

## **CHAPTER III**

### **EXPERIMENTAL**

#### **3.1 Materials**

- Bisphenol-A (BPA) (Aldrich), (100% purity), 500 g
- Formaldehyde (Merck), (37%wt. in water), 1L
- Triethylenetetramine (TETA) (Fluka), (80% purity), 100 g
- Palladium(II)chloride anhydrous(Aldrich), (60% purum), 5 g
- Biodiesel, Weerasuwan Co., Ltd.
- Dimethylformamide (DMF) (Lab-Scan), 2.5 L
- Commercial activated carbon(Fluka), 250 g

#### **3.2 Measurements**

- Field Emission Scanning Electron Microscopy (FE-SEM)
- Quantachrome/Autosorb-1 Surface Area Analyzer (SAA)
- Fourier Transformed Infrared Spectrometer (FTIR)
- X-ray diffractometer (XRD)
- Air-circulating Oven
- Oil bath
- Magnetic bar
- Stirring plate
- Furnace
- Temperature controller
- Stainless steel batch reactor
- Mass flow controller
- Peristaltic Pump

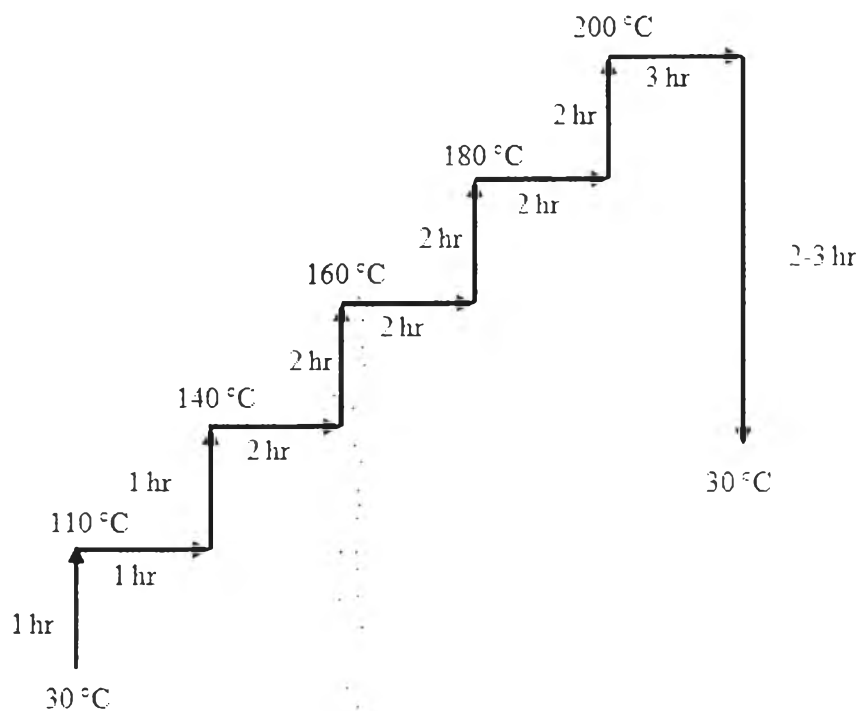
### 3.3 Methodology

#### 3.3.1 Synthesis of Polybenzoxazine Precursors

- a. Polybenzoxazine precursor is synthesis by dissolving bisphenol-A (2.283 g) in Dimethylformamide(DMF) (13.655 g) in a glass bottle and stirring until the clear solution is obtain.
- b. Formaldehyde solution (3.243 g) is then add into the bisphenol-A solution and stir continuously for approximately 20 min while the temperature is keep under 10oC using an ice bath.
- c. After that triethylenetetramine(TETA) (1.827 g) is add dropwise into the mixture and stirred continuously for approximately 1 hour while the reaction is cool with the ice bath until transparent yellow viscous liquid is obtain.

#### 3.3.2 Preparation of Polybenzoxazine carbon xerogel

- a. Polybenzoxazine precursor obtained from the reaction is transfer into vial and sealed at room temperature for 48 hours.
- b. The precursor is placed in an oil bath at 80 oC for 48 hours for gellation and then left at room temperature for ambient drying for 96 hours.
- c. Finally, the benzoxazine aerogel were placed in an air-circulating oven for step curing at 110oC for 1 hour and at 140, 160, 180 oC for 2 hours at each temperature, and at 200 oC for 3 hours, and then cooled down to room temperature as shown in figure 3.1.



**Figure 3.1** Schematic of a step of curing.

### 3.3.3 Preparation of Activated Carbon from Benzoxazone-derived Carbon Xerogel

- Benzoxazone-derived carbon aerogel will be calcined under  $N_2$  gas at  $800^\circ C$  for 2 hours.
- After that it will be activated at  $400^\circ C$  in Air for 30 min.

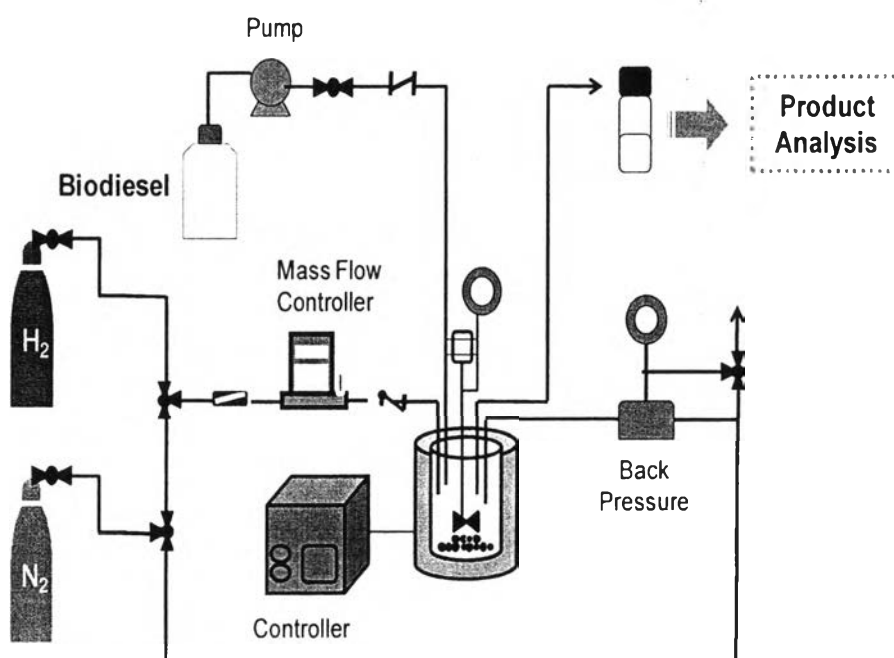
### 3.3.4 Catalyst Preparation for Activity Measurement

#### 3.3.4.1 *Biodiesel upgrading*

- Polyunsaturated FAMES will be carried out in a 300 ml stainless steel batch reactor at temperature and pressure of 80,100,120°C and 1,2,4 bar, respectively. Stirring speed will be maintained at 250,500 rpm to prevent external mass

transfer limitation. The flow rate of hydrogen gas will be 30,50 ml/min and will be controlled by mass flow controller.

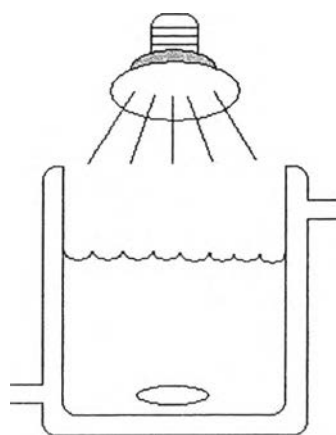
- b. The supported-Pd catalyst will be placed into the reactor. Then, the system will be purged with nitrogen to remove the remaining air.
- c. After that, the feed biodiesel obtained from Weerasuwan Co., Ltd. will be fed into the reactor by peristaltic pump. The reaction will be started by increasing temperature and pressure to the desired point.
- d. Finally, the liquid products will be collected and analyzed by Hewlett Packard gas chromatography 5890 Series II equipped with a FID detector. The schematic of the partial hydrogenation experiment is shown in Figure 3.2.



**Figure 3.2** Schematic of the partial hydrogenation reaction.

#### 3.3.4.2 Photocatalytic degradation of 4-nitrophenol

In order to study a photocatalytic activity of assynthesized porous titania compared with titania loaded on carbon xerogel, a photocatalytic reactor was used. The batch reactor itself consists of 500 mL glass container with 2 jacks for inline and outline water cooling. 100W Hg Philip UV Lamp was used as a light source. A magnetic stirrer and a cooler were used to keep the reactor at room temperature (Figure 4.3). The concentration of 4-NP used was 10 ppm and the solution was continuously, magnetically stirred. The concentration of a catalyst was fixed at 0.8 g./litre. Ten millimoles per litre of  $\text{H}_2\text{O}_2$  were then dropped into the mixture. Sampling of 10 mL of solution was done every 30 minutes until the illuminating time reach 4 hours to determine the concentration of 4-NP using a Shimadzu UV-240 spectrophotometer.



**Figure 3.3** The illustration of as studied photocatalst system

The concentration of 4-nitrophenol was resolved with UV-Visible Spectrometer by calibration curve of 4-nitrophenol solution (5, 10, 20, 30 and 40 ppm).

Preparation of porous titania using carbon xerogel as a support. Porous titania was prepared by dissolving titanium tetrabutoxide in ethanol to obtain a concentration of 0.25 M. Then 0.2 g of carbon xerogel was dispersed in 60 ml of TBOT solution by ultrasonic agitation and vacuum for 30 min, respectively. After that, the sample was being placed in air at room temperature for a day and at 80°C for 24 hr to complete the preparation process. TiO<sub>2</sub>/carbon xerogel were then pyrolyzed in N<sub>2</sub> at 500°C for 4 h., and calcined in air at 500°C for 24 h, resulting in porous titania.

### 3.3.5 Characterization of catalysts

The FT-IR spectra of polybenzoxazine precursor and carbon aerogel were obtained on a Nicolet Nexus 670 FT-IR spectrometer. KBr pellet technique was applied in the preparation of powder samples. X-ray diffractometer (Bruker AXS, Germany Model D8 Advance) with CuK<sub>α</sub> radiation, a generator voltage of 40 kV, and current of 30 mA. Field Emission Scanning Electron Microscopy (FE-SEM), Hitachi S-4800, was used to observe the surface morphology of carbon xerogel. Surface areas were calculated using the BET equation. The pore size distributions were constructed based on Barrett, Joyner and Halenda (BJH) method using the desorption branch of the nitrogen isotherm, measurement Microporous properties were calculated from t-plot method (Junbing *et al.*, 2008), mesoporous properties were analyzed by BJH method (Dresselhaus *et al.*, 2008), and specific surface area was determined by BET theory. UV-VIS spectrophotometer (Shimadzu UV-2550) was used to follow the adsorption of 4-nitrophenol.