

CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1. Natural rubber latex	: Thai Rubber Latex Corporation
2. Carbon black	: Thai Carbon Product Co. Ltd.
3. Sulfur	: Ajax Chemicals
4. Stearic acid	: Ajax Chemicals
5. Zinc oxide (ZnO)	: Ajax Chemicals
6. 2-Mercaptobenzothiazole (MBT)	: Fluka
7. Tergitol NP 9	: Union Carbide
8. Tergitol NP 10	: Union Carbide
9. Sodium dodecyl sulfate (SDS)	: Fluka
10. Formic acid	: Ajax Chemicals
11. Acetic acid	: Merck
12. Toluene	: Carlo Erba

3.2 Instruments and Apparatus

1. Ball mill
2. Mastersizer : Malvern Instruments Ltd.
3. Optical Microscope : OLYMPUS BX 60M
4. Scanning Electron Microscope : JSM-5800LV
5. Rheometer : Gibitre Rheocheck OD 2000
6. Universal testing machine : Instron Corporation Series IX
7. Hardness testing machine : The Shore Instrument & MFG.Co.Inc.
8. Two-roll mills : Lab Tech Engineering Co. Ltd.

3.3 Procedures

3.3.1 Analysis of Concentrated Natural Rubber Latex

Total solids (ASTM D 1076)

Natural rubber latex was weighed (2.5 g) in a dish. Then the cover was removed and the latex was distributed over the bottom of the dish (about 32 cm²). Distilled water was added (1 cm³) to the latex and the dish was gently swirled. The specimen was dried in an oven for 16 hours at 70 °C, cooled in a desiccator to room temperature, and weighed. The percentage of total solids is calculated in Appendix A.

Dry Rubber Content (ASTM D 1076)

Natural rubber latex was weighed (10 g) in a dish. Distilled water was added until the total solids content was approximately 25%. Acetic acid (2% by volume) was added while stirring constantly over 5 min. Then the dish was placed on a steam bath and left undisturbed for 15 to 30 min. The coagulated latex particles were picked up with the main body of the coagulum. The coagulum was washed with running water and passed between rolls. This process was repeated 5 times and reduced the sheet of coagulated rubber to a maximum thickness of 2 mm and dried at 70 °C for 16 hours. The coagulated rubber was cooled in a desiccator to room temperature and weighed. The dry rubber content is calculated in Appendix A.

Particle Size

The particle size analysis was determined by the Mastersizer and the average diameter and size distribution of rubber particle is shown in Appendix B.

3.3.2 Preparation of Natural Rubber Product Containing Carbon Black and Vulcanizing Agents

The natural rubber latex containing carbon black and vulcanizing agents was prepared by ball mills. All compounding ingredients were used without further purification. Aqueous dispersion of carbon black, sulfur, stearic acid, zinc oxide and MBT were prepared by ball mills under room temperature. The rate of ball mills was 130 rpm. After the required mixing time, concentrated natural rubber latex was added,

the compounding was mixed for 0.5 hours. The compounded rubber latex was coagulated by 5% formic acid by volume. The compounded sheet was washed with running water and passed between rolls. The compounded sheet was dried at 60 °C for 24 hours.

The compounded sheets were prepared and subjected to various investigation as following.

1. Effect of surfactant concentration

The aqueous dispersions of the additives were prepared by ball mill for 2 hours. The surfactant was used by changing the concentration of 1, 2, 3, and 4 phr. The formulation of compounded sheets are shown in Table 3.1.

Table 3.1 Compounded sheets formulations

Ingredient	Quantity of mix (phr)
Concentrated natural rubber latex	100 parts by dry weight
Filler : carbon black (N330)	25
Vulcanizing system:	
Sulfur	2
MBT	1
Stearic acid	2
ZnO	5
Surfactant : Tergitol NP 10	1,2,3,4

2. Effect of mixing time

The mixing time was studied by varying the time of 1, 2, 3, 4, and 5 hours so as to attain the appropriate time. The formulations of compounded sheets were same as Table 3.1.

3. Effect of carbon black, vulcanizing system and types of surfactant

The aqueous dispersions of the additives were prepared at appropriate time. The formulations of compounded sheets were changed as shown in Table 3.2.

Table 3.2 Formulations to study effect of carbon black, vulcanizing system and types of surfactant

Ingredient	Type	Quantity of mix (phr)
Concentrated natural rubber latex	-	100
Filler : carbon black	N220, N330	25,35,45
Vulcanizing system :		
Sulfur	-	1,2,3,4
MBT	-	0.5,1,2
Stearic acid	-	1,2,4
ZnO	-	2,5,10
Surfactant	Tergitol NP9, NP10	3
	SDS	1.5

4. Effect of mixing type

The two-roll mills was used to prepare the compounded sheets. The formulations in Table 3.2 which had good properties were used.

3.3.3 Determining Selected Vulcanization Characteristics of Vulcanized Rubber Compounds

The Gibitre Rheocheck OD 2000 was used to determine the vulcanization characteristics of vulcanizable rubber compounds. The time required to obtain a cure curve was a function of the characteristics of rubber compound and of the test temperature.

testing condition :

Temperature	150 °C
Testing time	24 min
Rotational amplitude	3°

3.3.4 Preparation of Vulcanized Sheets (ASTM D 3182)

Preparation of Sheets

The compounded sheets were placed on a flat, dry, clean metal surface. Then the pieces were cut (4.5 ± 1.5 mm) shorter in width and length than the corresponding dimensions of the mold cavity.

Vulcanization Procedure

The vulcanized sheets were prepared by the press. The mold was brought to curing temperature (150 °C) in the closed press and held at this temperature for at least 20 min before the unvulcanized pieces were inserted. The press was opened, then the unvulcanized pieces were inserted into the mold and closed the press in the minimum time possible. When the mold was removed from the press to insert the pieces, the mold was then taken precautions to prevent excessive cooling by contact with cool metal surfaces or by exposure to air drafts. The mold was held under a pressure of 120 kg/cm² (1700 psi) on the cavity areas during vulcanization. After the required vulcanization time, the press was opened. The vulcanized sheets were removed from the mold and cooled on metal surface which used water cooling for 10 to 15 min. The vulcanized sheets were conditioned at 23 ± 2 °C for at least 16 hours before preparing and testing.

3.4 Characterization of Dispersions

3.4.1 Determination of the Degree of Dispersion

The dispersion ratings (DR.) of carbon black were determined by microscopic examination of freshly razor-cut surfaces of the vulcanized sheets.

Preparation of the Test Piece

The vulcanized sheets were cut by the sample cutter with a cross section of approximately 5×8 mm. The surface that used for rating was not touched by anything. The razor blade was replaced before the edge wears such that the cut became lined.

Microscopic Examination of the Freshly-cut Vulcanized sheets

The surfaces were examined by using dark-field illumination from directly above, at a magnification of $100\times$. The diagram of optical dark field microscopy is shown in Figure 3.1

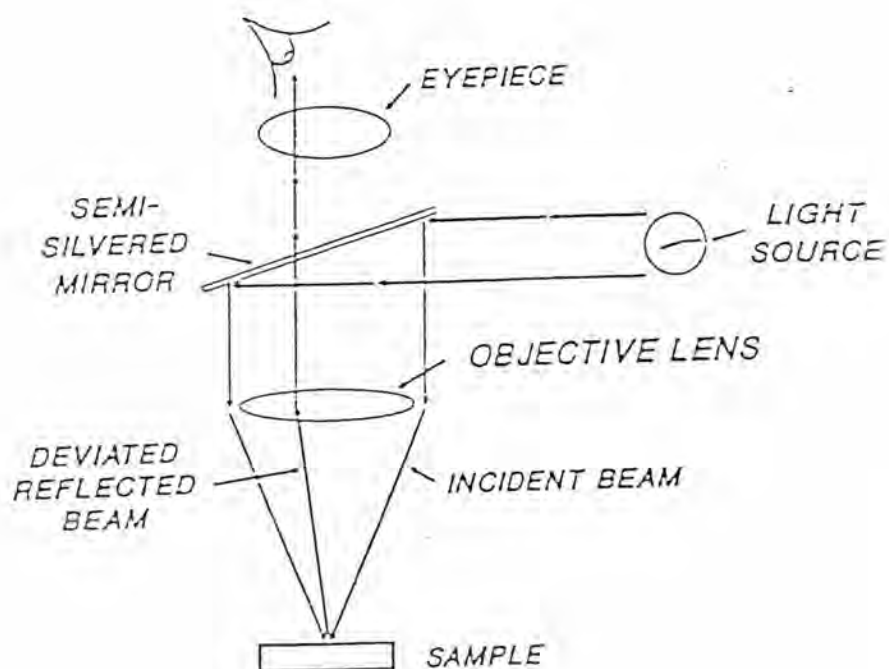


Figure 3.1 Schematic diagram of optical dark field microscopy

An Olympus BX 60 M with an Olympus PM-20 exposure control unit was used. Photographs of the prepared surface of all of the compound were obtained. These were compared to the standard photographs of ISO 11345 (Method A). The compounded sheets were assigned the most closely matched numerical rating. For close matching, the fractional ratings were used. For example 5 ½ would indicate a rating between 5 and 6.

3.4.2 Determination of the Dispersion by Scanning Electron Microscope

A scanning electron microscope (SEM) Model JSM-5800LV was used to determine the dispersion of some vulcanized sheets.

3.5 Mechanical Testing

Mechanical properties of the vulcanized rubbers were measured by following the ISO and the ASTM test methods as follows:

ISO 37 : Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties

The tensile strength, the elongation and the stress at a given elongation (modulus) were determined.

The test specimen dimension (type 2) is shown in Figure 3.2.

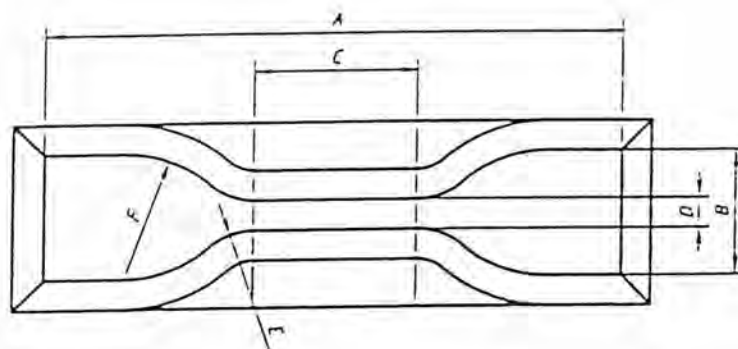


Figure 3.2 Schematic of tensile test specimen (type2)

Dimension	Type 2 (mm)
A Overall length (minimum)	75
B Width of ends	12.5 ± 1.0
C Length of narrow portion	25.0 ± 1.0
D Width of narrow portion	4.0 ± 0.1
E Transition radius outside	8.0 ± 0.5
F Transition radius inside	12.5 ± 1.0

The tensile testing condition

Temperature	25 °C
Humidity	50 %
Test length of dumb-bells	20.0 ± 0.5 mm
Crosshead speed	500.0 mm/min
Full scale load range	5.0 kN

ISO 34: Rubber, vulcanized or thermoplastic – Determination of tear strength.

Part 1 : Trouser, angle and crescent test pieces

The value of tear strength obtained depended on the shape of the test specimen, speed of stretching and temperature of test. In this experiment, the vulcanized sheets were used method B, procedure (a) that used an angle test specimen without nick.

The test specimens were cut from vulcanized sheets of uniform thickness. The dimension and the shape of specimen is shown in Figure 3.3.

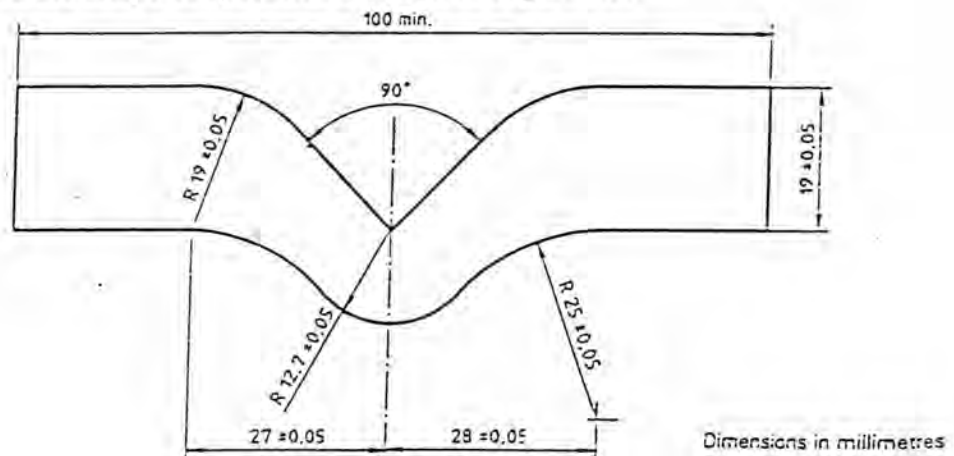


Figure 3.3 Schematic of angle test specimen

The tear testing condition

Temperature	25 °C
Humidity	50 %
Crosshead speed	500.0 mm/min
Full scale load range	5.0 kN

ASTM D 2240: Standard test method for rubber property – Durometer hardness

In this experiment, vulcanized sheets were used type A durometer. The shape of the indenter is shown in figure 3.4.

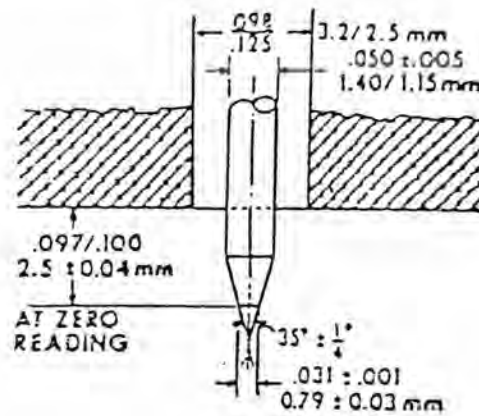


Figure 3.4 Indenter for type A durometers

The test specimen was at least 6 mm. in thickness unless it was known that results equivalent to the 6 mm. values were obtained with a thinner specimen. The minimum requirement for the thickness of the specimen was depended on the extent of penetration of the indenter into the specimen. The lateral dimensions of the specimen was measured at least 12 mm from any edge. The surface of the specimen was flat and parallel over a sufficient area to permit the presser foot to contact the specimen.

3.6 Solvent Swelling

A test piece was cut from vulcanized sheets and weighed about 0.20 g. Then it was immersed in a large excess of toluene (300 ml) for three days. After the desired time, it was taken out, loose liquid was rapidly removed by blotting with filter paper and the swollen weight was immediately measured. The test piece was subsequently deswollen and reweighed.