



CHAPTER III EXPERIMENTAL

3.1 Materials

- 99.5% Bio-ethanol (Sapthip Co., Ltd.)
- SAPO-34 catalyst (Tianjin Chemist Scientific Ltd.)
- Quartz wool
- Alumina ball
- Helium gas (High purity)

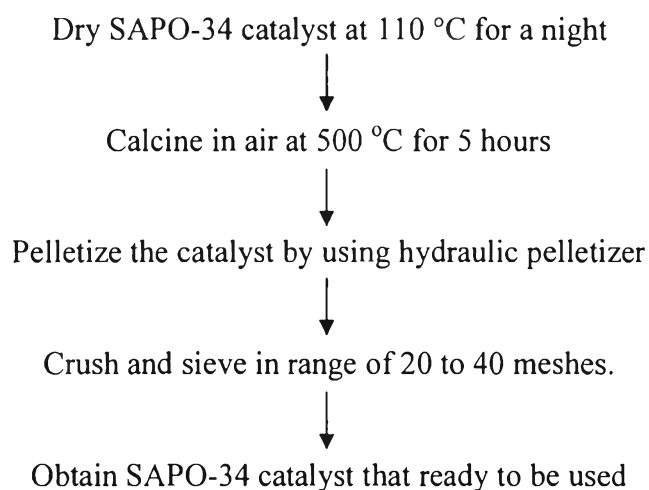
3.2 Equipment

- Bench scale reactor
 - Stainless-steel tubular reactor
 - Electric furnace
 - Mass flow controller
 - Thermocouples
 - Temperature program controller
 - Syringe pump
 - Syringe and needle
 - Valve and fitting
 - Heater cable
 - Tedlar bag
- Agilent Technologies 6890, Gas chromatography (GC)
- Rigaku/Rint 2200HV (X-Ray Diffraction Spectroscopy)
- Thermo Finnigan Sorptomatic 1990 (Surface Area Analyzer)
- Oxford/6111 (Scanning Electron Microscope, SEM)
- Perkin Elmer/Pyris Diamond (Thermogravimetric/Differential Thermal Analysis, TG/DTA)

3.3 Methodology

3.3.1 Catalyst Preparation

SAPO-34 catalyst used in this work was supplied from Tianjin Chemist Scientific Limited. SAPO-34 catalyst was dried overnight at 110 °C and calcined with the heating rate of 10 °C/min at 500 °C for 5 hours in order to remove impurities from the catalyst. Before catalyst was used in the reactor; it was pelletized using a hydraulic pelletizer. Then the pellets were crushed and sieved in range of 20 to 40 meshes. The catalyst preparation procedure can be simplified into the diagram as shown below;



3.3.2 Catalytic Reaction

The experimental set-up is shown in Figure 3.1. The reaction was carried out in an isothermal fixed bed stainless steel U-tube reactor with 0.402 inch inner diameter at atmospheric pressure. Three grams of catalyst sample was loaded into a middle part of the reactor and held in place using a quartz wool support. The ethanol was fed into helium stream using a syringe pump. The ethanol passed through the catalysts at various LHSVs in the range of 0.2 h⁻¹ to 1.0 h⁻¹, and time on stream of 45 – 180 minutes. The temperature of the reactor was varied in the range of 350 °C to 500 °C. The reaction products were passed through a condenser in order to drop liquid components. The gaseous products were analyzed by online gas

chromatography equipped with thermal conductivity (TCD) and flame ionization (FID) detectors.

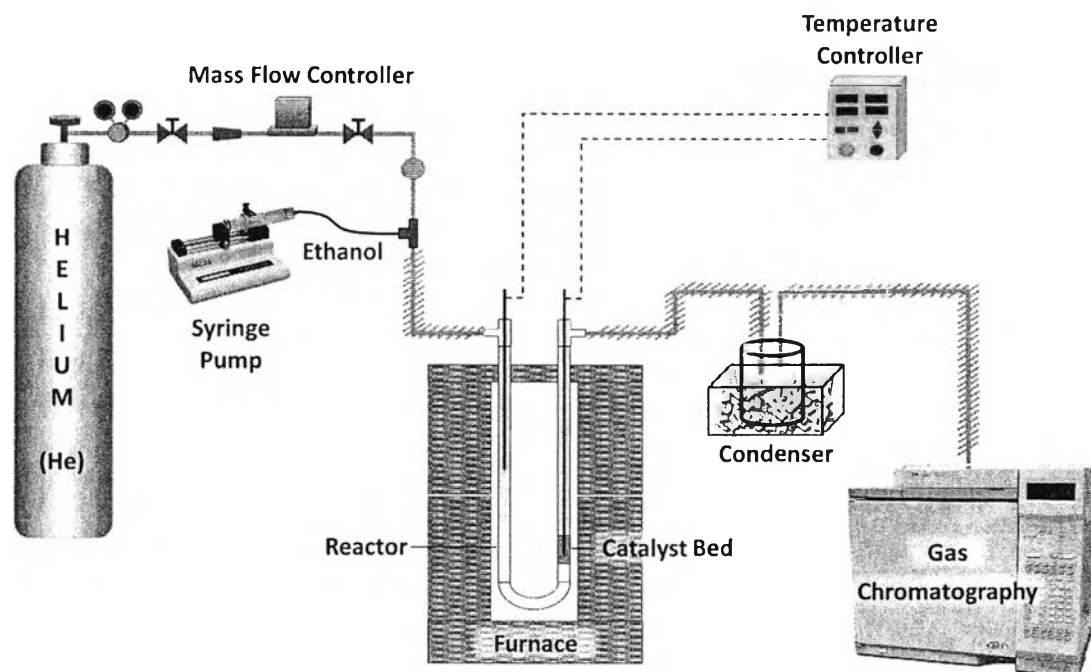


Figure 3.1 Experimental set-up.

3.3.3 Catalyst Characterization

3.3.3.1 *X-ray Diffraction Spectroscopy (XRD)*

The crystalline phase of catalyst was analyzed by a Rigaku, Rint X-Ray diffractometer system (RINT-2200) with Cu tube for generating $\text{CuK}\alpha$ radiation (1.5406 \AA) and nickel filter. A catalyst sample was packed in a glass specimen holder that places in the goniometer using $\text{CuK}\alpha$ small radiation and operated at 40 KV and 30 mA. This prepared catalyst sample was scanned from 5 degrees to 60 degrees (2θ) with a scanning rate of 0.02 degrees/min. The data from XRD were analyzed and recorded by an on-line computer. This analysis is generally performed based on the fact that an x-ray diffraction pattern is unique for each crystalline substance. Thus, if an exact match can be found between the pattern between the unknown and sample, the chemical identity can be examined. It is also possible to make a relatively quantitative analysis by comparing the intensity of the

diffraction lines. When comparing the same crystalline substance of different samples, the higher intensity indicates the higher content.

3.3.3.2 Thermogravimetric/Differential Thermal Analysis (TG/DTA)

The coke formation on catalysts was determined by Thermogravimetric /Differential Thermal Analysis (TG/DTA). The samples were heated from the room temperature to 800 °C with the heating rate of 10 °C/min. Nitrogen and oxygen flow rate was about 100 ml/min and 200 ml/min, respectively.

3.3.3.3 Scanning Electron Microscope (SEM)

The microstructure of the sample was identified by the scanning electron micrographs (JEOL/JSM 5200). The SEM uses the electrons that are backscattered or emitted from the specimen surface. The samples were placed on a stub and coated with gold by using ion sputtering device. The specimen was scanned with a narrow beam of electrons. The quantity of electrons scatters or emit as the primary beam bombards each successive point of the metallic specimen surface will be measured by electron detector and used to modulate the intensity of a second electron beam and forms an image on a TV screen.

3.3.4 Product Analysis

The gaseous products leaving from the reactor were analyzed by the online analysis of gas chromatograph (Agilent Technologies 6890 Network GC system) using HP-PLOT Q column (30 m x 0.32 mm ID and 20 µm film thicknesses) with a thermal conductivity detector (TCD) to determine hydrocarbons products and using DBWAX column (30 m x 0.32 mm ID and 20 µm film thicknesses) with a flame ionization detector (FID) to determine oxygenate components.