CHAPTER III
EXPERIMENTAL SECTION

3.1 Apparatus

1. A 2-litre jacketted stainless steel autoclave of Buichi A.G., model BEP 280 equipped with a variable speed motor and an anchor-type agitator.
2. Mass flow meter of Omega engineering Inc., model FMA-5610-ST. Ranged 0-10 standard litres per minute.
3. Mold temperature controller of Matsui, model MCO-15H.
4. Gel Permeation Chromatography (GPC) of Waters, model 150-C, for measuring the molecular weight of high density polyethylene (HDPE).

3.2 Raw Materials

Gas

- Ethylene (polymerization grade) supplied by National Petrochemical Public Co. was dried over molecular sieves (3 Å).
- Hydrogen (ultra high purity grade) supplied by Bangkok Industial Gas Co., Ltd. was used as received.
- Nitrogen (ultra high purity grade) supplied by Bangkok Industial Gas Co., Ltd. was used as received.

Catalyst

- $MgCl_2$-supported titanium halide catalyst
- Triethyl Aluminum

Diluent

- N-hexane supplied by Shell Chemicals Co., Ltd. was fractionally distilled before used.

3.3 Polymerization Procedure

The schematic diagram of polymerization reactor is shown in Figure 3.1. The following procedure was used for each experiment:

1. Check leak of reactor at 8 kg/cm^2.
2. Purge reactor with nitrogen for 1 hour. Set temperature at 80 ^oC, bubble hexane with nitrogen for 1 hour in cylindrical flask, and purge pipette with nitrogen for 10 minutes.
3. Cool down the reactor to ambient temperature.
4. When the reactor is slightly purged with nitrogen, open the feed port and feed 1 litre of hexane .
5. Pipette the desired quantity of triethylaluminum (under nitrogen) and feed into the reactor via the feed port.
6. Pipette the desired quantity of $MgCl_2$-supported titanium halide catalyst and feed into the reactor via the feed port.
7. Close the feed port. Keep the reactor pressure close to 0 kg/cm^2 and heat it up to the desired temperature.
8. Feed hydrogen at the desired temperature.
9. Feed ethylene at the desired pressure and start timer.
10. Polymerize for 2 hours at the desired conditions.

11. Cool down the reactor to ambient temperature.
12. Release pressure in the reactor, disconnect the reactor and then remove the powder slurry inside.
13. Filtrate the polymer slurry.
14. Dry high density polyethylene powder (HDPE) in an oven under nitrogen atmosphere and weigh it.
15. Evaporate the filtrate hexane to obtain the low molecular weight polyethylene (LMWPE).
16. Dry LMWPE in an oven under nitrogen atmosphere and weigh it.

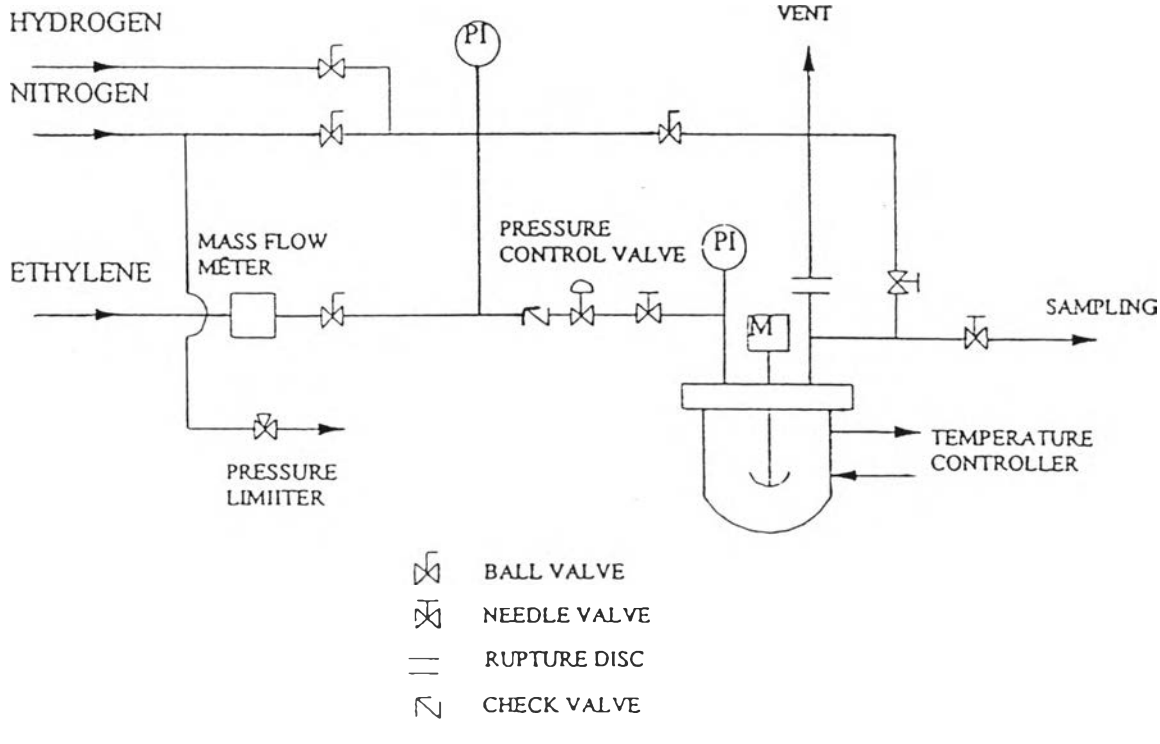

Figure 3.1 Schematic diagram of polymerization reactor.

3.4 Molecular Weight Determination

The molecular weight of HDPE samples was determined by Gel Permeation Chromatography (GPC) by the following procedure:

1. Dissolve 0.0075 gm. of HDPE samples in o-dichlorobenzene solvent of 5 ml.
2. Heat the solution samples at 145 ^{o}C and filter into vial and close with Teflon lid.
3. Take the solution samples to the GPC holding sample case.
4. Set GPC temperature at 145 ^{o}C and the flow rate of solvent at 10 ml/min
5. Start autoinject button of the GPC.

3.5 Variables

The effect of the following variables on the amount of HDPE and LMWPE produced was studied.

1. Agitator stirring speed (300-900 rpm.)
2. Partial pressure of hydrogen (H_2/C_2H_4 ratio 0.22-0.78)
3. Polymerization temperature (70-90 ^{o}C)
4. Alkylaluminum concentration (Al/Ti ratio 50-300)
5. Polymerization time (30-120 minutes)