CHAPTER 3

EXPERIMENTAL

3.1 Materials

Materials used in the present studies are given in Table 3.1. All materials are of the same lot throughout the experiment.

Table 3.1 Materials used in the present study

Material	Tradename /Grade	Manufacturer
High density polyethylene (HDPE)	RR1760	Thai Petrochemical Ind. Co., Ltd.
Natural Rubber (NR)	STR 5L	RRIT
Compatibilizer Epolene wax (EPW)	E-43p	White Group Co., Ltd.
Blowing agent Azodicarbonamide (AZDC)	AZ-H	Lautan Otsuka Chemical Co., Ltd.
Activator Zinc oxide (ZnO)	-	Global Chemical Co., Ltd.
Cross-linking agent Sulphur (S)	-	Ajax Chemical Co., Ltd.

More details of these materials can see in appendix A.

3.2 Instruments

3.2.1 Two-roll mill : Labtech Model LP 110

- Roll diameter x length 15 x 38 cm
- Roll speed 24 rpm
- Friction ratio 1:1.14

3.2.2 Press for compression moulding : Labtech Model LP 20

- Size of plates 20 x 20 cm
- Maximum pressure 160 kg/cm^2
- Heating system electric heater
- Cooling system water

3.3.3 Mould

The stainless steel mould dimension is 15 x 15 x 0.3 cm

3.3.4 Crushing machine : Bosco

3.3.5 Thermogravimetric analyser : Netzsch Model STA 409

3.3.6 Dynamic Mechanical Analysis: Perkin Elmer DMA 7e

3.3.7 Universal testing machine : Hounsfield Model H10KM

3.3.8 Optical microscope : Olympus Model BH-2

3.3.9 Scanning electron microscope : Jeol Model JSM-6400

Table 3.2 Steps and preparative conditions of HDPE/NR blends in foaming process.

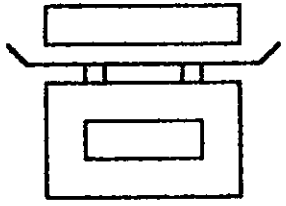
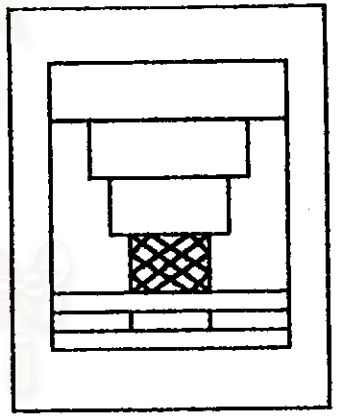
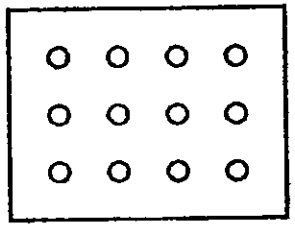
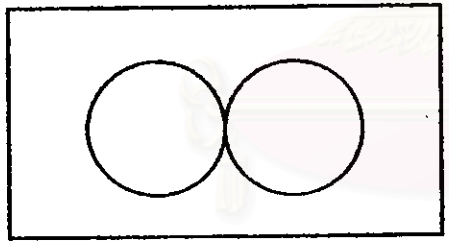
Composition	Mixing			Foaming	Sample Appearance
	stage 1	stage 2	stage 3		
HDPE NR EPW AZDC ZnO 	HDPE NR EPW	AZDC ZnO	S (where needed)		
	 Two roll mill ● Temp. 150 °C ● Nip ~1 mm			Compression moulding ● Temp. 170 °C ● Pressure 120 kg/cm^2 ● Time 20 min	

Table 3.3 Formulation of polymer blends of HDPE/NR compounds.

HDPE/NR	EPW (pphp)	AZDC (pphp)	ZnO (pphp)	S (pphp)	Time (min)	Test series
80/20	2	2	0.2	-	10	
80/20	2	2	0.2	-	15	
80/20	2	2	0.2	-	20	A
80/20	2	2	0.2	-	25	
80/20	2	2	0.2	-	30	
80/20	2	1	0.1	-	20	
80/20	2	2	0.2	-	20	
80/20	2	3	0.3	-	20	B
80/20	2	4	0.4	-	20	
80/20	2	5	0.5	-	20	
90/10	2	2	0.2	-	20	
80/20	2	2	0.2	-	20	
70/30	2	2	0.2	-	20	C
60/40	2	2	0.2	-	20	
50/50	2	2	0.2	-	20	
70/30	2	2	0.2	0.2	20	
70/30	2	2	0.2	0.4	20	
70/30	2	2	0.2	0.6	20	D
70/30	2	2	0.2	0.8	20	
70/30	2	2	0.2	1.0	20	
90/10	2	2	0.2	0.6	20	
80/20	2	2	0.2	0.6	20	
70/30	2	2	0.2	0.6	20	E
60/40	2	2	0.2	0.6	20	
50/50	2	2	0.2	0.6	20	

A: for the experiments of the effect of heating time

B: for the experiments of the effect of blowing agent loading.

C: for the experiments of the effect of HDPE/NR ratio.

D: for the experiments of the effect of cross-linking agent loading.

E: for the experiments of the effect of HDPE/NR ratio at a fixed cross-linking agent loading

3.3 Foaming Process

Table 3.2 gives steps and preparative conditions of HDPE/NR blends in the foaming process used in the present studies. The detailed procedure is as follows :

1. Formulation

High density polyethylene, natural rubber and other ingredients were measured according to the formulations in Table 3.3

2. Mixing and milled sheet

Mixing was carried out in a two-roll mill at the set temperature at 150°C , using a 3-stage mixing.

Stage 1 : Mixing of HDPE, NR and EPW for 3 minutes.

Stage 2 : adding AZDC, ZnO and mixing for 5 minutes.

Stage 3 : adding Sulphur and mixing for 3 minutes.

The HDPE/NR compound was sheeted out and milled in a crushing machine to give a small pieces of sheet.

3. Foaming (Single stage process)

The milled sheet of HDPE/NR compound having a weight of 55 grams was put in the mould of size15 x 15 x 3 cm. The filled mold was pressed between plates of the hot press at 170 °C. The pressure of 120 kg/cm^2 was applied for 10-30 minutes.

3.4 Physical Measurements

3.4.1 Determination decomposition of temperature of the blowing agent

The decomposition temperature of the blowing agent was determined by means of an apparatus shown in Figure 3.1

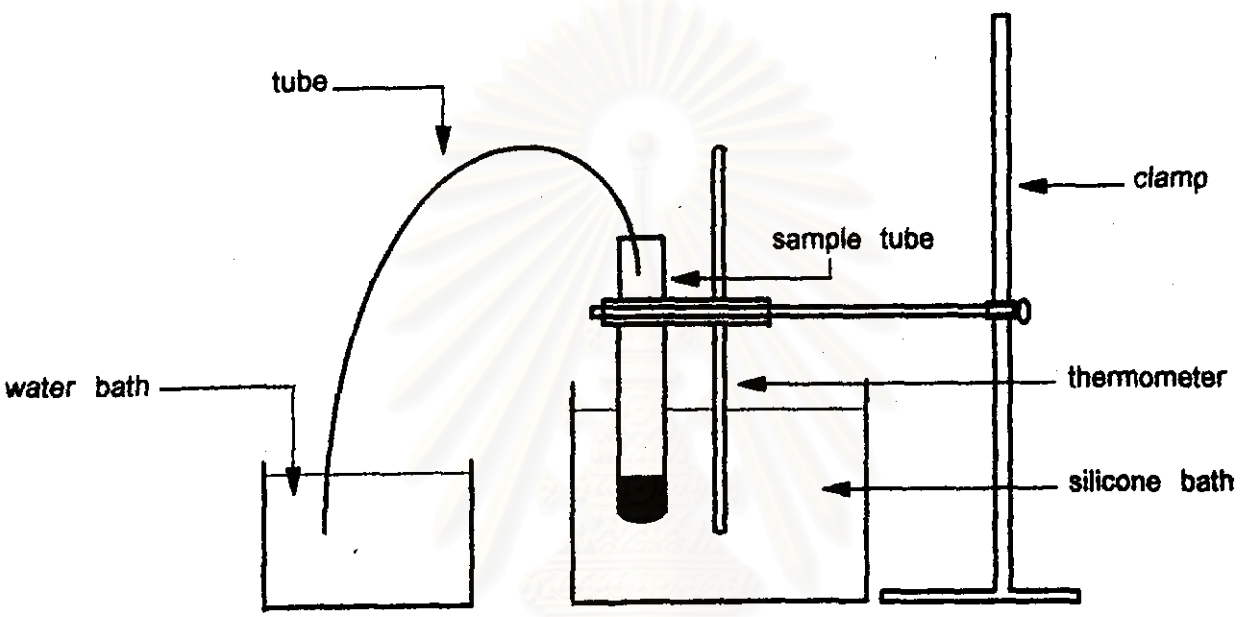


Figure 3.1 Equipment setup for decomposition temperature measurement.

The blowing agent alone and in the presence of various amounts of activator were put in the sample tube and heated in the oil bath. The decomposition temperature of the blowing agent was read from a thermometer at maximum volume of the evolved gas that replaced the volume in water.

3.4.2 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was carried out for the blowing agent alone and in the presence of activator. The sample formulated was prepared by a simple dry-blending of various amounts of the activator. The sample weight was 10 mg and the heating rate used was 5 °C/min.

3.4.3 Dynamic Mechanical Analysis (DMA)

Dynamic mechanical measurements of HDPE/NR foam were done with the specimens as a rectangular sheet having the dimension as shown in Figure 3.2.

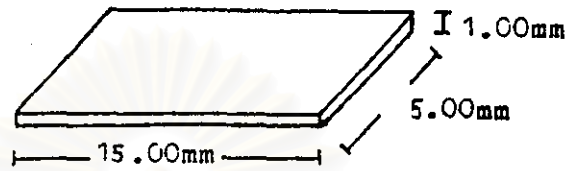


Figure 3.2 Rectangular sheet.

The three point bending mode was used to determine the storage modulus, loss modulus, and tanδ (loss modulus/storage modulus). The samples were tested at a frequency of 1 Hz. The heating rate of the samples was 5°C per min from -120 to 150°C. Liquid nitrogen was used to cool down the sample to sub-ambient temperature.

3.3.4 Physical Property Measurements

1. Density

The density of the foam samples was determined from the foam weights in air and water, according to ASTM D 792 method A.

2. Gel content measurement[2]

For measuring the degree of cross-linking, the foam sample having the weight of 1 gram was soaked in toluene for 48 hours at room temperature and the dried in an oven at 110 °C for 24 hours. The deswollen weight was recorded. The gel content was calculated from the following equation:

$$\text{Gel content} = \frac{\text{the deswollen weight} \times 100}{\text{the original weight of sample}} \qquad (3.1)$$

3. Hardness

The hardness of the foam samples was measured by Durometer hardness type A, according to ASTM D 2240.

4. Tensile measurement

The stress-strain properties comprising tensile strength and elongation at break were measured using a Universal testing machine, Hounsfield model H10KM, in accordance with the test procedure of ASTM D 638. A crosshed speed was 50 mm/min.

5. Tear strength measurement

The tear strength of the foam samples was measured according to ASTM D 624. Die C was used to cut the test specimen. Measurements were made on the Hounsfield mechanical tester model H10KM using the test speed of 500 mm/min at room temperature.

6. Flexural measurement

The flexural properties comprising flexural strength and elastic modulus were measured according to ASTM D 790 method I. The test specimens were rectangular. Measurements were made on the Hounsfield mechanical tester model H10KM using the test speed of 1.71 mm/min and a span length of 64 mm at room temperature.

3.4.5 Morphological studies

The morphological appearance of polymer blends was elucidated using Scanning Electron Microscopy (SEM). Since polymer blends of HDPE and NR cannot be easily identified under SEM, therefore, the NR was etched with toluene for 5 hours. The etched surface was dried at 80°C in oven for 4 hours in order to remove the excess solvent. Then they were stained with osmium tetraoxide (OsO_4) to increase the contrast between plastic and rubber phase. They were then coated

with gold prior to observation under SEM. And the uniformity of NR in HDPE phase was determined by a "width parameter" program of the optical microscope.

3.4.6 Characterization of cell structure of foam

The cell structure of HDPE/NR foam was studied using the optical microscope (Olympus Model BH) at 40 X magnification. The specimens were sliced by microtoming. The sliced of sample was slightly stained with a refill blue ink before examination to achieve adequate contrast. The stained samples were photographed using the CCD camera of the microscope.

The average cell size, cell size distribution (as measured by the standard deviation of cell size), maximum cell size, and minimum cell size were recorded by a "cir parameter" program of the optical microscope. The "cir parameter" program measured a diameter of cells by click three point on the circumference of the cell and then the "cir program" will create a circle and calculate diameter of the cells. Each of samples was measured 200 cells.

3.5 Flow chart of the whole experiment

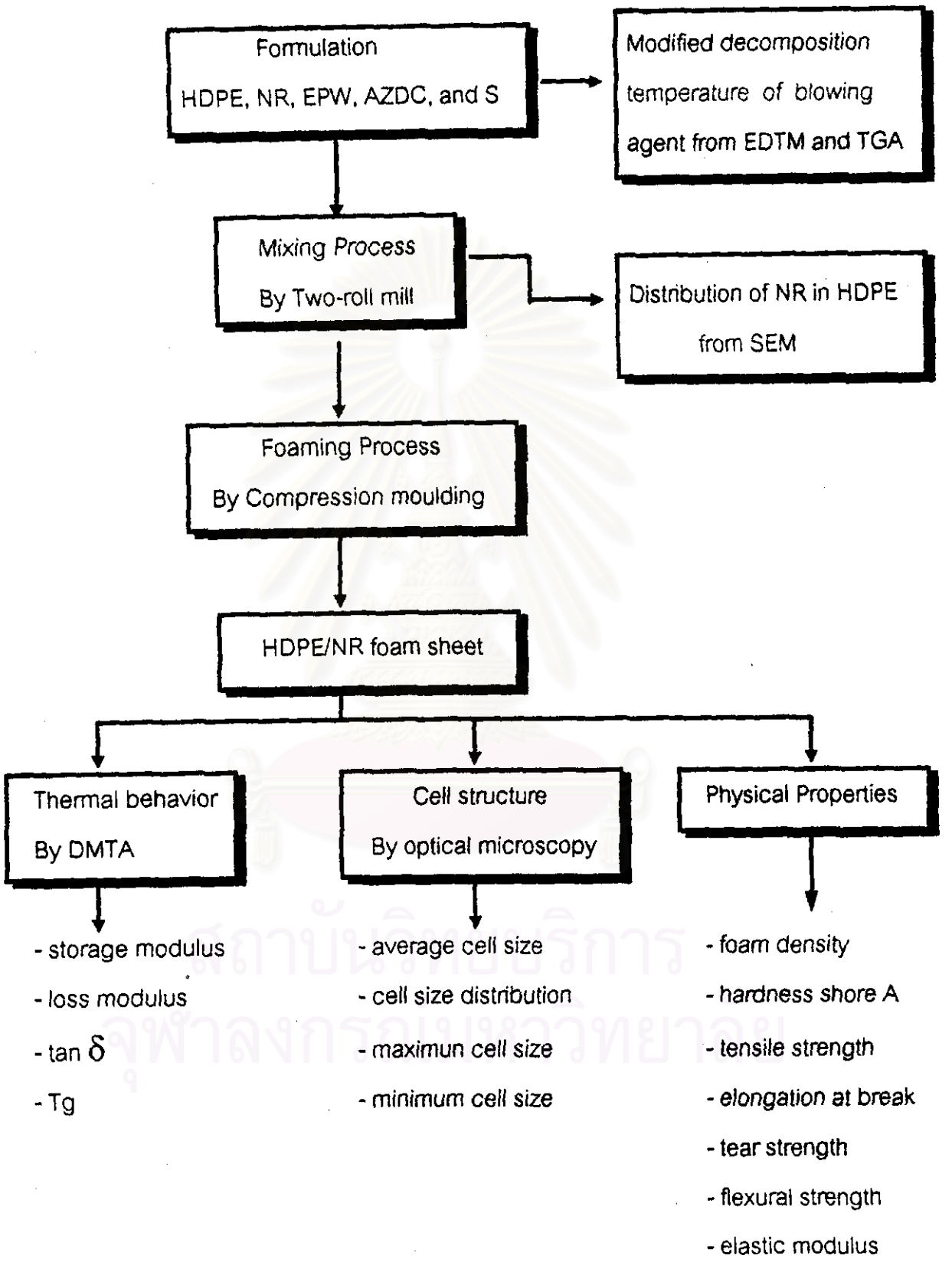


Figure 3.3 Flow chart of the whole experiment