CHAPTER III
EXPERIMENTAL

3.1 Materials

The following chemicals were used for the preparation of titania nanotubes: commercial TiO_2 nano-powder (99.7% anatase, Sigma-Aldrich), NaOH pellets (98%, Ajax Finechem), and HCl (37% aqueous solution, LAB SCAN Analytical Sciences). For photocatalyst activity study, 4-nitrophenol (99%, Sigma-Aldrich) was selected as substrate and H_2O_2 (30%, Ajax Finechem) was used to provide hydroxy radical in the system.

3.2 Equipments

3.2.1 Milestone ETHOS SEL Microwave laboratory systems

The reaction was conducted using microwave laboratory system. Samples were heated in a Teflon-vessel sealed with a Teflon cap, using time-to-temperature program. The power of microwave irradiation during heating period was utilized at 800 W maximum and maintaining target temperature at 500 W maximum.

3.2.2 Hitachi FE-SEM S4800 /Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray Spectroscopy (EDX)

FE-SEM & EDX was employed to observe morphology of presursor/products and to determine the sodium ion content in the prepared samples.

3.2.3 JEOLJEM-2100/Transmission electron microscope (TEM)

TEM images was utilized to provide further exploration in morphology and structure, including inner/outer diameters and length of the nanotubes.

3.2.4 Rigaku DMAX 2200 HV/X-ray diffractrometer (XRD)

For crystallinity and phase formation, XRD with Cu K_α emission line (0.154 nm) was utilized to find the phase changing of the titania precursor at given reaction time and temperature.

3.2.5 Quantachrome Autosorb-1/Surface area analyzer (SAA)

SAA with Brunauer-Emmett-Teller equation provided specific surface area, pore volume, and pore diameter of the samples.

3.2.6 UV-Visible Spectrometer/Shimadzu UV 1800

The concentration of 4-nitrophenol was analysed by UV-vis spectrometer at a wavelength of 317 nm.

3.2.7 Photocatalyst reactor system

Study of photocatalytic activity of the as synthesized titania nanotubes followed the procedure described elsewhere (Phonthammachai *et al.*, 2005). The batch reactor itself consists of 250 mL glass container with 2 jacks for cooling water in and out, 100W Hg Philip UV Lamp as a light source, magnetic stirrer and cooler to keep the reactor at room temperature (Figure 3.1).

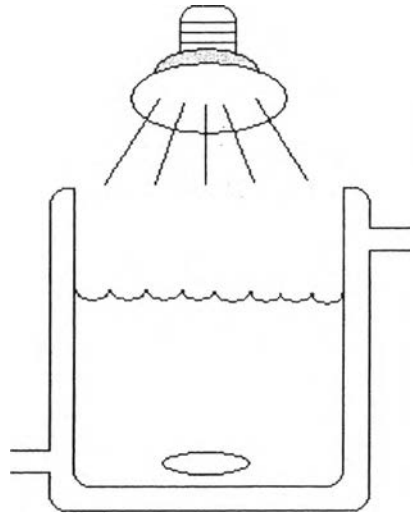

Figure 3.1 Illustration of the studied photocatalst system

3.3 Methodology

3.3.1 Preparation of Titania Nanotubes

Titinia nanotubes (TNTs) were prepared by dispersing 0.5g of 99.7% anatase nanoparticle titanium oxide in 50 mL of 10 M NaOH *via* microwave heating method (Figure 3.2). The mixture was stirred for 30 min before transferring into a Teflon vessel autoclave equipped in a microwave reactor. The experiment was conducted under microwave irradiation for various reaction times (30, 60, 90, and 120 min) and various temperatures (120°, 150°, and 180 °C). Afterwards, the obtained powders were filtered and washed with distill water until pH of the solution reached around 7. Removal of Na content was done by dispersing the obtained powder in 250 mL of 0.1 M HCl (Lee *et al.*, 2010) and shaking for overnight. The soaked powders were separated by filtration and washed with distill water until neutral. Finally, the washed solid was dried under vacuum.

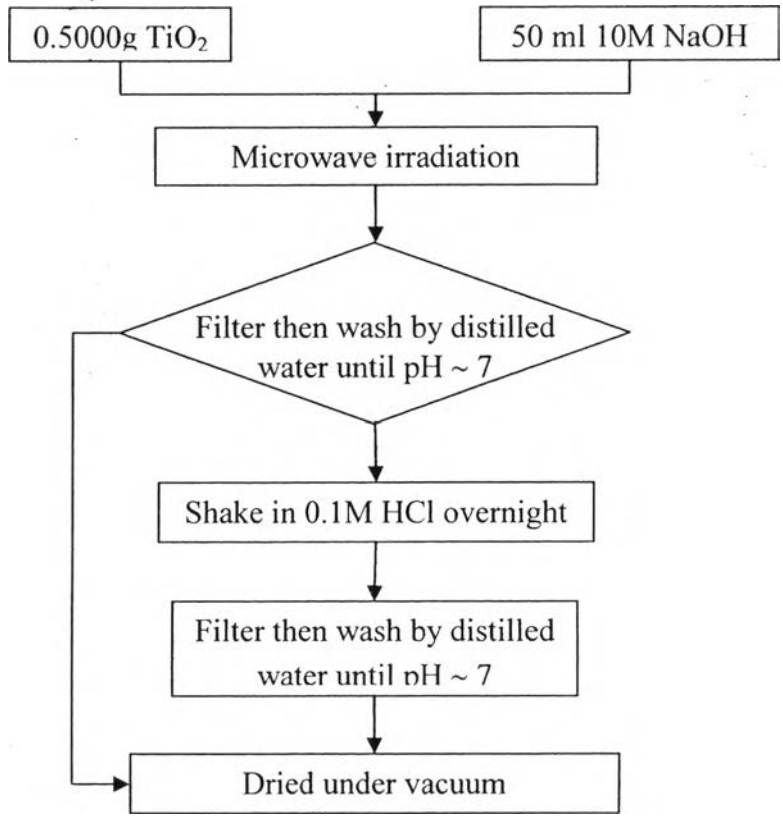

Figure 3.2 Flowchart of the preparation pathway

3.3.2 Photocatalyst Decomposition of 4-Nitrophenol

According to the literature (Phonthammachai *et al.*, 2005), 0.2 g of catalyst was mixed with 240 mL of 40 ppm 4-nitrophenol solution and 0.26 mL of 30% H_2O_2. The mixture was then vigorously stirred in dark for an hour to let 4-nitrophenol be adsorbed on the surface of the ctalyst. Before switching on the UV-lamp, 10 mL of the solution was taken as the starting concentration. Then 10 mL of the solution was taken every 30 min during the irradiation until the illuminating time reached 4 h. All sampling solutions were analyzed the 4-nitrophenol using UV-vis spectrometer.