

CHAPTER III EXPERIMENTAL

3.1 Photocatalyst Preparation by Microemulsion Technique

3.1.1 *Materials*

Heptane (C_7H_{16}), AR grade, supplied by Lab-Scan Asia was used as a continuous phase in W/O microemulsion. Anionic surfactant, Bis(2-ethylhexylsulfosuccinate) or AOT ($C_{20}H_{37}NaO_7S$) supplied by Fluka Chemicals was used to form microemulsion system. Chloroplatinic (H_2PtCl_6) supplied by Fluka Chemicals was used as a precursor to be reduced to Pt particle. Commercial titanium(IV)oxide (TiO_2) supplied by Fluka Chemicals was used as a supporting material. Hydrazine hydrate (N_2H_4) supplied by Carlo Erba Reagenti was used as a reducing agent. Acetone, AR grade, supplied by Lab-Scan Asia was used to break microemulsion in the separation process.

3.1.2 *Methodology*

The system of W/O microemulsion was prepared by dissolving the surfactant (AOT) into 50 ml of heptane to obtain 0.1 mole/l of AOT. Microemulsion was prepared by adding 1 ml of aqueous solution containing 0.05 mol/l of chloroplatinic acid dropwisely into the AOT/heptane phase. The mixture was then mixed vigorously throughout the preparation period. The aqueous solution 1 ml containing 0.5 mol/l of reducing agent, hydrazine hydrate, was then added dropwisely into the surfactant solution. The solution was left around 24 hours to allow the reducing agent react with the precursor completely. The transformation of the precursor to platinum particles could be

observed by the change in the color of the solution from slightly yellow to black. The platinum particles formed were then separated out from the microemulsion system by adding small amount of acetone about 5 ml to break the microemulsion. Consequently, the platinum particles could then precipitate out from the solution. The precipitate was washed by heptane, acetone, and hot water successively and then be collected by centrifuging by 10 min at 4000 rpm.

In the case of preparing platinum loaded on titanium(IV)oxide, the calculated amount of titanium(IV)oxide, corresponding to weight percent that would like to prepare, was added to the microemulsion before separation process. After the separation process, the catalyst was dried absolutely at 323K and further dried at 383K for 12 hours. Finally, the surfactant left behind the separation process was then removed by calcination at 573K for 3 hours.

3.1.3 *Catalyst Characterization*

X-Ray diffraction was employed to characterize the form of the synthesized catalysts and to confirm the complete formation of platinum particles.

Transmission electron microscopy (TEM), Philips 200 CX, was used to determine the particle size and size distribution of the photocatalyst particles synthesized. The sample was prepared by dropping an ultrasonically dispersion of particles onto a copper microgrid and dried in air.

BET surface areas of different types of catalysts were determined by Autosorb 1.

3.2 Photocatalytic Study

3.2.1 *Materials*

Chemicals

Isopropyl alcohol (C_3H_8O), IPA, AR grade supplied by Carlo Erba Reagenti was used as a reaction substrate in this study. Nitric acid (HNO_3) and Sodium hydroxide (NaOH) were used for adjusting the solution pH.

Gases

UHP nitrogen gas, air zero, and hydrogen gas were used for gas chromatography analysis.

UHP nitrogen gas and oxygen gas were used in the experiment of the influence of dissolved oxygen.

3.2.2 *Experimental Apparatus*

The photocatalytic reactor used in this experiment was a batch reactor, which is illustrated in Figure 3.1. The apparatus consisted of a cylindrical vessel containing 80 ml of the substrate solution. The vessel was wrapped with aluminium foil to prevent and reflect the UV light. The top of the vessel was closed to prevent the vaporization. A thermostat bath and magnetic stirrer were applied to the system to control the temperature and induce continuous mixing throughout the experimental period. The UV light source was a 11W low pressure mercury lamp (Philips) with a wavelength range of 100-280 nm which gave the energy high enough to activate the molecule of TiO_2 . The lamp was dipped into the reaction vessel in order to illuminate thoroughly the solution. The reactor was covered by the opaque box to prevent the outer effects especially, light and to protect the personnel from exposure to the harmful illumination of the UV light.

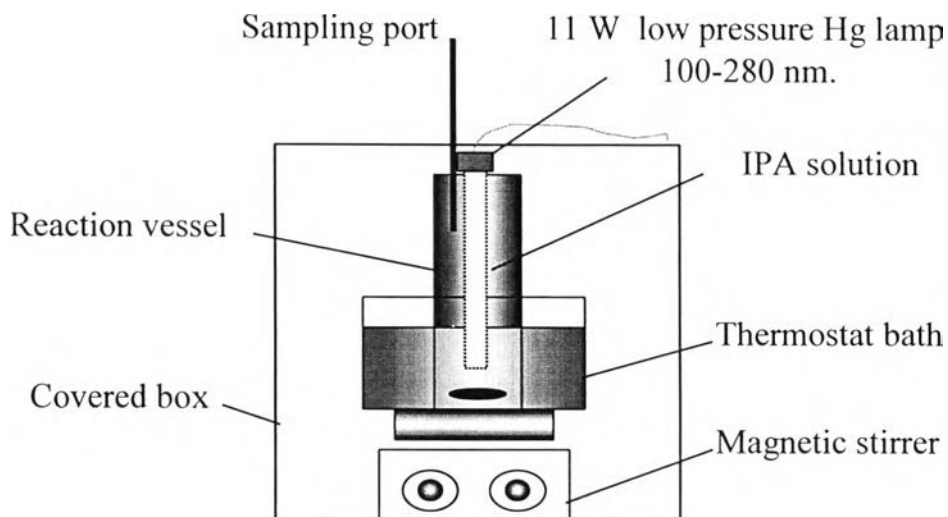


Figure 3.1 Schematic of the photocatalytic reactor used in this experiment

3.2.3 Methodology

The experiment was started from preparing a known concentration of isopropyl alcohol solution and then analyzed for concentration by using gas chromatography (GC-MS). The IPA solution of 80 ml was used for all runs of experiment. The required amount of the catalyst was added to the solution with vigorous stirring. Nitric acid and sodium hydroxide were used to adjust the pH. After the preparation step, the photocatalytic reactor was allowed to reach the set temperature. The timer was then started when the UV lamp was turned on. The solution was taken out every 90 minutes until 360 minutes to measure the IPA concentration.

In this study, the experiment was divided into three parts;

- 1) to study the photocatalytic degradation of isopropyl alcohol by using Pt, TiO₂, and Pt/TiO₂ as the photocatalysts.
- 2) to determine the effect of initial concentration of isopropyl alcohol, catalyst dosage, and initial pH on the rate of degradation.
- 3) to investigate the role of dissolved oxygen on the rate of degradation.

3.2.4 Analytical Method

Since the sample taken to analyze the IPA concentration is in the liquid phase that has the catalyst particles dispersed in the solution, the most appropriate instrument applied to analyze the IPA concentration should be gas chromatography equipped with head-space. Gas chromatography is able to analyze the composition of the reaction solution during the experiment both qualitatively and quantitatively. A Perkin-Elmer gas chromatograph was used in this work with a flame ionized detector (FID). Qualitative and quantitative analysis could be obtained from the chromatogram. By comparison of the retention times with standard substances, all chemicals produced could be identified. The concentration of any compounds could be calculated by using the calibration factor obtained from the calibration curve of a known concentration of that compound. The specifications and operating conditions of the gas chromatography and head-space are given in the Tables 3.1 and 3.2, respectively.

In the experiment involving to the role of dissolved oxygen, a DO meter, Orion model 860, was used to measure the value of the dissolved oxygen in the solution.

Table 3.1 Specifications and operating conditions of gas chromatography

Item	Specifications and operating conditions
Packing Column	10% CARBOWAX 20M, 80/100 Supelcoport
Carrier gas, flow rate	N ₂ , 10 ml/min
Injector temperature	150 °C
Detector temperature	160 °C
Oven temperature	250 °C
Detector	FID

Table 3.2 Specifications and operating conditions of head space

Item	Specifications and operating conditions
Sample temperature	80 °C
Needle temperature	90 °C
Transferred temperature	90 °C
Thermostat time	15 min
Injection time	0.1 min
Withdrawal time	0.2 min
Pressurized time	0.5 min