



CHAPTER III EXPERIMENTAL

3.1 Materials

Titanium (IV) oxide nano powder (99.7% anatase) and chromium (III) chloride hexahydrate (96%) were purchased from Sigma-Aldrich. Sodium hydroxide (99%) of analytical reagent grade was purchased from RCI Labscan. Hydrochloric acid (37% aqueous solution) was purchased from LAB SCAN Analytical Sciences. Iron (III) chloride hexahydrate (99%) was purchased from Merck Chemicals. Distilled water was used throughout the experiments. For photocatalytic activity study, methyl orange as substrate was purchased from Carlo Erba Reagents.

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 the target temperature at 500 W maximum.

3.2.2 Hitachi FE-SEM S4800 /Scanning Electron Microscope (SEM)

The morphology of samples was characterized using scanning electron microscope.

3.2.3 JEOLJEM-2100/Transmission Electron Microscope (TEM)

TEM images were utilized to provide further exploration in morphology and structure, including dimension of samples.

3.2.4 Rigaku DMAX 2200 HV/X-Ray Diffractometer (XRD)

The crystallinity and phase formation of metal doped TNTs were characterized by X-ray diffractometer with Cu K_α emission line ($\lambda=0.154$ nm) over a 2θ range of 20–80° at a voltage 40 kV and a current of 40 mA.

3.2.5 Quantachrome Autosorb-1/Surface Area Analyzer (SAA)

Specific surface area, pore volume and pore diameter of the samples were determined with nitrogen adsorption-desorption by the Brunauer-Emmett-Teller (BET) method on Quantachrome Autosorb-1.

3.2.6 AXIOS PW 4400/X-Ray Fluorescence Spectrophotometer (XRF)

The X-ray fluorescence spectrophotometer (XRF) was employed to observe the element contents in samples.

3.2.7 Shimadzu UV 1800/UV-Visible Spectrometer

All sampling solutions were analyzed the methyl orange content using a UV-vis spectrometer at 464 nm.

3.2.8 Shimadzu UV 2500/UV-Visible Spectrometer

Diffuse reflectance spectra (DRS) in the 200–900 nm were measured using UV-vis spectrophotometer, and reflectance spectra were referenced to BaSO₄.

3.3 Methodology

3.3.1 Synthesis of Titania Nanotubes (TNTs)

TNTs were produced using microwave irradiation similar to that described by Wongtaewan in 2011. As summarized in Figure 3.1, TNTs were prepared by dispersing titanium dioxide in NaOH via microwave heating method. The reaction was conducted at 150 °C for 90 min using microwave laboratory system (Milestone ETHOS SEL). The obtained powders were filtered and washed with distilled water until pH of the solution reached around 7. Removal of Na content was carried out by dispersing the obtained powders in HCl and shaking for overnight. The soaked powders were separated by filtration and washed with distilled water until neutral. Then, the washed solid was dried under vacuum. After the prepared material was calcined at 500 °C for 2 h, TNTs were obtained.

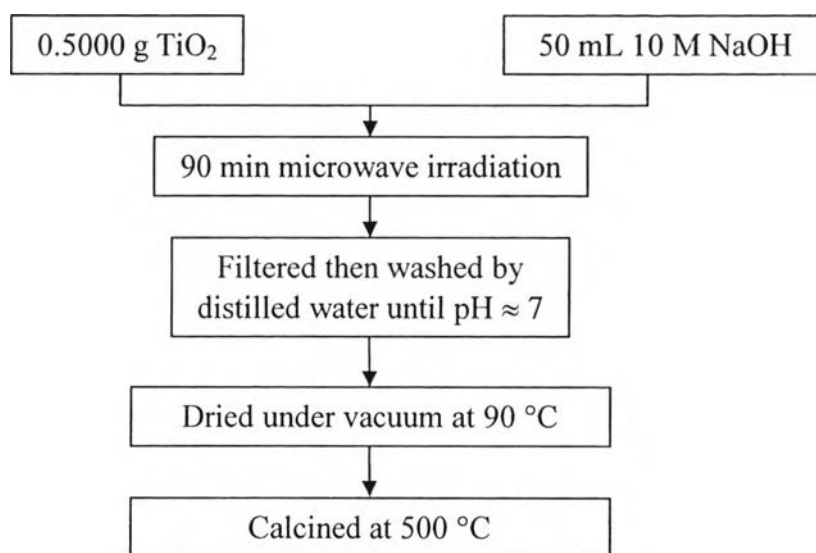


Figure 3.1 Flow diagram of TNTs' synthesis.

3.3.2 Synthesis of Metal Doped Titania Nanotubes (TNTs)

TNTs was stirred in aqueous solution containing chromium (III) chloride hexahydrate or iron (III) chloride hexahydrate for 30 min. The solution was transferred into autoclave before sealing. The sample was maintained at 90 °C for 2 h. Subsequently, the mixtures were centrifuged, washed, and dried at 60 °C to obtain metal-doped TNTs. Then the materials were calcined at different temperatures (300 °C, 500 °C, 600 °C and 700 °C) for 2 h. The process was summarized in Figure 3.2.

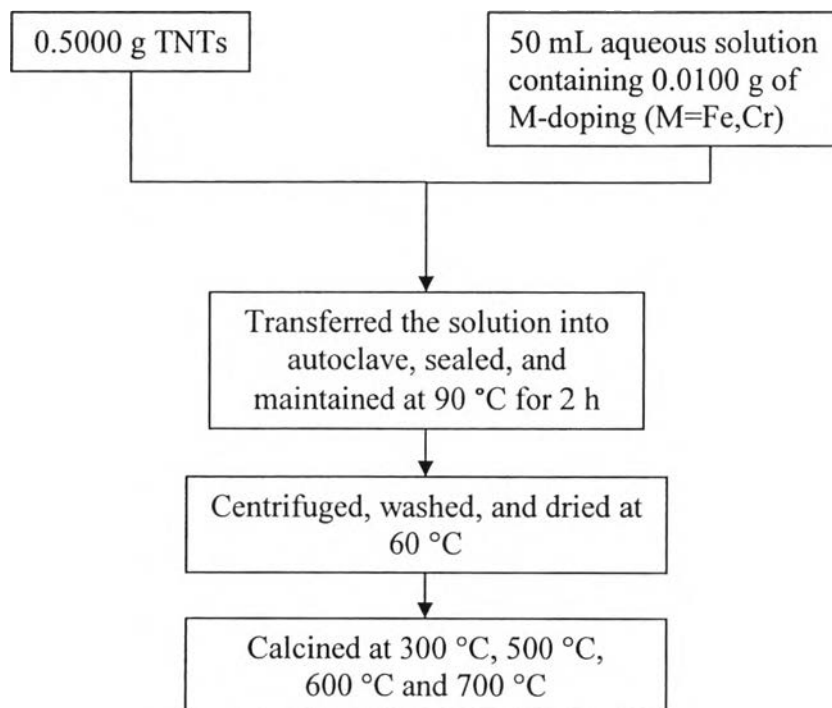


Figure 3.2 Flow diagram showing synthesis of M-doped TNTs.

3.4 Photocatalytic Activity of Methyl Orange

The 0.2 g of catalyst was mixed with 200 mL of 20 ppm methyl orange solution. The mixture was then vigorously stirred in dark for an hour to let methyl orange be adsorbed on the surface of the catalyst. Before switching on the UV-lamp (100 W Hg Philip UV lamp), 10 mL of the solution was taken as the starting concentration. After irradiation, 10 mL of the solution was taken every 1 h during the irradiation until the illuminating time reached 8 h at room temperature. All sampling solutions were analyzed the methyl orange content using a UV-vis spectrometer (Shimadzu UV 1800) at 464 nm.