CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 Catalyst Preparation Materials

-Titanium dioxide was obtained from J.J. Degussa Hüls (T) Co. Ltd.

-Glass wool

-Distilled water

3.1.2 Reactant Gases

-Helium (He) of 99.95% purity was obtained from Thai Industrial Gas (Public) Co., Ltd.

-Oxygen (O_2) of 99.5% purity was obtained from Thai Industrial Gas (Public) Co., Ltd.

-Ethylene (C_2H_4) of 99.99% purity was obtained from National Petrochemical (Public) Co., Ltd.

3.2 Catalyst Preparation

Catalysts used in this work were prepared by dipping a sheet of glass wool as the catalyst support in TiO_2 slurry. The treated glass wool was cut to the size of 2×2 cm^2. Since there was wax or binder in the glass wool, it was treated by calcination a furnace with the heating rate of 3°C/min and maintained at 450°C for 2.5 hr and the flow rate of air was held at 10 ml/min.

The TiO_2 slurry was prepared by mixing 0.2 g of Degussa P-25 with 9.8 ml of distilled water. The treated sheet of glass wool was immersed in this slurry, and then dried in the oven at 100°C for 10 min. The coated glass wool tubes were annealed in the furnace with the heating rate of 3°C/min and maintained at 450°C for 3 hr and then cool down with the cooling rate of 5°C/min. The catalyst was white in appearance after annealing. The amount of TiO_2 loading was about 8-10 wt %.

3.3 Catalyst Characterization

Surface areas of the prepared catalysts were obtained by a Quantachrom surface area analyzer (Autosorb-1). A sample was degassed at 150°C overnight before characterization. Nitrogen was used as the probe gas. A small sample was dried and outgassed in the sample cell at 150^0C for at least 4 hours before adsorption. The specific area of each catalyst was calculated from the 5 points adsorption isotherm. The results were analyzed by using Autosorb ANAGAS software version 2.10.

Crystalline phases of catalysts were determined by a Rigaku X-ray diffractometer (RINT-2200) equipped with graphite monochromator and a Cu tube for generating CuK_α radiation (λ = 1.5406 ^{0}A) at a generator voltage of 40 kv and a generator current of 30 mA. Nickel filter was used as the K_α filter. The goniometer parameters were divergence slit = $1^0(2\theta)$; scattering slit = $1^0(2\theta)$; and receiving slit = 0.3 mm. The samples were a fine powder and fiber. They was held on a glass slide holder and was examined between 5 to $90^0(2\theta)$ range at scanning speed 5^0 (2θ)/minute with scan step of 0.02^0 (2θ). The digital output of proportional x-ray diffractor and the goniometer angle measurements were sent to an online microcomputer to record the data and subsequent analysis. The X-ray patterns of the catalysts were compared with that of Degussa P-25.

Dispersion of TiO_2 on the glass wool was investigated by a JOEL scanning electron microscope (JSM-5200). A sheet of samples were placed on the stubs and coated with gold by JOEL ion sputtering device (JFC-1100) before characterization.

3.4 Experimental Procedure

A schematic diagram of the experimental set-up in this work is shown in Figure 3.1. All gas flow rates were controlled by Sierra mass flow controllers. 0.7 μ m in-line filters were used to protect contaminants from the feed gas before passing to the mass flow controllers. Check valves were also placed at the outlet of the mass flow controllers to prevent backpressure effects. A reactor was made from quartz

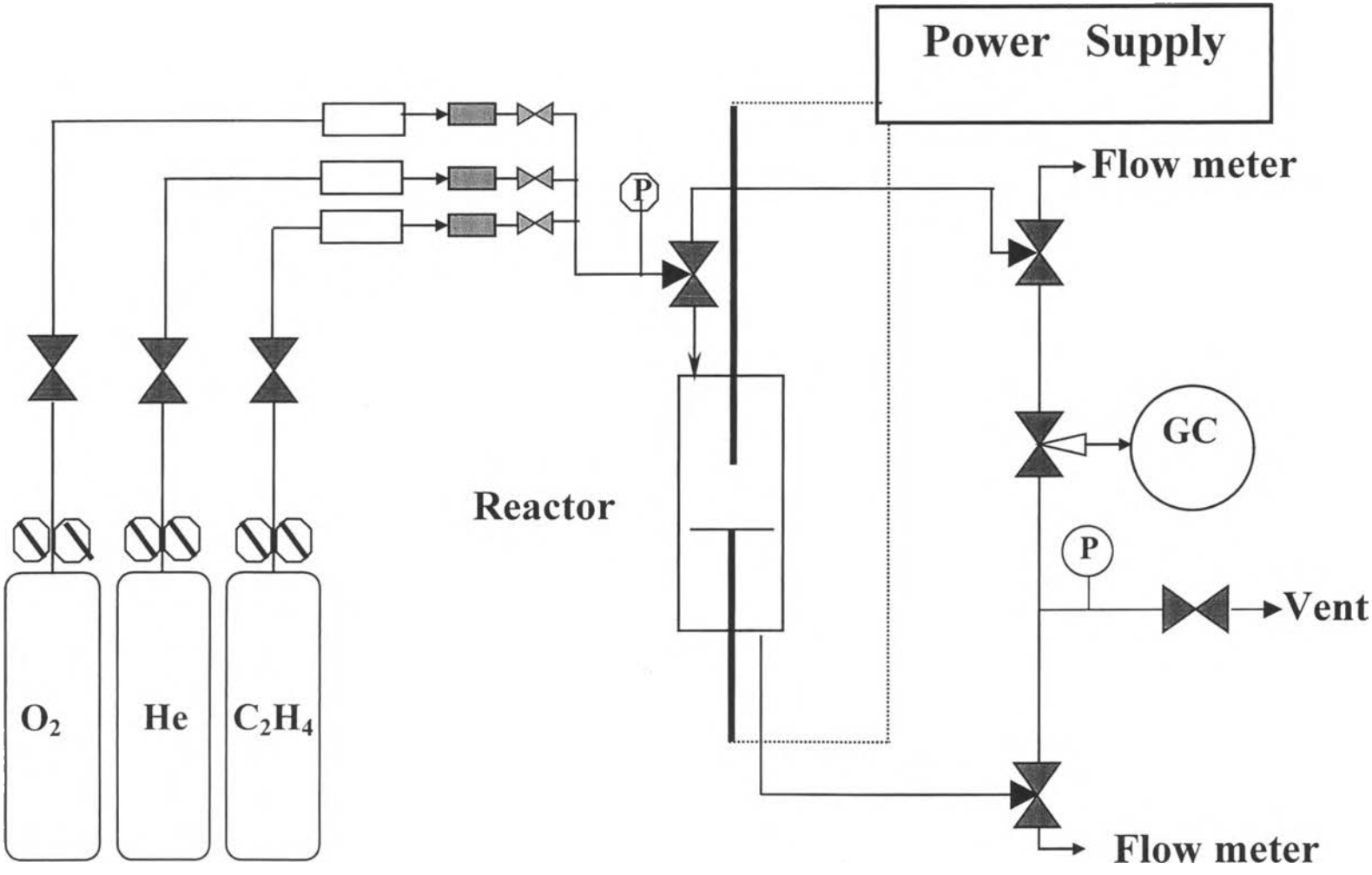

Figure 3.1 The schematic diagram of the experimental set-up

with 10 mm OD and 8 mm ID. Plasma was generated in the reactor through a stainless steel rod and circular disk. Power used for generating plasma was an alternative current power, 220V and 50 Hz, which was transmitted to a high voltage current. The output voltage was increased up to the maximum by 125 times and the signal of alternative current was a sine form.

The experiment started with analyzing the feed gas composition using a Perkin-Elmer gas chromatograph equipped with a thermal conductivity detector. The GC conditions were summarized as follows:

Injection temperature:	160°C
Oven temperature:	120°C for 5 min
	170°C (heating rate 10°C/min)
	held for 20 min
Carrier gas:	High purity helium
Carrier gas flow rate:	30 mL/min
Column type:	Packed column (Carboxen 1000)
Detector temperature:	200°C

The reactant gases consisted of oxygen and ethylene, which was diluted by 82 vol %of high purity helium. The ratio of oxygen to ethylene was set at 5:1. After the concentrations of reactant gases were constant, the supply power unit was turned on. The gas products were analyzed after 30 min.

3.5 Studied Conditions

The experiments were divided into 2 main parts: effect of plasma, and plasma with photocatalyst. All parameters studied were summarized in Table 3.1.

Table 3.1 Experimental conditions studied

Effects	**Gas flow rate (ml/min)**	**AC Frequency (Hz)**	**Input power (W)**	**Gap (mm)**	**TiO_2 Loading (g)**
Plasma	20-80	200-400	3.5-5	4 - 10	8 – 10
Plasma and Photocatalyst	40	200	3.5, 4.2		