CHAPTER IV

EXPERIMENT

4.1 Raw material and chemical

4.1.1 All of plastics, polypropylene polyethylene and polystyrene used in this experiment were supplied from Thai Petrochemical Industry Public Company Limited. It was virgin plastic of 3-4 mm. diameter pellets.

4.1.1.1 Polypropylene 's specification: Gas phase process

Properties	Value
1. Melt flow index 230/216	23-25 g/10 min
2. Ethylene content	3-4%
3. Notched impact strength at 23^{0}C	> 3 mJ/mm^2
4. G-modulus	> 400 N/mm^2
5. Hardness	> 50 N/mm^2
6. Yield stress	> 23 N/mm^2
7. Elongation	> 32 %
8. Xylene soluble	7-11 %
9. Melting point (^{0}C)	< 145

4.1.1.2 Polystyrene's specification: Bulk polymerization

Properties	Value
1. Melt flow index 230/216	8.12 g/10 min
2. Color	blue
3. Haze (Turbidity)	1.9 %
4. Tensile strength	437 Kg*cm/cm
5. Tensile modulus	3.3 x 10^4 Kg/cm^2
6. Vicat softening temp.	97.8 ^{0}C

4.1.1.3 Polyethylene's Specification: Bulk polymerization (LDPE)

Properties	Value
1. Melt flow index	7.5 g/10 min
2. Density	0.917 g/cm^3
3. Elongation at break	> 600 %
4. Tensile strength at yield	> 10 N/cm^2
5. Tensile strength at break	> 10 N/cm^2
6. Vicat softening temp.	88 ^{0}C
7. Impact strength	> 150 g
8. Bulk density	0.52 g/cm^3

4.1.2 The palm-oil shells for the production of activated carbon were crushed and sieved to six particle sizes of < 0.6, 0.6-1.18, 1.18-2.36, 2.36-4.75, 4.75-6.00 and 6.00-8.00 mm before being treated.

4.1.3 Toluene 99.5%

4.1.4 Hydrogen gas 99.5%

4.2 Apparatus

The reaction was carried out by using apparatus shown in **Figure 4.1**. The microreactor is a stainless steel tube SS 316 with an inner volume of 75 cm^3, 30-mm inner diameter, showed in **Figure 4.2**. It was heated by electricity 450 watt, and the temperature was measured by thermocouple size 1.6-mm diameter. The temperature was controlled to an accuracy of +/- 5^{0}C by means of programmable temperature controller. Rotameter is used to control shaking of microreactor. The oil product was separated from solid by using the vacuum filter.

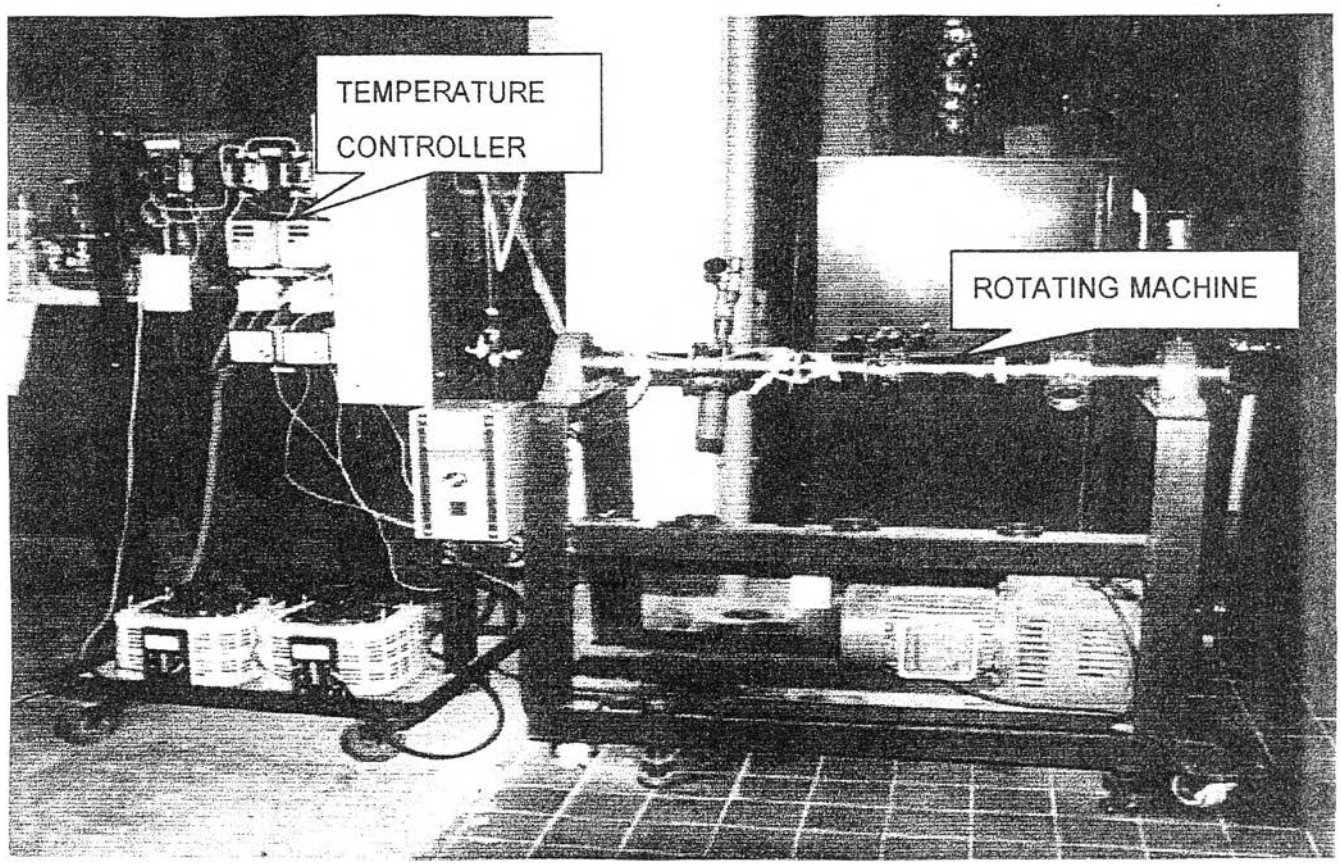

Figure 4.1 The reaction unit for conversion of mixed plastics into oil products by using Fe/Ac as catalyst

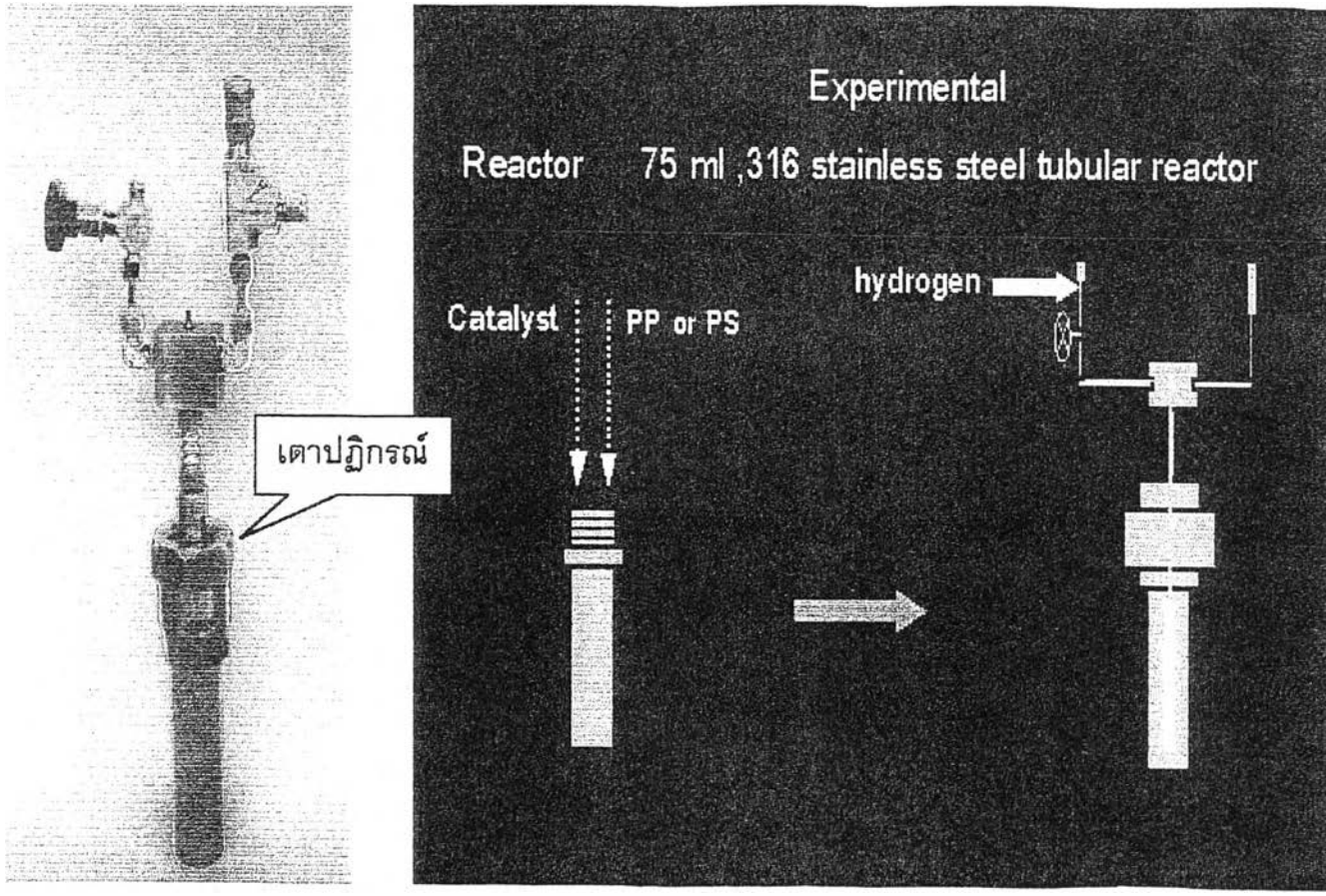

Figure 4.2 A set of microreactor

4.3 Processes of the experiment

4.3.1 The processes prepared for catalyst is as follows [13]:

- A preparation of iron on activated carbon from palm-oil shell catalyst (Fe/Activated carbon) has been done through impregnation method with different percentage of iron at 1, 5 and 10 %.

- About 200 g of each type of catalyst are prepared with the following steps:

1. Deaeration from 2 sizes of activated carbon 0.5 mm and 1.0 mm by injecting distillate water at an equal level of a height of activated carbon in a container before allowing it to enter in vacuum equipment for 3 hours and finally air bubble will be found on its surface.

2. Impregnation with aqueous solution of ferric nitrate($Fe(NO_3)_3$with required percentage of iron on deaerated activated carbon at room temperature for 2 hours.

3. Drying slurry with vacuum rotary evaporator to a temperature of 80 $^{\circ}C$ for 3 hours, and then dry and powdered activated carbon with $Fe(NO_3)_3$ which is in pore putting it in an for baking overnight to a temperature of 120$^{\circ}C$.

4. The next day, calcination activated carbon with ($Fe(NO_3)_3$ which is in pore to temperature of 450$^{\circ}C$ for 3 hours for driving off nitrate, oxide on surface of activated carbon will be yielded.

5. Reducing catalyst with hydrogen to a temperature of 400$^{\circ}C$ for 1 hour and refilling presulfation with hydrogen sulfide to a temperature of 450$^{\circ}C$ for 1 hour and keeping it cool at room temperature, catalyst usable for the intended purposes will be given.

4.3.2 Physical property of Fe/AC was bulk density, range of 0.425 to 0.455 g/cc, average surface specific area of 706 m^2/g.[13]

4.3.3 Procedure of the experiment for mixed polypropylene with polystyrene and mixed polypropylene with polyethylene were following:

- A preparation of oil product from mixed plastics using catalyst (Fe/Activated carbon) has been done through hydrocracking method with different condition.
- About oil product of each mixed plastic is prepared with the following steps:

1. After cleaning and drying the microreactor, weighing each plastic that used be material and catalyst into the reactor.
2. Close the reactor and input initial pressure of hydrogen gas. After that take the reactor to weigh again for finding total weight.
3. Install microreactor with the reaction unit and settled condition .

   3.1 Mixed plastics (polypropylene and polystyrene) conversion reaction was carried out under the following conditions:

   - Reaction temperature range of 390-435 ^{0}C
   - Pressure of hydrogen gas range 15-40 Kg/cm^{2}
   - Reaction time range of 30-75 min
   - Weight ratio of catalyst per 15 g of mixed plastics : 0.30, 0.45, 1.0 g
   - Percentage of Iron on activated carbon : 1, 5, 10%

   3.2 Mixed plastics (polypropylene and polyethylene) conversion reaction was carried out under the following conditions:

   - Reaction temperature range of 415-465 ^{0}C
   - Pressure of hydrogen gas range 25-40 Kg/cm^{2}
   - Reaction time range of 30-75 min
   - Weight ratio of catalyst per 15 g of mixed plastics : 0.30, 0.45, 1.0 g
   - Percentage of Iron on activated carbon : 1, 5, 10%
4. After reaction was end, cooled down the reactor to room temperature and then weighed it again.
5. Filtered the oil product from the reactor to find the fractions by GC simulated distillation.

The experiment scheme of the hydrocracking of mixed polypropylene with polystyrene and polypropylene with polyethylene by Fe/AC catalyst shown in figures 4.3 and 4.4 respectively.

4.4 Analysis by gas chromatography

The boiling points of oil yields from the reaction were analyzed by gas chromatography (GC Simulated Distillation) at PTT Research and Technology Institute. From the distillation result can be separated to the composition such as:

	Fraction	Boiling point (^{0}C)	Fuel applications
1.	Gas	< 20	Refinery fuel
			Liquefied petroleum gas
2.	Light gasoline	20-75	Gasoline blending
3.	Naphtha	75-200	feed stock in refinery plant
4.	Kerosene	200-250	Jet fuel
			Domestic fuel
			Tractor fuel
5.	Light gas oil	250-300	Diesel fuel
			Heating fuel
6.	Heavy gas oil	300-350	Heating fuel
7.	Long residues (Atmospheric residue)	>350	Heavy fuel oil
8.	% Solid		

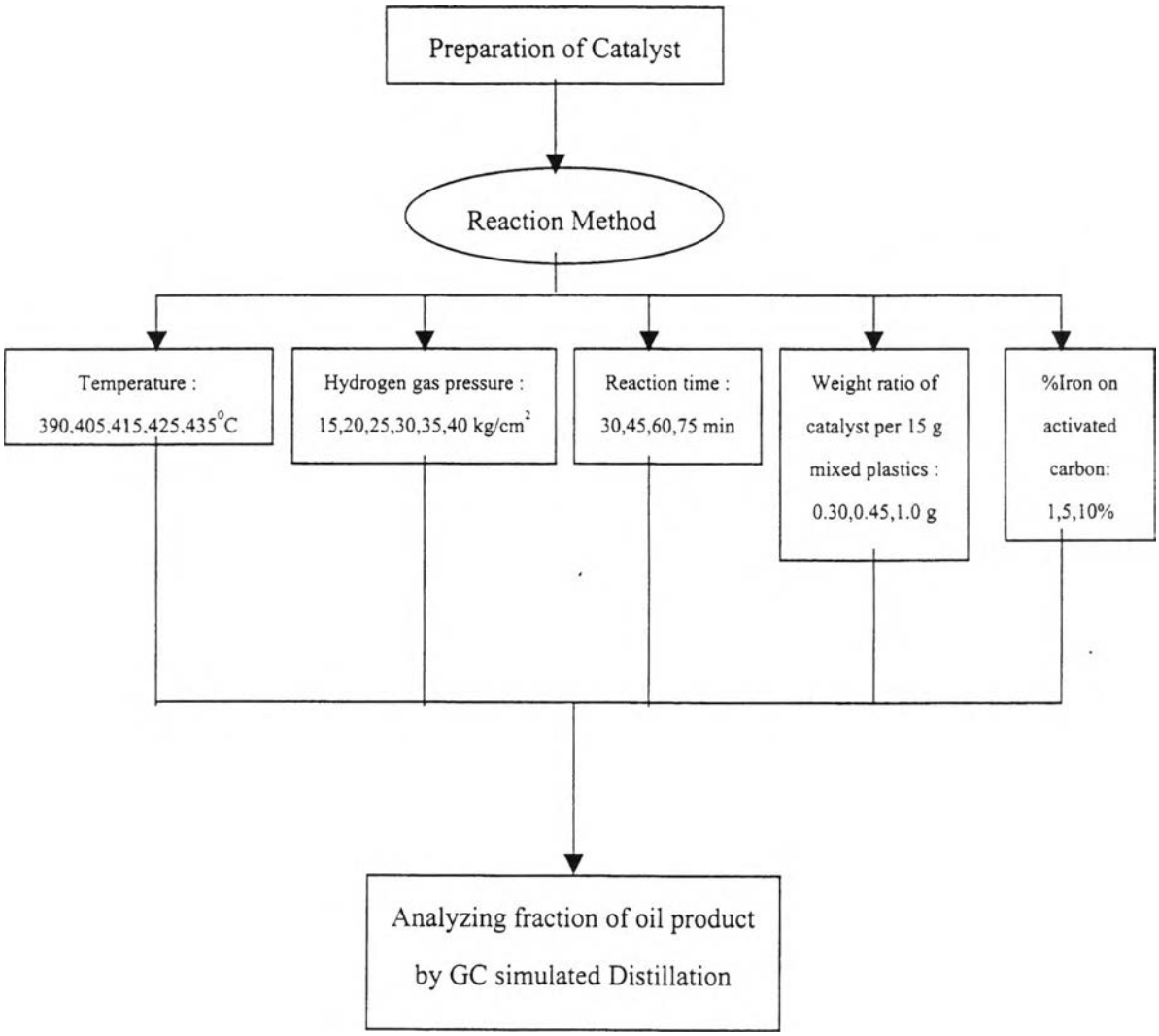

Figure 4.3 : Experiment scheme of the hydrocracking for mixed plastics of polypropylene and polystyrene by using Fe/Ac as catalyst

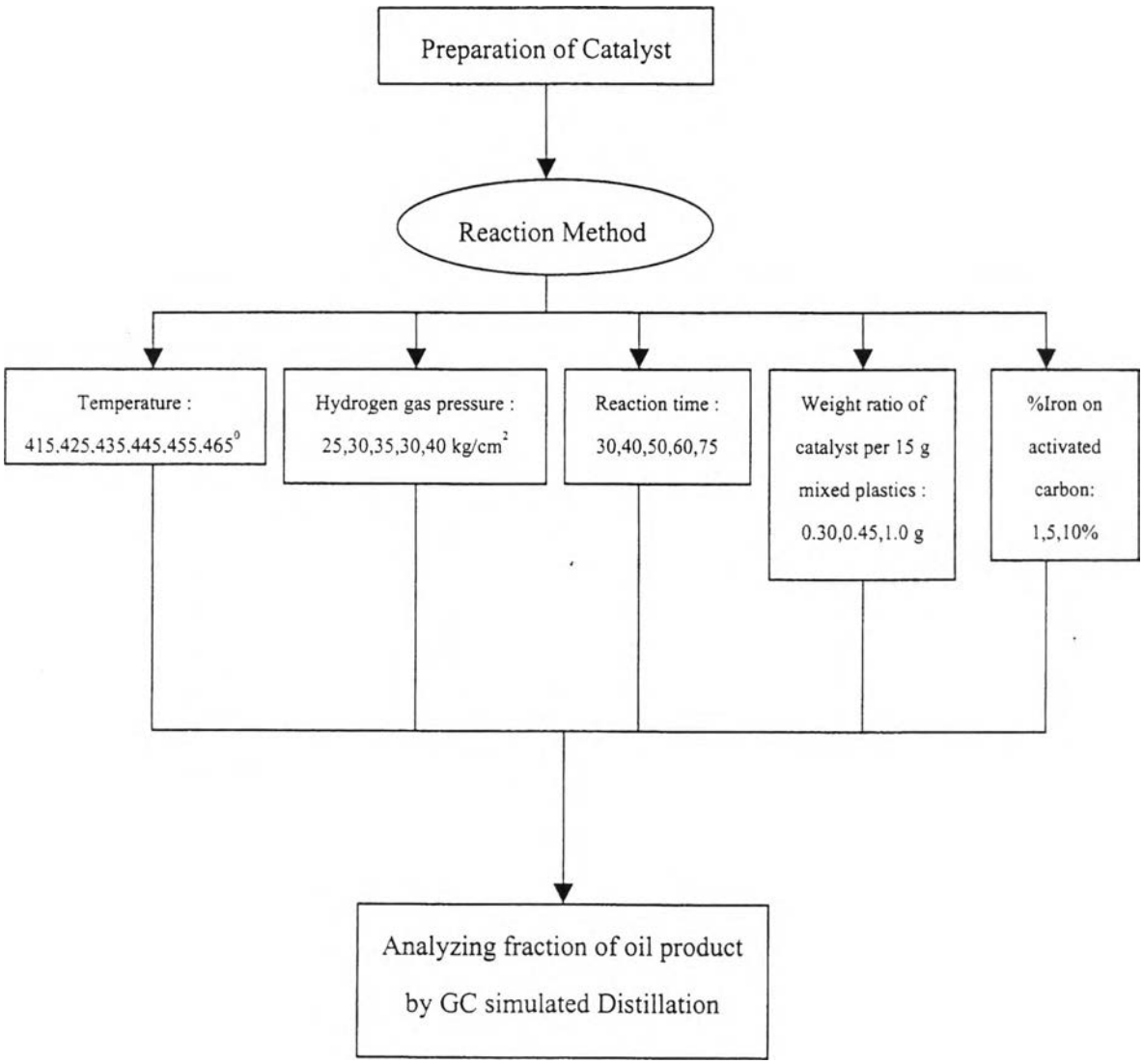

Figure 4.4 : Experiment scheme of the hydrocracking for mixed plastics of polypropylene and polyethylene(LDPE) by using Fe/Ac as catalyst