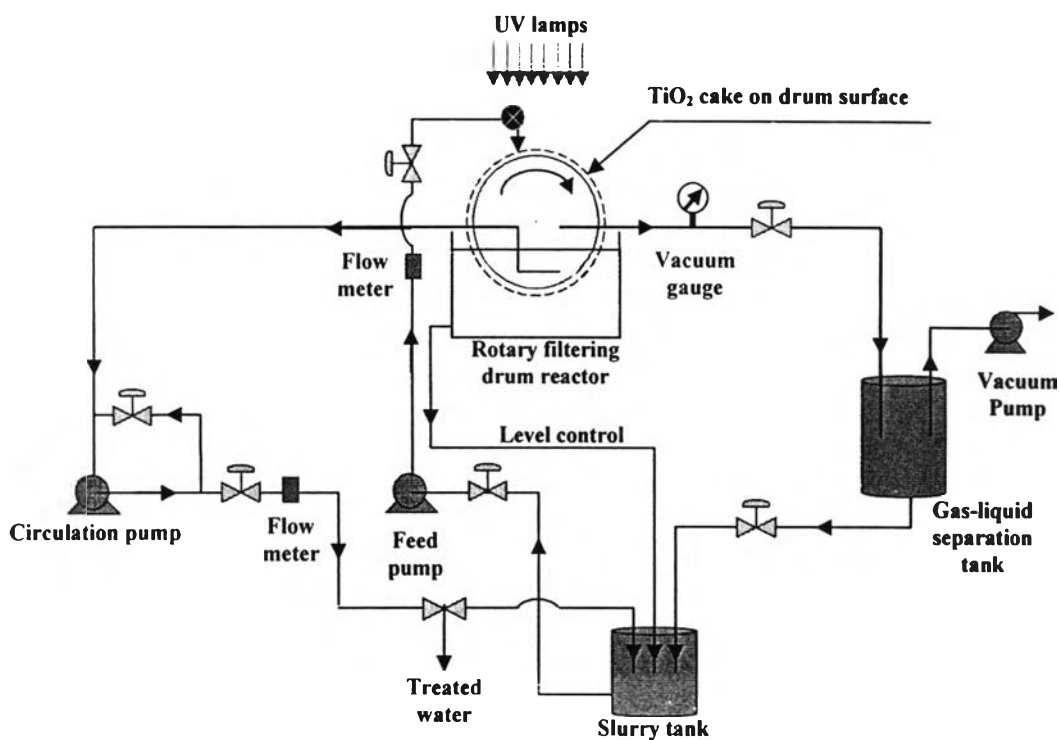
**Figure 3.4.** Arrangement of the batch reactors and lamps in the shaker (T1, T2, T3, and T4 are test tubes with samples of organic solution and titania; B1 and B2 are test tubes with samples of only organic solution).

Prior to each residual concentration analysis,  $\text{TiO}_2$  powder suspended in the phenol solution was separated out as much as possible by centrifuging the suspension at 3800 rpm for 15 min. Then the residual concentration of phenol in the supernatant liquid was analyzed with the HPLC. The mobile phase composition was methanol/water at a ratio of 40/60. The UV-detection was operated at 270 nm. The column temperature was  $30\text{ }^\circ\text{C}$ . The flow rate of the mobile phase was  $0.6\text{ cm}^3\text{ min}^{-1}$  and the injected sample volume was equal to  $10\text{ }\mu\text{L}$ . The TOC in the phenol solution was analyzed with a TOC analyzer.

### 3.4.2 Rotary drum filtering reactor

A simple rotary drum filtering reactor (see Fig. 3.5) was proposed and fabricated to solve the problem of  $\text{TiO}_2$  loss during elutriation and catalyst recovery. The  $\text{TiO}_2$  filter cake formed on the filter mat covering the outer surface of the rotary drum was simultaneously used to decompose phenol. The simple mechanism for forming and

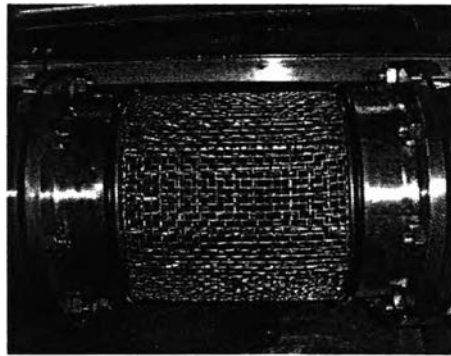
holding onto the cake was to reduce the internal drum pressure in order to suck in the filtrate continuously. Photocatalytic decomposition therefore occurred as the thin water film on the drum's outer surface contacted the UV light and ambient oxygen.



**Figure 3.5.** A schematic diagram of the setup of TiO<sub>2</sub> cake-covered rotary drum filtering reactor.

This rotary drum filtering reactor consisted of four 15-Watt UV-C lamps (Philips G15T8) and the maximum intensity of phenol's spectrum was at 253.7 nm as the light sources and a cylindrical drum frame. The drum was made of stainless steel with 14.5 cm of outer diameter and 14.5 cm long (see Fig. 3.6). A HEPA filter (pore size of 0.3  $\mu\text{m}$ ) wrapped around the cylindrical drum frame was supported by a metal mesh. Its role in this reactor system was to trap and stop the titanium dioxide nanoparticles from escaping during the cake formation stage. In addition, a

box shaped stainless steel was covered with a reflection sheet for the photocatalytic reaction. The drum could be rotated around the horizontal axis at variable speed and was immersed in a tank containing the test solution to be filtered and treated. The entire photocatalytic reaction occurred at room temperature. A rotary drum filtering reactor was used to investigate the performance of  $\text{TiO}_2$  cake in degrading phenol. An experimental set-up of rotary drum filtering reactor was shown in Fig. 3.7.



**Figure 3.6.** A photo of the bare rotary drum filtering reactor.



**Figure 3.7.** A photo of the experimental set-up of rotary drum filtering reactor.

### 3.5 Experimental procedure

The whole experimental run was separated into 3 parts. It could be broken down onto the following stages:

#### I) Cake formation

The titania cake was generated by dispersing titania powder into 25 dm<sup>3</sup> of demineralized water and then this suspension was to the outer surface of the rotary drum which was covered by HEPA filter and reduced the internal drum pressure immediately by adjustment the vacuum degree before forming it as the cake layer on HEPA filter. In this study, a TiO<sub>2</sub> cake thickness of 200 μm was calculated and the surface of HEPA filter.

#### II) Photocatalytic reaction

The operation started by mixing 25 dm<sup>3</sup> of phenol solution a residual concentration of 25 ppm after 5 minutes and then this solution was circulated in the system by “spraying” the solution to the outer surface of rotary drum with a flow rate of 4.5 dm<sup>3</sup> min<sup>-1</sup> for 10 minutes in order to ensure well-mixing. The photocatalytic decomposition occurred when the UV lamps were turned on and we set this time as the zero time reading. The samples (20 cm<sup>3</sup>) were collected before and at regular intervals during the irradiation. The reaction was followed up for 6 hours.

#### III) Titanium dioxide activation

Before starting the following batch of experiment, we must activate the TiO<sub>2</sub> cake with UV irradiation for 90 minutes in order to regenerate the photocatalyst.