CHAPTER III

EXPERIMENTALS

3.1 Materials

3.1.1 Ethylene-Vinyl Acetate copolymer / Natural rubber composite source

- Ethylene-vinyl acetate copolymer(EVA)/natural rubber(NR) composites from shoe sole scrap was supplied by a local company. The scrap contains three layers. The scrap and bottom layers are consisted of 70% NR blend with 30% EVA, whereas the middle layer is made from 70% EVA blended with 30 % LDPE.

3.1.2 Silicon carbide

- Silicon carbide used as a microwave absorber in this research, was supported by National Metal and Materials Technology Center (MTEC). Its approximate size is 16 mesh.

3.1.3 Carrier gas

- Argon and Air Zero (purified air) was supplied from Thai Industrial Gases Public Company Limited, Thialand.

3.1.4 Dichloromethane analytical grade (DCM)

- DCM (Analytical grade) used for solvation of liquid products was purchased from LABSCAN Co.,Ltd (Thailand).

3.2 Equipments

3.2.1 Microwave Oven

- Microwave oven equipped with inverter (Panasonic's Straight Microwave Oven model NN-S674MF) was modified and used as microwave power source to ensure smooth and continuous power supply to the reactor.

3.2.2 Quartz Tube

- Quartz tube that has 13 mm outside diameter and 9 mm inner diameter was used in this research as a reactor.

3.2.3 R-Type Thermocouple

- R-Type thermocouple was applied to acquisition of temperature profile during the experiment.

3.2.4 Gas sampling bag

- 300 ml dual-valve gas sampling bags for gaseous products collection were purchased from Cole-Parmer, U.S.A.

3.2.5 Quartz wool

- Quartz wool diameter 5-20 μm was placed under the raw materials in the quartz tube acting as a filter to trap the impurity dust and raw materials retainer.

3.2.6 Mass Flow Controller

- Mass flow controller was applied to control the rate of reacting gas that flow through the reactor. AALBORC® Mass Flow controller was supported by National Metal and Materials Technology Center (MTEC).

3.2.7 Standard mixed gas

- Standard mixed gas with constant composition was used to determined species and content of gas products. The composition of standard mixed gas was show in table 3.1

Table 3.1 Show the composition of standard mixed gas that used in this experiment

Gas type	% Content by volumn	Gas type	% Content by volumn
Methane	5	1-Butene	10
Ethane	10	cis-2-Butene	5
Ethylene	1	Isopentane	2
Propane	5	n-Pentane	1
Propylene	1	CO_2	5
Isobutane	10	H_2	15
n-Butane	5	CO	5
trans-2-Butene	5	N_2	15

3.3 Instruments

3.3.1 Thermal properties

Thermal degradation of raw materials was studied by TGA to determine quantity of volatiles, fixed carbon, and ash. Figure 3.1 displays the METTLER TOLEDO TGA model TGA/SDTA 851 with STARe software version 8.1 for data evaluation used in this research. Moreover, elemental analysis technique (FISONS model NA-2000) was applied to determine CHON/S composition.

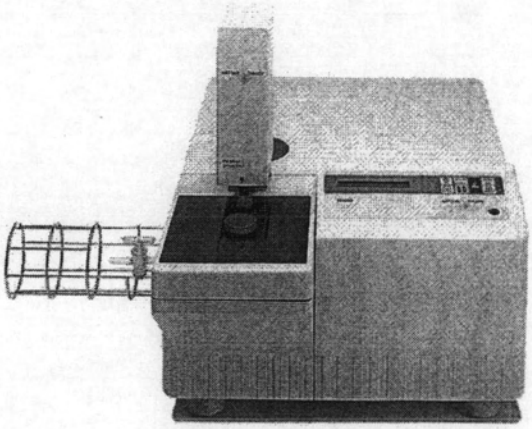


Figure 3.1 METTLER TOLEDO TGA

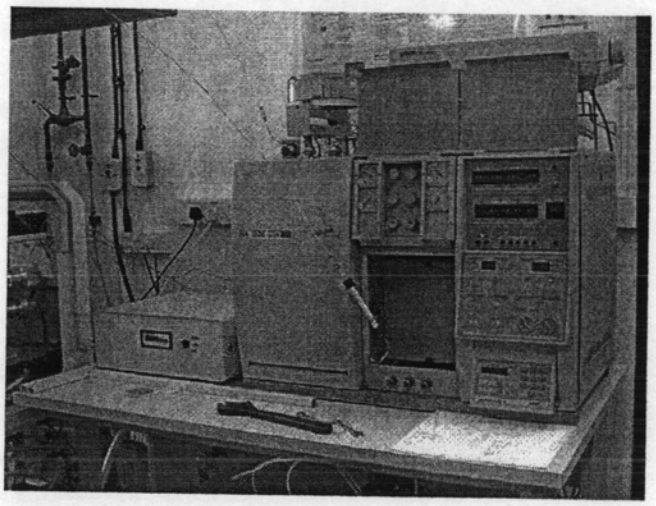


Figure 3.2 Elemental analyzer

3.3.2 Gas verification

Thermal conversion gas product was analyzed by gas chromatography technique in a Varian CP-3800 gas chromatography coupled to Flame Ionization Detector (FID) and Thermal Conductivity Detector (TCD) using a CP-Sil 8 CB low bleed capillary column with 1.0 μm thickness coating of 95% dimethyl polysiloxane, 0.32 mm inner diameter and 30 m long, as shown in Figure 3.3.

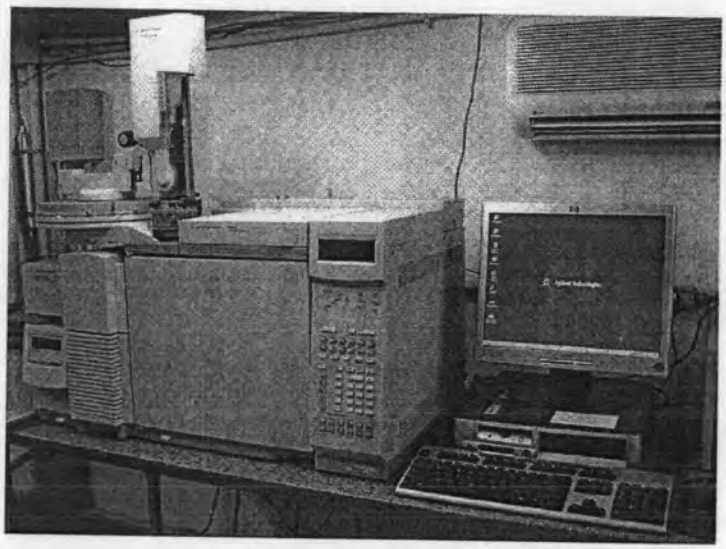
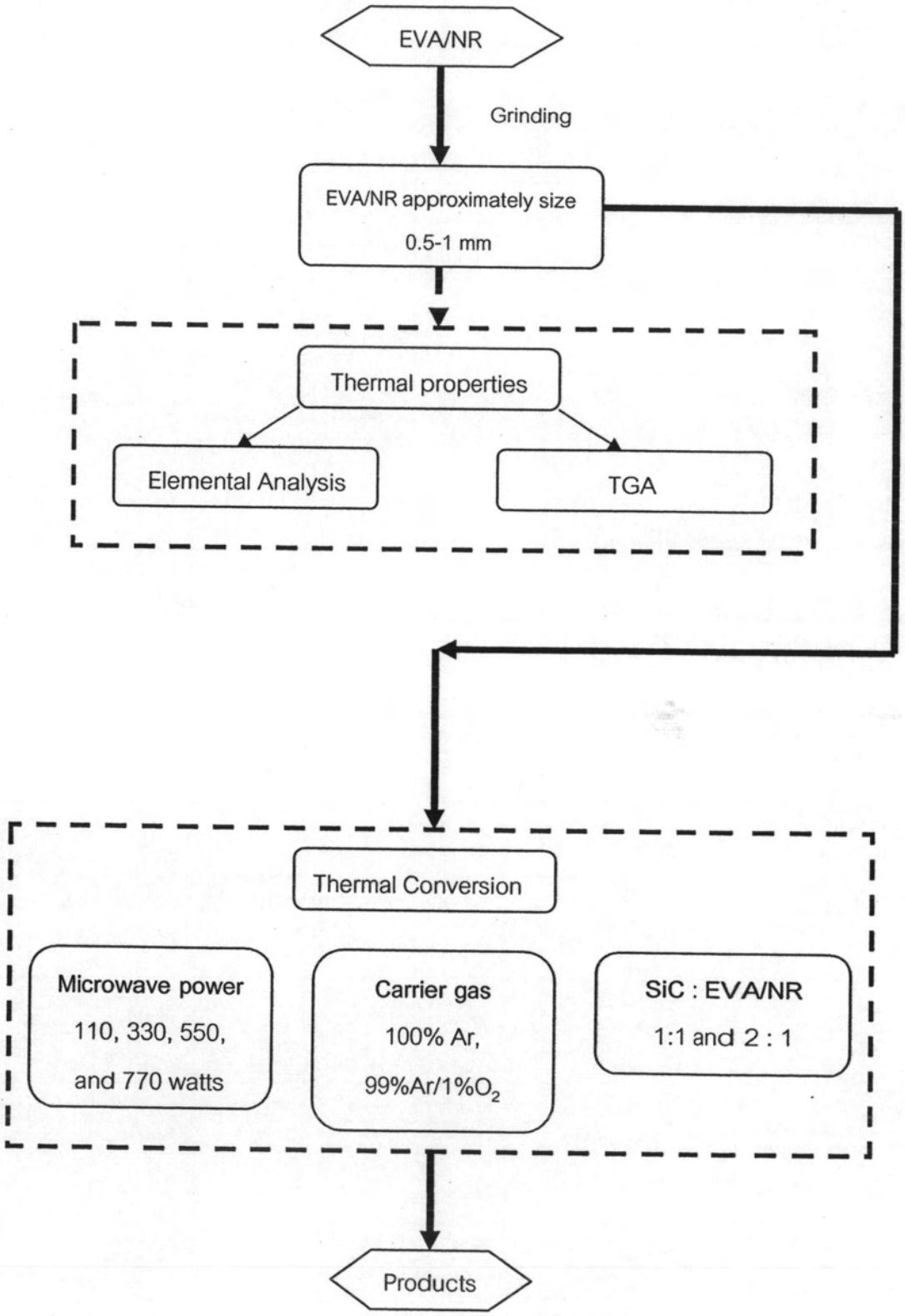
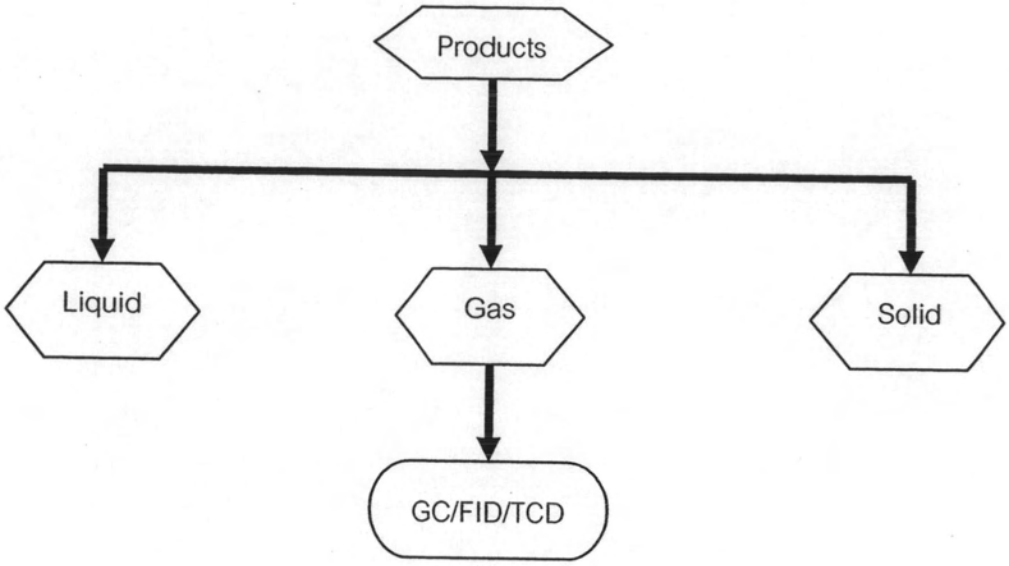


Figure 3.3 Gas Chromatrography / Flame Ionized Detector / Thermal Conductivity Detector (GC/FID/TCD).

3.4 Methodology

The flow chart of the entire experimental procedure is shown below in Figure 3.4





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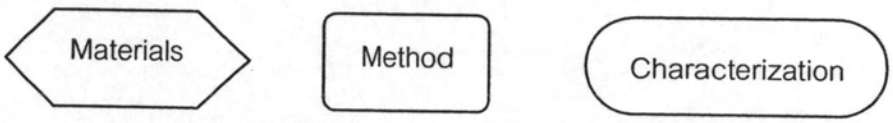

Figure 3.4 Flow chart of the experimental procedure.

3.4.1 EVA/NR investigation

EVA/NR was ground into evenly distributed sizes of approximately 0.5-1.0 mm by MING LEE STRONG CRUSHER machine that shown in Figure 3.5. Next, thermal conversion properties, volatile evolution, and fixed carbon content of ground EVA/NR powder was investigated by Thermogravimetric Analysis (TGA) under heating rate of 20°C/min from 50°C to 600°C under N_2 atmosphere at a gas flow rate 20 ml/min. Finally, elemental analysis of raw materials using CHON/S was performed by FISONS model NA-2000. The sample was pyrolyzed at high temperature.

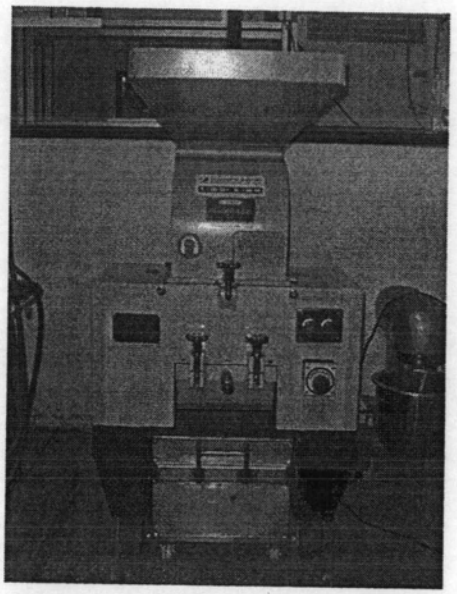

Figure 3.5 Ming Lee Strong Crusher.

3.4.2 Thermal conversion studies

Before the operation, EVA/NR and silicon carbide were mixed according to predetermined volume ratio and packed into quartz tube by sandwiching EVA/NR layer between silicon carbide layers as shown in Figure 3.6.

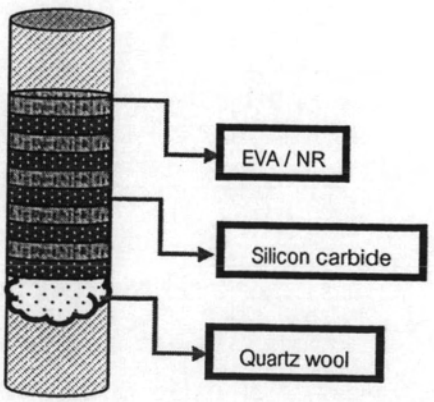

Figure 3.6 Sandwiching EVA/NR layer

The thermal conversion of EVA/NR composite was carried out in a single process using microwave cavity. Experiments were carried out by insertion of quartz tube into microwave chamber by vertically suspending the quartz tube at the middle of

the cavity and plugging type R thermocouple into the raw material bed for acquisition of temperature profile during the experiment as shown in Figure 3.7 and actual photographic of the experimental setup is included in appendix. Silicone tube was applied for condenser to collect condensable and semi-condensable gas from the operation.

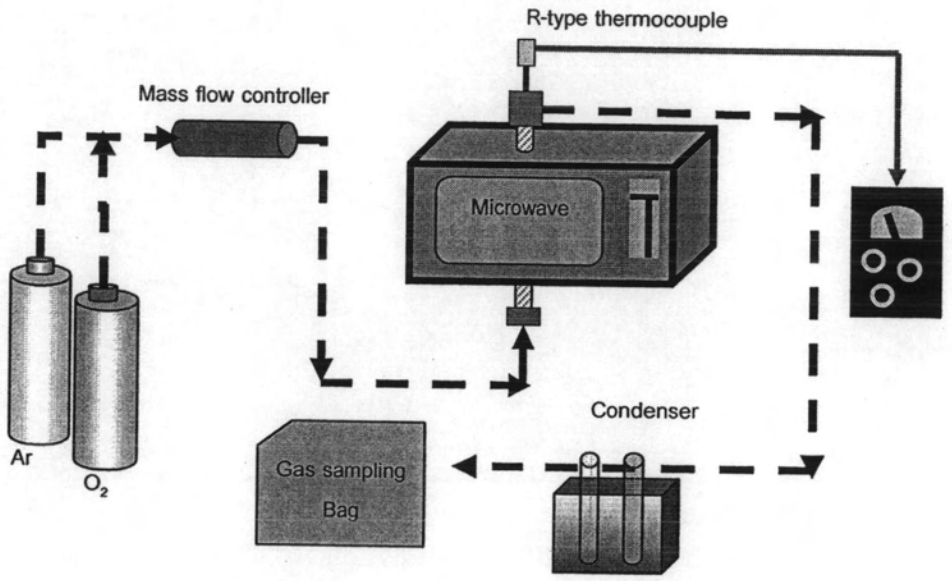

Figure 3.7 Operation of thermal conversion by microwave heating

In order to maintain the atmosphere during the operation, gas with desired fraction at flow rate of 100 ml/min was passed through the reactor for 10 minutes prior to each run. Atmosphere in this operation was composed of two different components; 100% Ar and 99% Ar/1% O_2. Gas feed was controlled by automatic mass flow controllers to maintain total flow of 100 ml/min. Input power of microwave oven was varied from 110, 330, and 550 watts. Silicon carbide was employed as microwave absorber. Thus, different amounts of silicon carbide ratio, 1:1 and 2:1, per EVA/NR was studied. Accurate measurement of temperature evolution during the process was very difficult because there were effects of microwave radiation to electronic apparatus as well as arching phenomena of thermocouple in the reactor which gave rise to hot spots. Some of volatiles evolve during the operation may be condensed on thermocouple which also affected the measurement. The gas product was passed through ice-cooled

condenser for trapping of condensables, and light gas was collected in series of gas sampling bags. First sample was collected from 0-180 seconds and the second one was obtained at 181-360 seconds. After each run, dichloromethane (DCM) was used to dissolve condensable products in the condenser and gas line, then collected. Weight of oil was measured by drying DCM at room temperature and weighted oil after dried. The solid and oil fraction yields were directly calculated from measured weight of each fraction, while gas yield was evaluated by difference. The weight of each products can be calculate by the following equations;

$$\text{Weight of gas and oil} = A - B = C \quad \text{.....(3.1)}$$

$$\text{Weight percent of gas and oil} = \frac{A-B}{A} \times 100 \quad \text{.....(3.2)}$$

When A and B were determined as weight of EVA/NR before and after thermal conversion, respectively, and C is weight of gas and oil.

Oil fraction can be calculated from the weight after subtracting weight of dichloromethane as solvent. Finally, gas fraction can be carried out by substracting weight of oil from the weight of gas and oil (C).

For comparison, EVA/NR was operated in an electrical furnace (Figure 3.8) using same quartz reactor, quantity, reaction time, and atmosphere. The temperature was set at 700°C and EVA/NR was heated to this temperature at the maximum heating rate. Thermal conversion products were collected and analyzed in the same manner as microwave heating experiments.

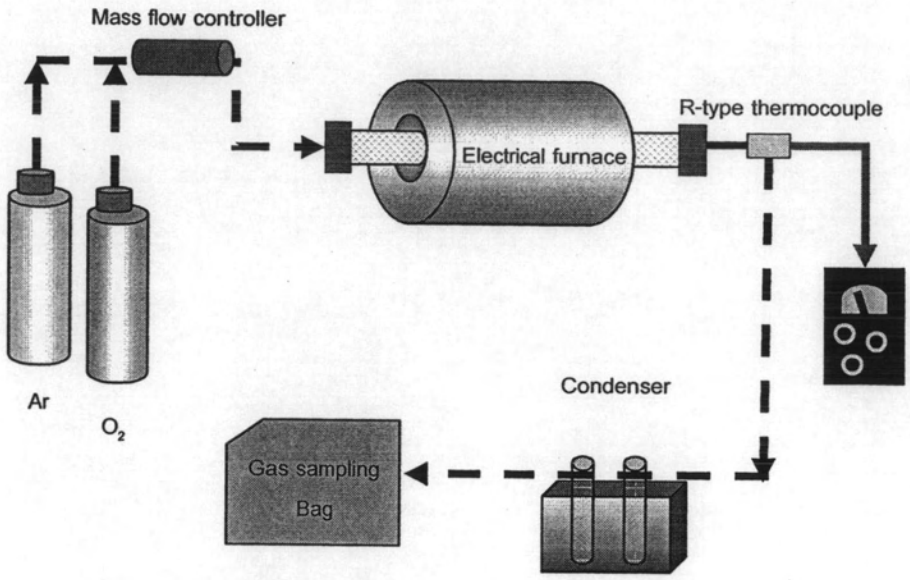

Figure 3.8 Operation of thermal conversion by electrical furnace

3.4.3 Characterization of gas products from thermal conversion

Gas products from the experiments were characterized by Gas Chromatography/Flamed Ionized Detector/Thermal Conductivity Detector. Species and content of gas were evaluated by standard mixed gas. The characterization by Gas Chromatography was run with following conditions.

Injector	=	35°C for 5 minutes
Column oven	=	50°C for 5 minutes
		75°C for 15 minutes with heating rate 10°C/min
		100°C for 10 minutes with hating rate 10°C/min
Flow rate	=	4°C/min

The species of hydrocarbon gas (C_1-C_5) and permanent gas (H_2, CO_2, and CH_4) were determined from standard mixed gas. Content of permanent gas were calculated from peak area of standard gas and plot as standard curve, next the peak area of gaseous products can be used to calculated the concentration by using the standard plot. For hydrocarbon gas (C_1-C_5), the content was reported in term of weight percent from carbon content in EVA/NR which measured from elemental analysis. The products

from different condition were compared. Moreover, the influenced of heating system between microwave heating and electrical furnace on products distribution (solid, oil, and gas), species of gas (hydrocarbon and permanent gas), and yield of each gas products were investigated and compared with other literatures. Temperature of microwave heating was evaluated by R-Type thermocouple for studied heating rate of experiment.