CHAPTER III

EXPERIMENT

3.1 Materials

3.1.1 Polyethylene terephthalate (PET)

PET was obtained from post-consumer bottles. It was shredded to an average size of 5x5 millimetres, approximately.

3.1.2 Polyvinyl chloride (PVC)

An average dimension of 5 to 5 millimetres of PVC food-packaging was used.

3.1.3 Sodium hydroxide (NaOH)

NaOH was obtained from Carlo Erba.

3.1.4 Potassium hydroxide (KOH)

KOH was obtained from Carlo Erba.

3.1.5 Hydrochloric acid (HCl)

37% HCl was obtained from J.T.Beaker.

3.1.6 Water

Deionized water was used.

3.2 Apparatus and Instruments

3.2.1 High Pressure Reactor

All experiments in this study was carried out in the apparatus which consisted of three parts as follows:

3.2.1.1 Reactor

The bench top reactor was a high pressure batch stirred autoclave model 4562 from Parr Instrument Company with a 600 ml stainless steel cylindrical bomb, split ring closures and a bomb heater. The working pressure must not exceed 3,000 psig and maximum operating temperature was 350°C

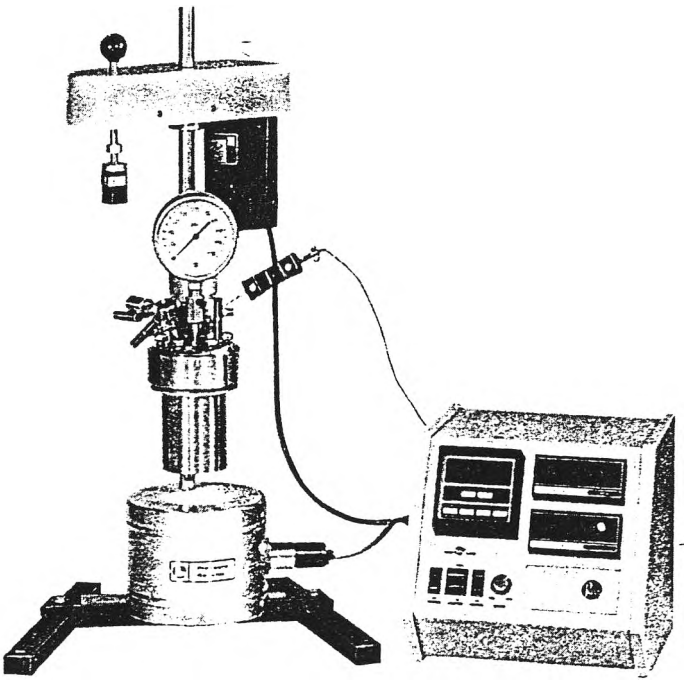

Figure 3.1 Reactor with Heater Removed.

3.2.1.2 Reactor Fitting

The stirring unit of reactor was equipped with convenient valves and fitting for handling the various functions. The parts are indicated below:

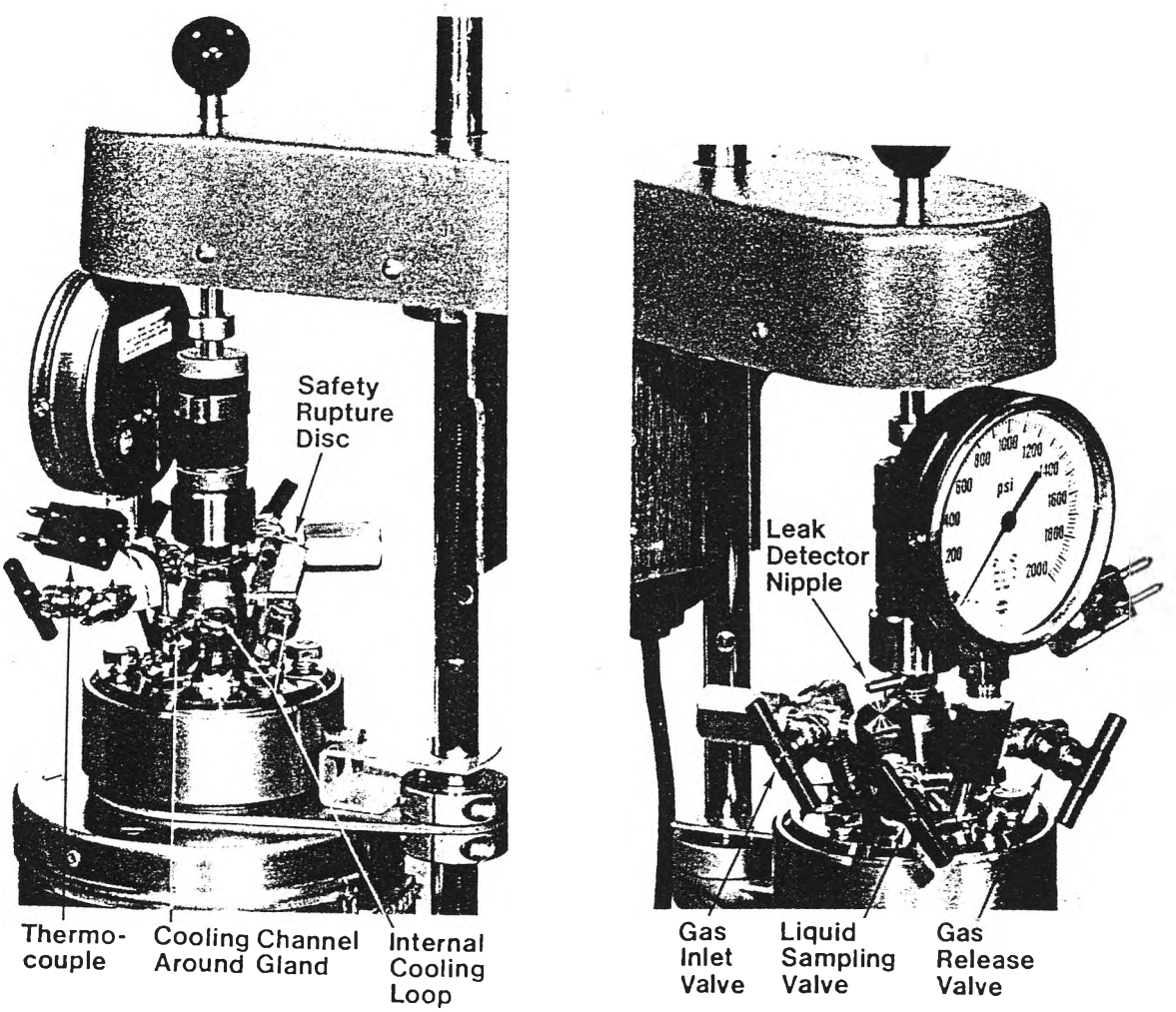

Figure 3.2 Reactor Fitting.

3.2.1.3 Temperature Controller

The controller model 4842 with Watlow Series 945, a microprocessor-based temperature control, from Parr Instrument Company was used. It was operated in conjunction with a type J thermocouple and included automatic LED indicators to aid in monitoring.

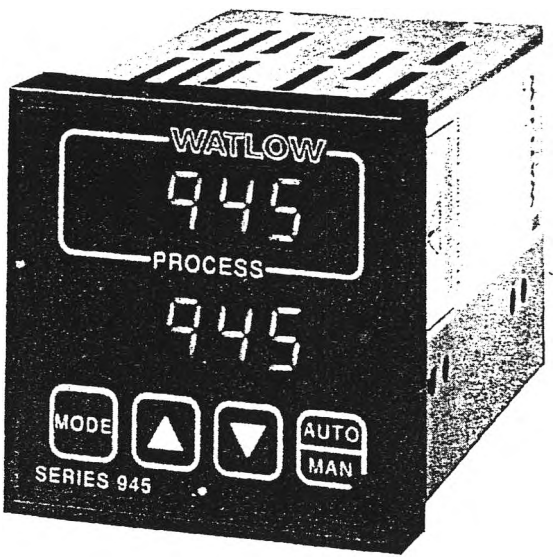

Figure 3.3 DIN Microprocessor-Based Auto-tuning Control.

3.2.2 Nuclear Magnetic Resonance Spectrometer (NMR Spectrometer)

The NMR model AC-F 200 from Bruker at 50 MHz for ^{13}C spectra was used.

3.2.3 Fourier-Transform Infrared Spectrometer (FT-IR Spectrometer)

The FT-IR model 1760x from Perkin Elmer was used.

3.3 Procedures

3.3.1 Procedure in study of saponification method for reclaiming of terephthalic acid

Fifteen grams of shredded used PET bottles and 50 ml of NaOH solution were placed in an autoclave. The stirrer was turned on, and the autoclave was heated to the desired temperature. The autoclave was pressurized to between 50 and 700 psig at the corresponding temperature. After stopping the reaction and cooling to ambient temperature, PET was converted into disodium terephthalate and ethylene glycol. The aqueous solution was then passed through a filter in order to remove any undissolved impurities.

Hydrochloric acid was then added to the filtrate until the pH was less than 7. TPA was recovered as the precipitate. This precipitate was removed using a filter and washed with 1,000 ml of fresh water. The yield of TPA was determined by weight.

3.3.1.1 The effect of NaOH content

The optimum NaOH content was studied by this procedure. The content of NaOH was varied from 25, 40, 50, 60, 75, and 100 percent by weight for 15 grams of PET. The reaction temperature was 270°C (at 700 psig) and reaction time was 1 hour. The results are presented in Table 4.1.

3.3.1.2 The effect of temperature

The different temperatures, 150, 180, 210, 240, and 270°C, were employed with the optimum NaOH content and 1 hour reaction time. The reaction pressure was varied between 50-700 psig according to the temperature. The results are shown in Table 4.2.

3.3.1.3 The effect of time

The reaction time was set at 5, 15, 30, 45, and 60 minutes. Time at set temperature, not including heating and cooling time. Table 4.3 shows these results.

3.3.2 Procedure in study of neutral hydrolysis method for reclaiming of terephthalic acid

PET (15 grams) and deionized water (50 millilitres) were placed in the autoclave. The stirrer was turned on, and the autoclave was heated to the desired temperature. The autoclave was autopressurized between 200 and 700 psig at the corresponding temperature. When the reaction was finished, it was allowed to cool to ambient temperature.

TPA was reclaimed from the aqueous solution by filtration. The yield of terephthalic acid was determined by weight.

3.3.2.1 The effect of temperature

Various operating temperatures, 210, 240, and 270°C, for 15 minutes were used. The results are presented in Table 4.5.

3.3.2.2 The effect of reaction time

The reaction time was set at 1, 3, 5, 15, and 30 minutes with an optimum temperature. The results are shown in Table 4.6.

3.3.3 Procedure for terephthalic acid reclamation from mixed polyethylene terephthalate and polyvinyl chloride

The sample was prepared by mixing PET and PVC in the ratio of 100:0, 75:25, 50:50, and 25:75, respectively. All samples were studied by the two procedures as before at each optimum condition. The results for this study are presented in Table 4.7.

3.3.4 Product characterisation

Product from each procedure was characterised by various techniques as shown below:

3.3.4.1 Study of the functional group

TPA was prepared in a form of KBr pellet. The functional group of the sample was determined by FT-IR.

3.3.4.2 Study of the structure

Deuterated dimethyl sulfoxide (d_6-DMSO) was used to dissolve TPA. The structure of the sample was analysed by ^{13}C-NMR.

3.3.4.3 Study of acid number

0.5 grams of TPA was dissolved in 20 ml of 0.5N KOH. The sample was back-titrated with 0.5N HCl solution to reach the phenolphthalein end point. A blank containing the same amount of 0.5N KOH was also titrated. Acid number was calculated in milligrams of KOH per gram of sample as follows:

$$\text{Acid number} = [\,(B-A)\,N \times 56.1\,] / W$$

where : A = HCl solution required for titration of the sample, ml.
B = HCl solution required for titration of the blank, ml.
N = Normality of the HCl solution, and
W = Sample used, grams.