



CHAPTER III

EXPERIMENTALS

3.1. Samples, Materials, and Chemicals

3.1.1 Waste Tire Samples

The waste tire used in this study was a used passenger car tire (about 50,000 kilometers). To avoid the difference caused by different tires, only one tire was used to prepare the sample. The tire was then cut into pieces by a lab-made up machine and then sieved to obtain the particle sizes of 8-18 mesh.

Table 3.1 summarizes the elemental composition of the waste tire used in this study determined by elemental analysis (LECO, US).

Table 3.1 Elemental composition of waste tire

Element	C	H	S	Other*
%wt	87.4	7.2	1.7	4.7

* Determined by subtraction.

3.1.2 Materials and Chemicals

- Dual Valve Tedlar PVF bag (Cole Palmer)
- Liquid chromatography column (Glass), 650 mm height, 26.6 mm inside diameter
- Silica for liquid chromatography (Particle size 0.063-0.200 ; 70-730 mesh ASTM)
- Neutral alumina for liquid chromatography
- Benzene (C_6H_6 , Assay $\geq 99.8\%$)
- Diethyl ether ($(C_2H_5)_2O$, Assay $\geq 99.5\%$)
- n-pentane ($CH_3(CH_2)_3CH_3$, Assay $\geq 99\%$)
- n-hexane ($CH_3(CH_2)_4CH_3$, Assay $\geq 99\%$)
- Methanol (CH_3OH , Assay $\geq 99.8\%$)

- Carbon disulfide, CS₂
- N₂ gas
- Beta (BEA), Y, Mordenite (MOR) Zeolites (Tosoh Corp., Singapore)
- Rh, Re, Ru, Pt, Ni (FLUKA, ALDRICH)
- KBr
- Teflon membranes (FLUKA)

3.2. Pyrolysis of Waste Tire

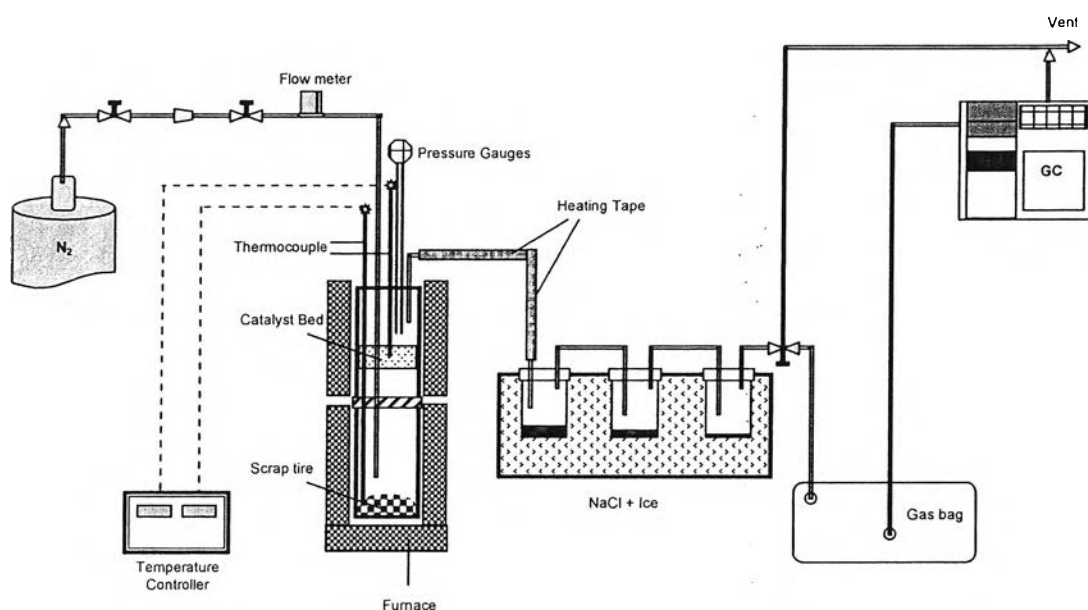


Figure 3.1 Schematic diagram of the pyrolysis process.

3.2.1 Pyrolysis

The experimental system is shown in Figure 3.1. A tire sample (30 g) was pyrolyzed in the lower zone of the reactor from room temperature to a final temperature of 500°C with a heating rate of 10°C/min. This pyrolysis zone was kept at the final temperature for 1 hour to ensure the total conversion of tire. The evolved product was carried by a 25 ml/min nitrogen flow to the upper zone controlled at a

desired temperature and packed with a catalyst. The influences of catalyst temperatures were investigated by varying the temperature in this zone. The obtained product was next passed through an ice-salt condensing system containing 3 condensers to separate incondensable compounds from the liquid product. The solid and liquid products were weighed to determine the product distribution. The amount of gas was then determined by mass balance.

3.2.2 Product Analysis

3.2.2.1 *Gaseous Products*

The gaseous product collected in a Dual Valve Tedlar PVF bag was analyzed by a Gas Chromatography, Agilent Technologies 6890 Network GC system, using HP-PLOT Q column: 30 m x 0.32 mm ID and 20 μm film thicknesses. A detector was FID type using He as the carrier gas.

3.2.2.2 *Liquid Product*

The analysis procedure of tired-derived oil was divided into two steps, which are: (i) asphaltene separation, and (ii) maltene analysis.

a. Asphaltene Separation

Table 3.2 The different solvents for maltenes separation (Sebor *et al.*, 1999)

Mobile phase	Volume (cm^3)	Prevailing compounds
Hexane	600	Saturated hydrocarbons
Hexane-benzene (24:1, v/v)	500	Mono-aromatics
Hexane-benzene (22:3, v/v)	500	Di-aromatics
Benzene	500	Poly-aromatics
Benzene-diethylether-methanol (1:1:3 v/v)	500	Polar-aromatics

For asphaltene separation, the liquid product was first dissolved in n-pentane with the ratio of 40:1 to precipitate asphaltene. The obtained solution was shaken in an ultrasonic bath at 30°C for 15 minutes, followed by leaving overnight at room temperature. Asphaltene was then filtrated using 0.45 μm teflon

membranes in a vacuum system. The filter membrane with filter cake was dried in a vacuum oven at 0.1 barg and 60 °C over 6 hrs. Finally, the filter cake was weighed to determine the amount of asphaltene. The solution after asphaltene removal was evaporated to separate n-pentane from maltene by a rotary vacuum evaporator at 37°C.

b. Maltene Analysis

Maltene (~ 4.5 g mixed with 10 cm³ n-hexane) was fractionated into saturated hydrocarbons, mono-, di-, poly- and polar aromatics by means of the liquid adsorption chromatography (Sebor *et al.*, 1999) using a standard column (650 height x 26.6 mm. I.D.), in which silica gel and alumina are packed at the upper and lower bed of the column, respectively. Alumina was activated at 400°C for 8 hours prior to being used. The column was pre-wetted with n-hexane. The flow rates of the mobile phases (solvents, Table 3.2) were kept constant at 20 cm³/min controlled by a fish pump. The mobile phases are then evaporated out by a rotary vacuum evaporator at 60°C.

A Varian CP 3800 Simulated Distillation Gas Chromatograph equipped with FID and a 15m x 0.25mm x 0.25µm WCOT fused silica capillary column (SIMDIST GC) was used to analyze the obtained maltene and hydrocarbon fractions according to the ASTM D2887 method. The conditions were as follows:

Initial temperature	30	°C
Time at initial temperature	0.01	min
Heating rate	20	°C/min
Final temperature	320	°C
Holding time	8.50	min

The maltene was also analyzed by mean of FT-IR technique using a Bruker instrument (EQUINOX55) with a resolution of 4cm⁻¹.