Chapter III

Methodology

The experiment of this research could be divided into 3 main parts.

Part 1: Designing and setting up fixed bed photocatalytic reactor
Part 2: Preparation of TiO2 thin films on stainless steel plates
Part 3: Investigation of the operational parameters that influence the photocatalytic-reduction efficiency of chromium (VI) using FBPR

3.1 Designing and setting up fixed bed photocatalytic reactor

3.1.1 Design criteria

The reactor configuration was planned to be a rectangular aluminum box with hyperbolic roofs for light concentration with UV lamps within reactor. This reactor was designed for continuous flow which be controlled by pump and valves for control water level and feed flow rate. See Figure 3.1 and 3.2 below. The criteria for this reactor design were as follow:

- the volume capacity of reactor is at least 5 liters
- minimum ability for remove chromium (VI) 5 liters of 50 ppm
- continuous mode reactor
- available equipment:
  - length of UV-lamp is 60 cm
  - flow rate of pump that can be adjusted is 20 - 140 mL/s

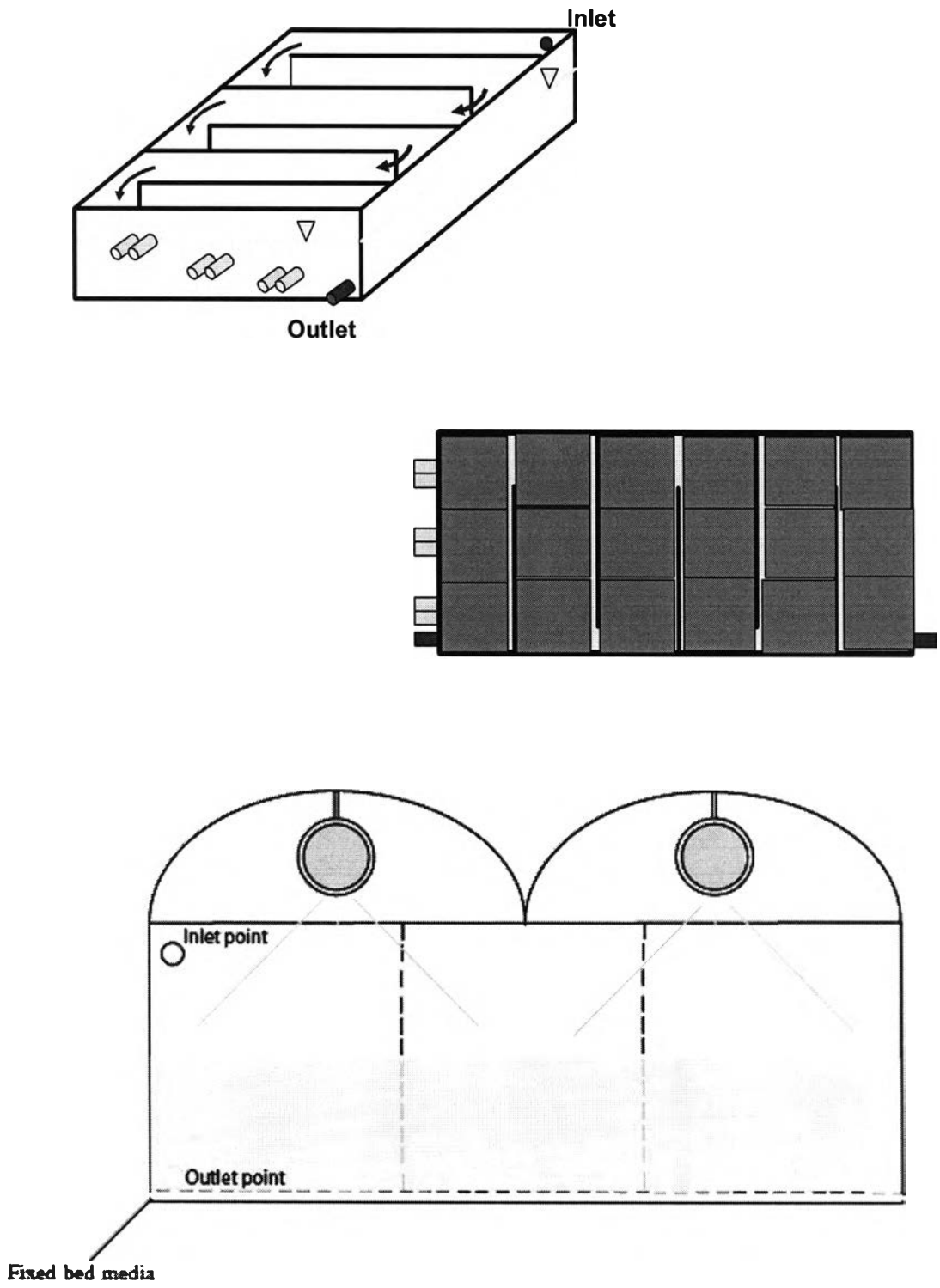

Figure 3.1 Components of fixed bed photocatalytic reactor

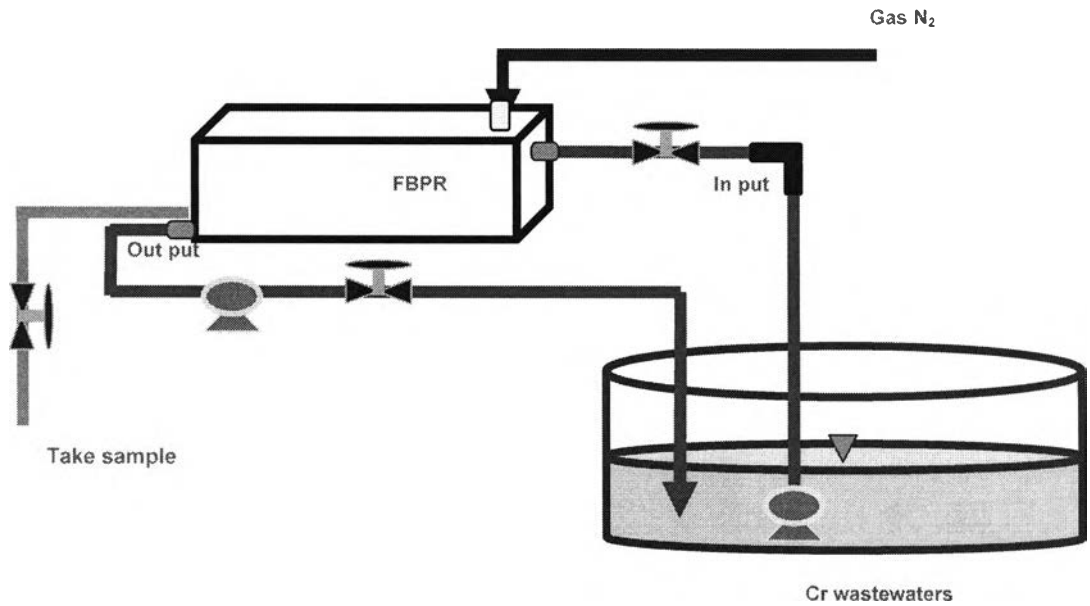

Figure 3.2 Schematic of wastewaters treatment using fixed bed photocatalytic reactor

3.2 Preparation of TiO_2 thin films on stainless steel plates

3.2.1 Preparation of coated solutions on stainless steel media

Stainless steel media (type of 316L) was washed by 2% HF for 5 min, and then dipped in a mixed solution of water and anhydrous ethanol, and dried at room temperature (Liqiang et al., 2003).

In the preparation of coated solutions, titanium (IV) butoxide ($Ti(OC_4H_9)_4$) was used as the source material for Ti. The solvent is ethanol. HCl and ethanol were mixed together and used as the acidic catalyst for the hydrolysis of titanium (IV) butoxide. The acidic catalyst was added drop wise to the titanium (IV) butoxide-ethanol solution under vigorous stirring at room temperature. After that, acetylacetone that used as the additive was added to the mixture of coated solutions and vigorous stirring was continued for 1 hr. The optimum ratio of mixture of coated solutions is titanium (IV) butoxide: ethanol: HCl: acetylacetone of 1: 30: 0.5: 1 by volume

(Kajitvitchyanukul et al., 2005). Finally, it got coated solutions that are yellow and transparent.

3.2.2 Immobilizing of TiO_2 thin films

TiO_2 thin films were immobilized on the media by sol-gel technique. Stainless steel plates (width 10.6 cm, length 10.8 cm and thickness 0.1 cm) were used as media. The coatings were carried out by dipping stainless steel plates into the coated solutions and then withdraw them at a constant velocity of 45 cm/min. After that, calcined the stainless steel plates that be coated with gel film at 500°C for 30 minutes in an electric furnace (Kajitvichyanukul et al., 2005). Then, coating it again until got 3 layers of thin films.

3.3 Investigation of the operational parameters that influence the photocatalytic-reduction efficiency of chromium (VI) using fixed bed photocatalytic reactor

Synthetic wastewaters

Chromium (VI) wastewaters were represented by chromate solutions. Chromate solutions (Use K_2CrO_4) were prepared in distilled water and stored as pollutant solutions to be treated and adjust the pH of solution to 3 (Watcharenwong, 2003) with sulfuric acid and sodium hydroxide.

Experiments in this part used the synthetic wastewaters feeding to the reactor with variation of operational parameters. Studied parameters consisted of:

- pH of wastewaters
- Flow rate
- Water level of wastewaters
- TiO_2 coating surface area
- Initial concentration

3.3.1 Investigation of initial pH of wastewaters that influence on the photocatalytic-reduction efficiency of chromium (VI)

Synthetic chromium (VI) wastewaters were prepared by contain 25 ppm. TiO_2 thin films plates prepared from Part 3.2 were placed into the reactor and then turned on inlet pump to feed synthetic wastewaters into the reactor until it got the required water level. Then, adjusted feed flow rate that required and turned on outlet pump to keep the water level of wastewaters. Before the irradiation, purged with N_2, took sample for analysis and kept the wastewaters in the dark with the TiO_2 thin films plates for 15 minutes to allow for the adsorption process. At that moment, the lamps were turned on. Samples were took at various time and stored for analysis. Experimental chart is provided in Figure 3.3.

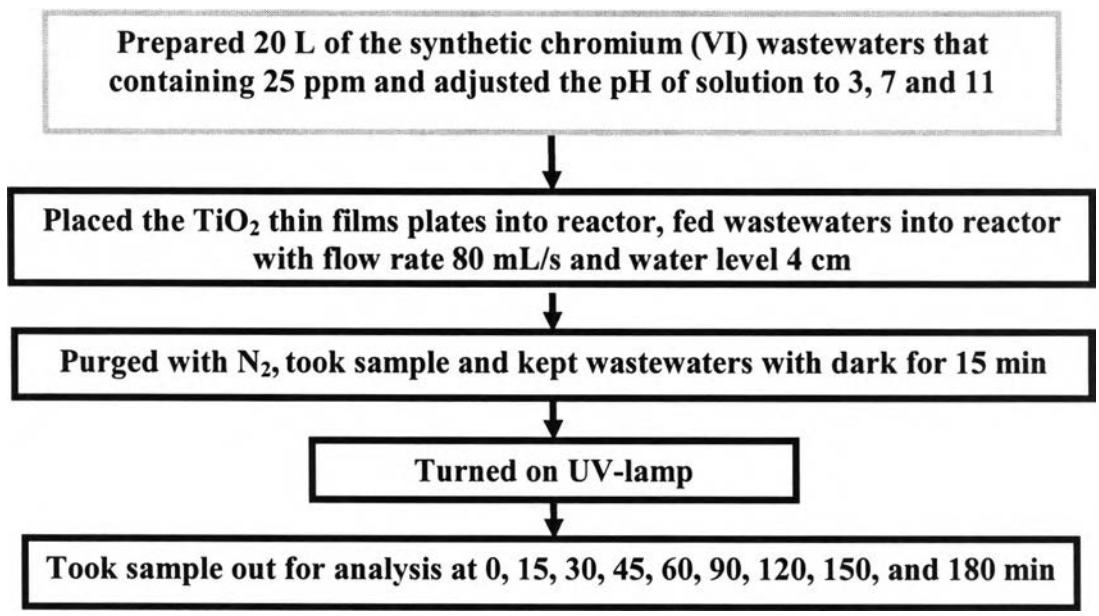

Figure 3.3 Flow chart of investigation effect of initial pH of wastewaters

3.3.2 Investigation of flow rate that influence on the photocatalytic-reduction efficiency of chromium (VI)

Prepared synthetic chromium (VI) wastewaters by contain 25 ppm. TiO_2 thin films plates prepared from Part 3.2 were placed into the reactor and then turned on inlet pump to feed synthetic wastewaters into the reactor until it got the required water

level. Then, varied feed flow rate that required and turned on outlet pump to keep the water level of wastewaters. Before the irradiation, purged with N_2, took sample for analysis and kept the wastewaters in the dark with the TiO_2 thin films plates for 15 minutes to allow for the adsorption process. At that moment, the lamps were turned on. Samples were took at various time and stored for analysis. Step chart is provided in Figure 3.4.

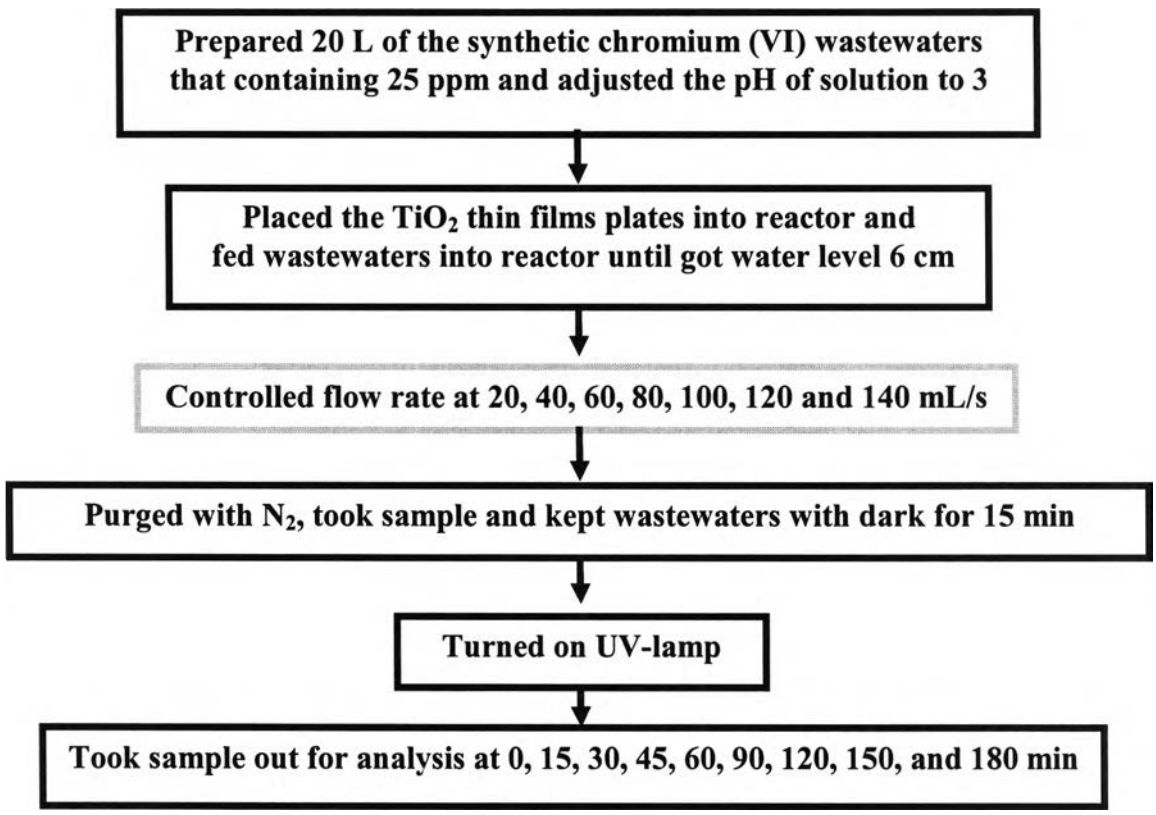

Figure 3.4 Flow chart of investigation effect of flow rate of wastewaters

3.3.3 Investigation of water level that influence on the photocatalytic-reduction efficiency of chromium (VI)

Prepared synthetic chromium (VI) wastewaters by contain 25 ppm. TiO_2 thin films plates prepared from Part 3.2 were placed into the reactor and then turned on inlet pump to feed synthetic wastewaters into the reactor until it got the required water level that want to vary. Then, adjusted feed flow rate that required and turned on

outlet pump to keep the water level of wastewaters. Before the irradiation, purged with N_2, took sample for analysis and kept the wastewaters in the dark with the TiO_2 thin films plates for 15 minutes to allow for the adsorption process. At that moment, the lamps were turned on. Samples were taken at various time and stored for analysis. Experimental chart is provided in Figure 3.5.

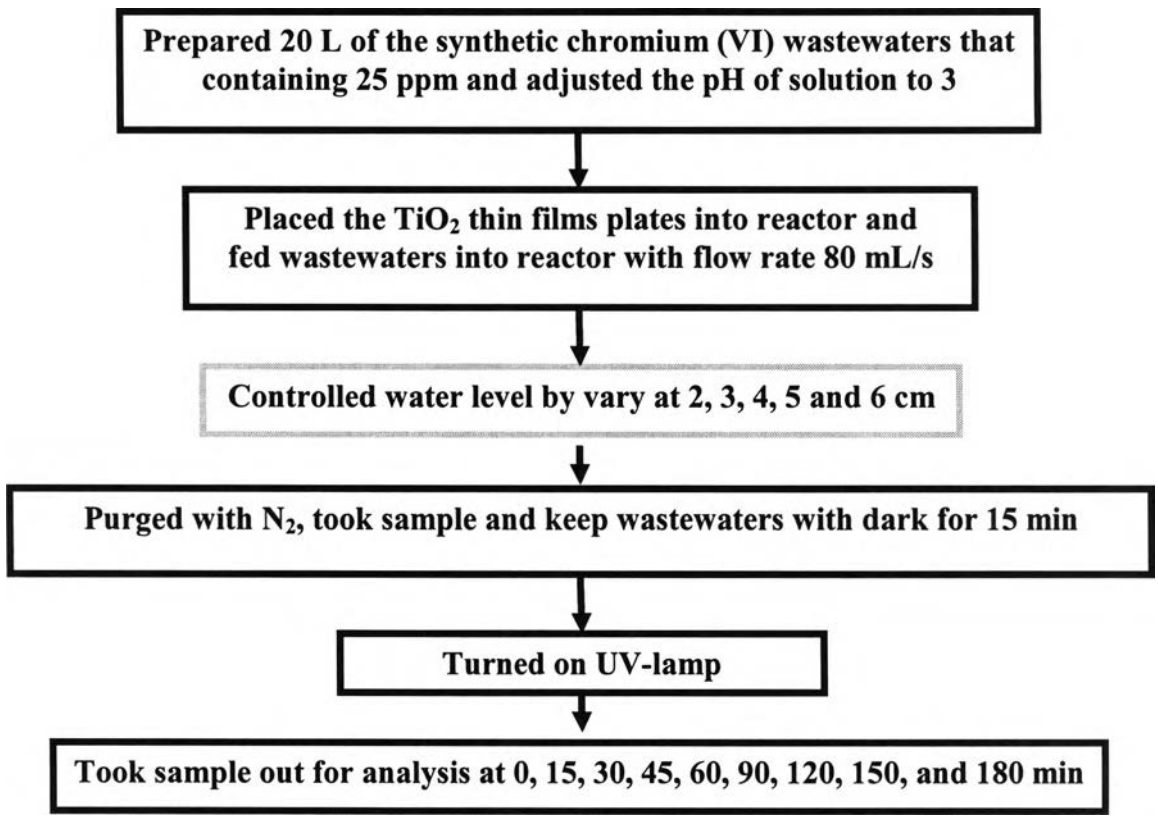

Figure 3.5 Flow chart of investigation effect of water level of wastewaters

3.3.4 Investigation of TiO_2 coating surface area that influence on the photocatalytic-reduction efficiency of chromium (VI)

Prepared synthetic chromium (VI) wastewaters by contain 50 ppm. TiO_2 thin films plates prepared from Part 3.2 were placed into the reactor by vary the number of plates and then turned on inlet pump to feed synthetic wastewaters into the reactor until it got the water level that required. Then, adjusted feed flow rate that required and turned on outlet pump to keep the water level of wastewaters. Before the

irradiation, purged with N_2, took sample for analysis and kept the wastewaters in the dark with the TiO_2 thin films plates for 15 minutes to allow for the adsorption process. At that moment, the lamps were turned on. Samples were taken at various time and stored for analysis. Flow chart is provided in Figure 3.6.

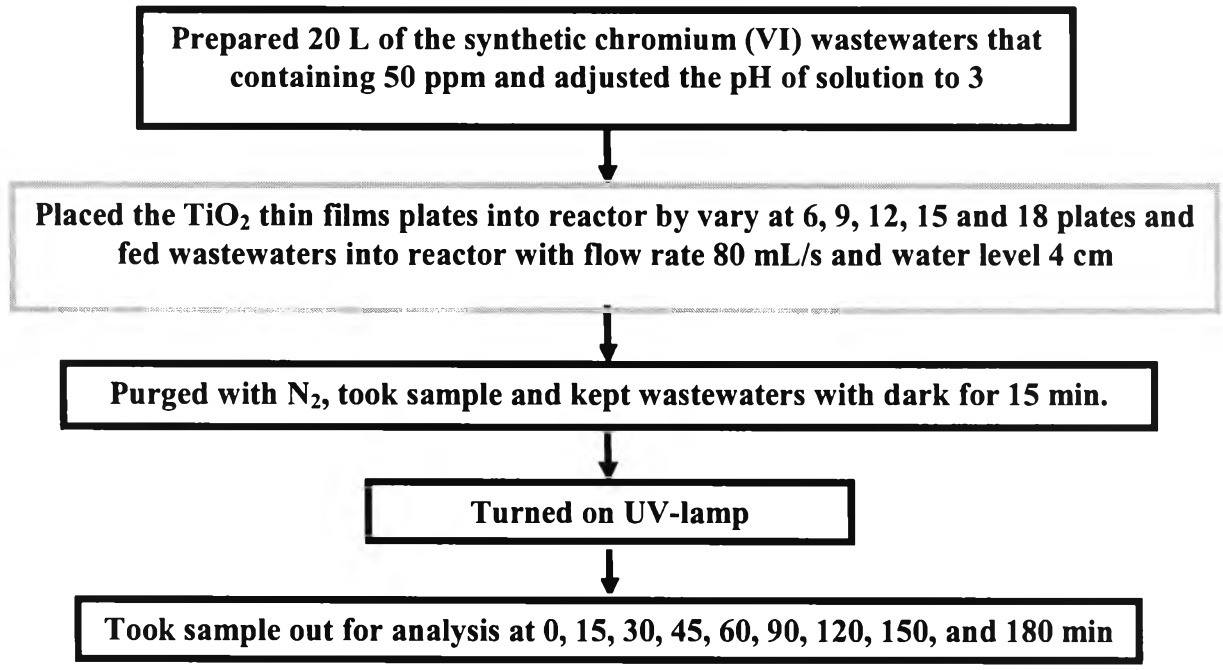

Figure 3.6 Flow chart of investigation effect of TiO_2 coating surface area

3.3.5 Investigation of initial concentration of chromium (VI) that influence on the photocatalytic-reduction efficiency of chromium (VI)

Prepared synthetic wastewaters by vary concentration. TiO_2 thin films plates prepared from Part 3.2 were placed into the reactor by vary the number of plates and then turned on inlet pump to feed synthetic wastewaters into the reactor until it got the water level that required. Then, adjusted feed flow rate that required and turned on outlet pump to keep the water level of wastewaters. Before the irradiation, purged with N_2, took sample for analysis and kept the wastewaters in the dark with the TiO_2 thin films plates for 15 minutes to allow for the adsorption process. At that moment,

the lamps were turned on. Samples were taken at various time and stored for analysis. Step chart is provided in Figure 3.7.

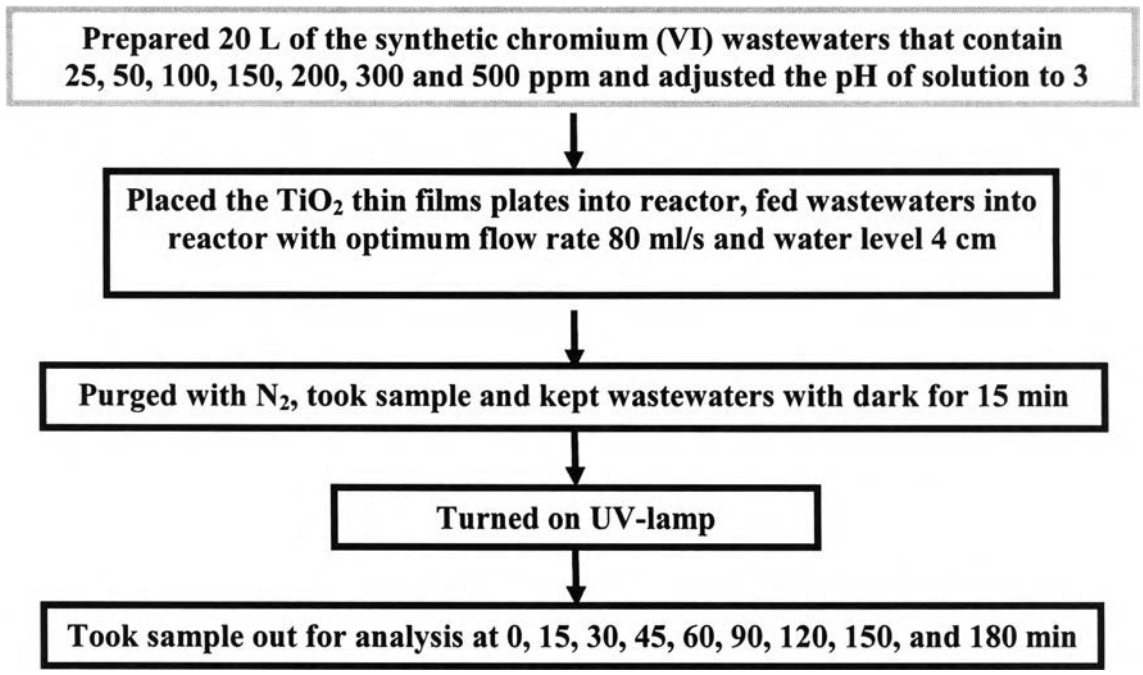

Figure 3.7 Flow chart of investigation effect of initial concentration of wastewaters

3.4 Measurement method

Chromium (VI) concentrations were determined by diphenyl-carbazide colorimetric method (APHA, AWWA, WPCF, 1998):

- take sample 5 mL
- add 0.25 mL conc.H3PO4 and adjust to pH 1 ± 0.3 with sulfuric acid and sodium hydroxide
- dilute with distilled water until get 100 mL
- add diphenyl-carbazide 2 mL
- measure adsorption with UV-VIS spectrophotometer