CHAPTER IV

EXPERIMENTAL

This chapter contains details about : catalyst preparation method, selective catalytic reduction (SCR) of nitric oxide with ammonia system, and characterization of catalyst. In each section, details of experimental procedures, including the materials and apparatus are described.

The scope of this study

type of $DeNO_x$ catalyst is used in this study :

TiO_2, V_2O_5, (6-30 %) V_2O_5/TiO_2

the reaction condition as follows :

Reaction Temperature	:	room temperature - 500	^{o} C
Operating Pressure	:	1	atm
Space Velocity	:	4,000-20,000	hr^{-1}

component of inlet gas are as follows :

Nitric oxide	:	0, 500	ppm
Ammonia	:	0, 500	ppm
Oxygen	:	0, 2 %	vol

4.1 Preparation of catalyst

TiO_2 support, (manufactured by Farmitalia Carlo Erba, Italy.), was grounded to the require mesh size of 60-80 mesh. Then 5 grams of the support was put into an aqueous solution containing an appropriate amount of ammonium metavanadate (NH_4VO_3), to yield the require vanadium loading. The mixture was continuously stirred and heated t 100 ^oC until all water evaporate.

The obtained catalyst was further dried in air at overnight. After drying the catalyst was calcined in air, 100 ml/min, at 380 ^oC for 3 hours. The heating rate was 10 ^oC/min.

4.2 Reaction of reduction of NO_x system

The reaction system consists of a microreactor installed in a tube furnace. The diagram of the system is exhibited schematically in Figure 4.1. The furnace temperature is controlled by a temperature controller. The microreactor is constructed from a quartz tube. A gas mixture ($NO+NH_3+O_2$) is used as a reactant gas. DeNO_x catalyst were prepared by passing the gas mixture through the catalyst bed which was maintained at a temperature of 500 °C. During the experiment, the reaction temperature is monitored using a thermocouple and a digital temperature indicator. The effluent gas is analyzed by a NO_x analyzer equipped with a NO_x detector by chemiluminescence method. The operating condition of NO_x analyzer are shown in Table 4.1.

Table 4.1 Operating condition of NO_x analyzer (model NOA-7000).

Model	NOA-7000
Measured Component	NO_x by Atmospheric pressure chemiluminescence method
Range	0 - 1 000 ppm
Response time	Approx. 30 seconds
Sampling flow rate	Approx. 1 000 ml/min
Air flow rate	1 750 ml/min
Display	LCD, 320 x 200 dots

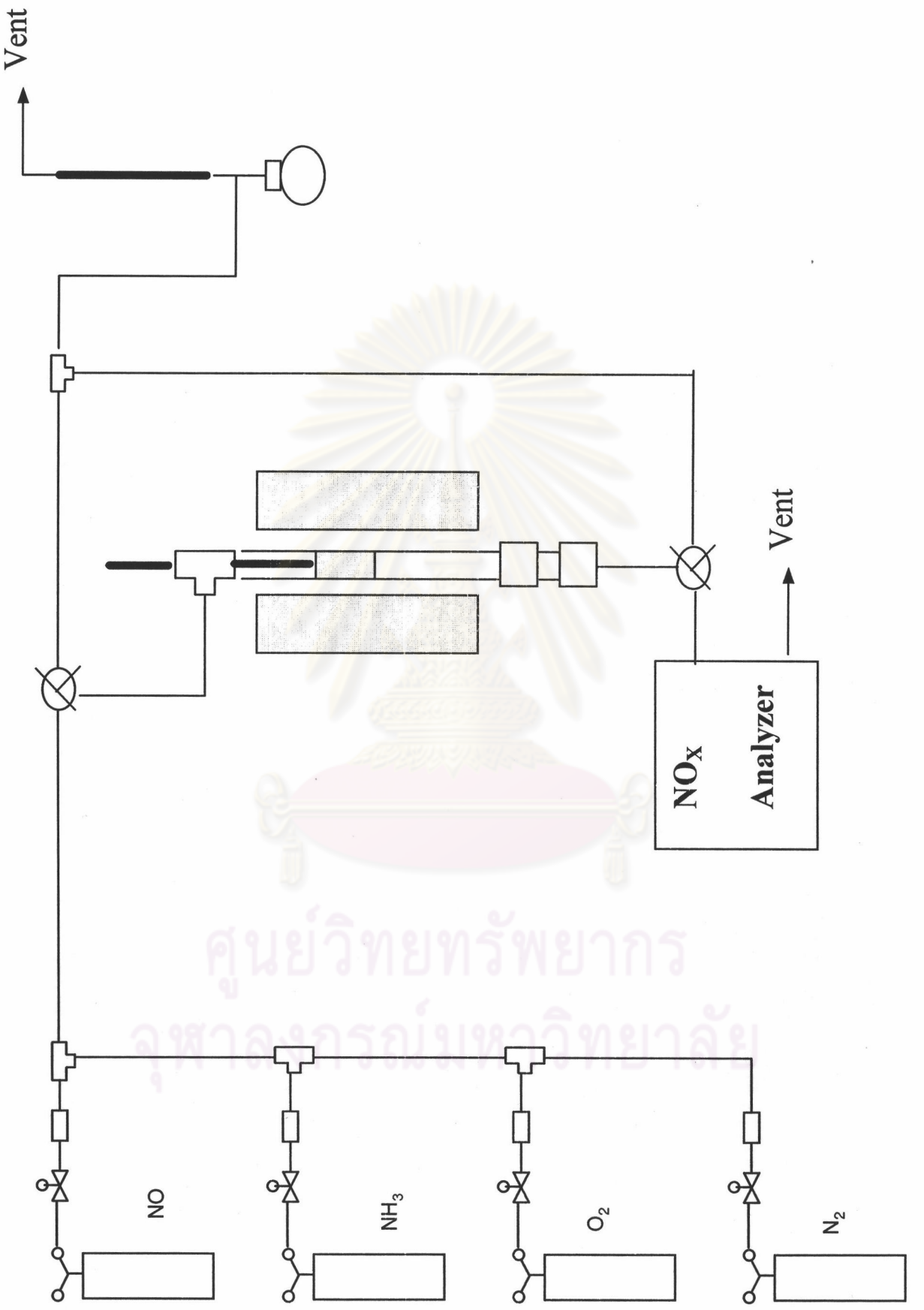

FIGURE 4.1 Flow diagram of the reaction of NO reduction system

4.3 Experimentation

The experimental procedures are described in detail below.

1. 0.5 grams of catalyst was packed in the middle of the quartz microreactor. The reactor was then placed in the furnace and the mixture gas ($NO+NH_3+O_2$) in nitrogen, including high purity nitrogen was balancing, was introduced into the reactor at a flow rate of 200 ml/min.

2. The reduction of NO_x was started up at 50 °C. The temperature was raised to 500°C at the heating rate of 5°C/min. When the temperature was 50°C, the effluent stream was sampling every 10 minutes by on-line gas sampler.

3. The amount of nitric oxide reduced was measured by NO_x analyzer.

4. After the catalyst temperature reached 500°C, these mixture gas in nitrogen was changed to high purity nitrogen and the reactor was cooled down.

Note : In some run SO_2 and H_2O were added to the reactant gas to observe their effect on the catalyst be property.

4.4 Characterization of the catalyst

4.4.1 X-ray Diffraction Pattern

X-ray Diffraction (XRD) patterns of the catalysts were performed using X-ray diffractor (model D-5000, SEIMENS) at Petrochemical Engineering Research Laboratory of Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University.

4.4.2 Specific surface area Measurement

Surface area of the catalysts were measured by the BET method, with nitrogen as absorbent using a micrometrics model ASAP 2000 at liquid-nitrogen temperature at Analysis Centre of Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University.

4.4.3 Chemical Analysis

Percentage of vanadia loading was analyzed by Atomic Absorption Spectrometry (AAS) method, at the Scientific and Technological Research Equipment Centre, Chulalongkorn University.

4.4.4 Fourier Transform Infrared Spectrometer (FT-IR)

FT-IR spectra were measured at room temperature on Impact 400 with a resolution of 4 cm^{-1} and an average of 50 scans, at Petrochemical Engineering

Research Laboratory of Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn