CHAPTER III
EXPERIMENTAL

3.1 Chemicals and Equipments

3.1.1 Chemicals

1. Bis (2,4-pentanedionato) - titanium (IV) oxide (reagent grade, TiO $(C_5H_7O_2)_2$, TCI Co., Ltd, Japan)
2. Vanadium oxide (99%, V_2O_5, analytical grade, Aldrich, Singapore)
3. Zinc oxide (99%, ZnO, analytical grade, Ajax, Australia)
4. Tropaeolin No. 2 (analytical grade, $C_{16}H_{11}N_2NaO_4S$, Fluka, UK)
5. Polyethylene glycol (100%, MW = 1000 g/mol, $(H(OCH_2CH_2)_nOH$, Wako, Japan)
6. Sodium hydroxide (analytical grade, NaOH, Merck, Germany)
7. Ethanol (99.8%, C_2H_5OH, Liquor Co., Ltd)
8. Nitric acid (65%, HNO_3, Merck, Germany)
9. Cleaning solution (MICRO-90, Cole-Parmer, USA)
10. Deionized water

3.1.2 Equipments

1. Photocatalytic reactor
2. Spin coator (WS-650Mz-23NPPB)
3. pH electrode
4. Vaccum pump (GAST0523-101Q-SG588DX)

3.2 Experimental Procedures

3.2.1 V_2O_5 and ZnO Doped TiO_2 Bilayer Films

A glass slide plate as the substrate was ultrasonically cleaned with the cleaning solution (MICRO-90) before coating. For first layer, TiO_2 film was coated on the glass plate with the solution containing 3 g of bis (2,4-pentanedionato)-titanium (IV) oxide and 3 ml ethanol by spin coating technique at 1,500 rpm for 20s and calcined at 400 °C for 1hr. On the second layer, ZnO and V_2O_5 layer was prepared by spin coating technique at the same conditions as above. The solution containing 4 ml TiO_2-sol with 0.6 g ZnO and 0.16 g polyethylene glycol was prepared. ZnO particles were obtained from the calcination temperatures at 300, 400 and 500 °C for 1 hr, and denoted as 300ZnO, 400ZnO and 500ZnO, respectively. For V_2O_5 bilayer, was prepared at the same condition whereas the solution containing 4 ml TiO2-sol with 0.01 g V_2O_5, then denoted as $300V_2O_5$, $400V_2O_5$ and $500V_2O_5$, respectively. TiO_2 sol was systhesized by sol-gel technique using titanium isopropoxide (TIPP) as a precusor. Firstly, 1.4 ml of 70 % HNO_3 was diluted with 200 ml deionized water then added to 16.7 ml of TIPP under vigorous stir for a couple of days until clear solution was obtained. The resulting solution was dialyzed by a membrane to obtain TiO_2 sol at a pH condition of 3.5 ± 0.1. The V_2O_5 layer was coated on the TiO_2 layer was calcined at 350 °C for 1 hr to obtain a V_2O_5/TiO_2 bilayer films (Buama *et al.*, 2012).

3.2.2 Photocatalytic Activity

A 200 ml of a 20 ppm acid orange 7 (AO7) solution was for the photocatalytic activity experiments. Adsorption of AO7 on the bilayer films was allowed before the start of each experiment. An experiment started by turning on the light source. The solution was subject to 2 hr illumination before the light source is turned off for 2 hr. The light was then turned on for another 2 hr before off for 2 hr. The experiment setup was blocked from any interference from other light sources by using black box throughout the experiment. The solution samples were taken every hour for quantitative analysis using a UV-Vis spectrometer (Buama *et al.*, 2012).

3.2.3 Physical Characterization

The morphology of V_2O_5/TiO_2, ZnO/TiO_2 bilayer films was examined by SEM (scanning electron microscope). The crystalline structure and crystal size was determined by X-ray diffraction measurement.

3.3 Analytical Technique

3.3.1 UV-Visible Spectrometer

The solution was tested every hour for quantitative analysis by using UV-Visible spectrometer (UV 1800). The degradation of AO7 was calculated by Eq 3.1.

$$\%Degradation = \frac{C_n - C_{n-2}}{C_0} \times 100\% \qquad (3.1)$$

where C_n was a concentration at time (2,4,6,8hr)

C_0 was a concentration at time = 0

3.3.2 XRD Spectrometer

The V_2O_5/TiO_2, ZnO/TiO_2 bilayer films were detected X-ray diffractometer (XRD, Rigaku, Smarthlab) to analyze crystal structure, chemical composition, and physical properties of thin films.

3.3.3 SEM Spectrometer

Surface characterization of V_2O_5/ TiO_2, ZnO/TiO_2 bilayer films were carried out by scanning electron microscopy (Hitachi, S-4800).

3.3.4 pH Analysis

pH value was determined by pH electrode.

3.3.5 Zeta Potential Analysis

Point of zero charge (PZC) of bilayer film catalyst was tested by Zetasizer (model ZS).