CHAPTER IV
TPO TESTING

4.1 Experimental Procedures Informations

4.1.1 Testing and characterization

4.1.1.1 FID Calibration

- CH_4 (flow-rate 30-60 cm^3/min) is fixed volume from six ports valve that varies with different volume, is fed into Flame Ionization Detector (FID) and it is observed in Temperature Programmed Oxidation (TPO) curves.

4.1.1.2 Methanator Calibration

- CO_2 (flow-rate 30-60 cm^3/min) is fixed volume from six ports valve that varies with different volume, is fed into methanator for methane conversion. The product from methanator is sent to Flame Ionization Detector (FID) and it is observed in Temperature Programmed Oxidation (TPO) curves.

4.1.1.3 Running process

In this experiment, 2%O_2 (flow-rate 30-60 cm^3/min) is fed into the quart reactor having catalyst inside. This quart reactor can vary the temperature from the room temperature to final temperature(900 ^oC) by adjusting of heating rate (10-15°C/min). The exit gases from the quart reactor consists of CO_2, O_2, H_2O, and He are passed through the methanator. In this equipment, the exit gas will convert to CH_4 by adding H_2 (using flow-rate at 20-25 cm^3/min). The final product (CH_4) will be transfered to Flame Ionization Detector (FID) and is observed in Temperature Programmed Oxidation (TPO) curves. The detection method greatly improves the sensitivity, the stability of the base line, and the resolution of the conventional TPO technique.

4.1.1.4 Characterization of TPO Curve

Type of coke can be analyzed by comparing the temperature at peak maximum with standard curve. The comparison between area under curve and standard curve results in, the concentration of CH_4 is known precisely. The accurate

concentration of CH_4 can be used to calculate the accurate quantity of coke by back conversion back to the previous reactions (Methanization and Oxidation)

4.2 Testing Steps

In the first place, gas valves on gas cylinder are opened. Then mass flow controller, temperature controller, FID and computer are switched on. Then the temperature controllers are set program for each case. FID is ignited in later. Next control panel is switched at measuring process. Next, hydrogen and 2%oxygen/He - measuring processes are measured by flow calibrator; consequently, the flow rate are 25 and 40 cc/min respectively. Then control panel is switched at running process. Next coke/catalyst is weighted about 10 mg. And loaded in the furnace. PeakSimple program on computer is opened, is the next process. In Acquisition menu, Run in pull down menus is pressed. To wait about 15-20 min for fine constant base line. Once base line is set to zero. Stop in pull down menus, in Acquisition menu is pressed. Then Channels in Edit menu is clicked.

Clicking on the Details button allows to adjusting the time for running (90min). Then O.K. button is clicked. Postrun in Channel controls is pressed. The post run testing name is input. Clicking on O.K. button. Run in pull down menus, in Acquisition menu is pressed again. To wait for 2 min, methane calibration process is done. Methane peak will appear. The running time is taken about 80 min. A sample peak appears too. Finally methane calibration process is done in 2 times again. In TPO which is in D:\ is provided for peak area calculation program. Coke amount is prepared coke amount calculation program.