

## CHAPTER III

### EXPERIMENTAL SECTION

#### 3.1 Materials

Natural rubber (NR) used for the experiment was STR 5L grade, purchased from Rayong Bangkok Rubber Co., Ltd.. Three different carbon blacks were used in this study:

1. N110 use as received from J.J. Degussa Co., Ltd., with a specific surface area of  $151\pm 6$  m<sup>2</sup>/g
2. N220 use as received from J.J. Degussa Co., Ltd., with a specific surface area of  $121\pm 5$  m<sup>2</sup>/g
3. N330 use as received from Mahidol University, with a specific surface area of  $82\pm 5$  m<sup>2</sup>/g

Three different amorphous precipitated silicas without any surface treatment were used in this study:

1. Hi-Sil 927 use as received from PPG-Siam Silica Co., Ltd., with a specific surface area of  $179\pm 4$  m<sup>2</sup>/g
2. Hi-Sil 255 use as received from PPG-Siam Silica Co., Ltd., with a specific surface area of  $175\pm 15$  m<sup>2</sup>/g
3. Ultrasil VN2 use as received from J.J. Degussa Co., Ltd., with a specific surface area of  $105\pm 20$  m<sup>2</sup>/g

Gypsum powders were 75 grade, use as received from Thai Gypsum Co., Ltd. Zinc oxide (ZnO) and stearic acid used as activators. 2,2-Dibenzothiazyl disulfide (MBTS) used as accelerator. Sulfur (S) used as vulcanizing agent. All of these materials use as received from the Rubber

Based Industry Research and Development Center, Rubber Research Institute of Thailand. The density of the materials used are given in Table 3.1.

**Table 3.1** Density of the materials.

<b>Material</b>	<b>Density (g/cm<sup>3</sup>)</b>
NR	0.90
Carbon Black	1.80
Precipitated Silica	2.00
Gypsum	2.32
ZnO	5.54
Stearic Acid	0.94
MBTS	1.54
S	2.08

## 3.2 Instruments

### 3.2.1 Two-Roll Mill

A Lab Tech LRM 110 two-roll mill was used for masticating NR and preparing rubber compound for compression at room temperature.

### 3.2.2 Brabender Mixer

The materials were mixed at room temperature in the internal mixer, Brabender Plasti-corder PL 2000. The mixer consists of a mixing chamber containing the counter rotating rotors. The rotor type N50, contains in the mixing chamber volume of 80 cm<sup>3</sup>. The batch size was adjusted to a fill factor of 0.80 for rubber (total = 72 g.). The rotor speed was set at 40 round per minute (rpm).

### 3.2.3 Rheometer

The gum rubber compounds were measured scorch time ( $t_2$ ) and cure time ( $t_{90}$ ) by a Rheo Tech rheometer (model TPD 1498) at 150 °C. The test was conducted according to ASTM D 2084-95.

### 3.2.4 Compression Molding Machine

The rubber compounds obtained from mixing processes were put into the mold such as a picture-frame and cylindrical molds which were made from stainless steel and normal steel coating with chromium. The molds were pressed at 150 °C with 10 tons force for a certain cure time ( $t_{90}$ ) and cut for dumbbell specimens for tensile testing and in cylindrical specimens for abrasion testing in the Wabash V50H Compression Molder.

### 3.2.5 Dynamic Mechanical Spectrometer

The dynamic properties of filled NR vulcanisates were carried out on Rheometric Scientific RHIOS instrument using a parallel plate (plate diameter = 2.5 mm) geometry operated in a torsion mode (shear). Frequency sweep curves were obtained between 0.1-100 rad/sec at strain amplitude 0.1% and the results were obtained at temperature 60 °C. It was always verified that the behavior of the sample was in the range of linear viscoelasticity.

### 3.2.6 Tensile Testing

Tensile strength at break, elongation at break and modulus of filled-rubber compounds were measured using an Instron Universal Testing Machine (model 1011) in accordance with ASTM D 412-98. The dumbbell specimens used were 25 mm in gauge length, 25 mm in width and 3.0 mm in thickness. The crosshead speed was set at 500 mm/min with load cell 5 kN. The test was operated at room temperature (25 °C).

### 3.2.7 Hardness Testing

The test for hardness was measured following ASTM D 2240 by using a Shore-type-A Lever Loader (model 716). The test temperature is carried out at 25 °C.

### 3.2.8 Rebound Resilience Testing

The rebound resilience is determined by measurement of energy absorbed by the rubber test piece during impact. It was measured by using Wallace Donlop Tripsometer (pendulum). The cylindrical specimens used were 4.0 mm in thickness and 4.5 mm in diameter. The test was operated according to an ISO 4662 test procedure at room temperature (25 °C).

### 3.2.9 Abrasion Testing

The abrasion in mm<sup>3</sup> is the volume loss under a defined test piece traversed through a defined abrasion distance under a specified contact pressure across an abrasive cloth. It was measured using Happen Abrasion Tester and the test was conducted according to DIN 53516 at room temperature (25 °C). The cylindrical in shape of specimens used were 16 mm in diameter and have a minimum thickness of 6 mm.

## 3.3 **Experimental Procedures**

### 3.3.1 Filler Preparation

The type of fillers used are given in Table 3.2. All fillers were dried in oven at 100 °C for 24 hours to remove the moisture. After that, fillers were stored in a desiccator before compounding.

**Table 3.2** Type of filler.

<b>Filler</b>	<b>Grade</b>
Carbon Black	N110
	N220
	N330
Silica	Hi-Sil 927
	Hi-Sil 255
	Ultrasil VN2
Gypsum Powder	75

### 3.3.2 Filler Characterization

#### 3.3.2.1 *Surface Area and Pore Volume*

The surface area and pore volume of dried fillers were determined by N<sub>2</sub> gas absorption technique using the Brunauer Emmett Teller (BET) procedure with Micromeritics FlowSorb II 2300 instrument.

#### 3.3.2.2 *Mean Agglomerate Particle Size*

The dried fillers were measured mean agglomerate particle size by Malvern Mastersizer instrument at room temperature (25 °C).

### 3.3.3 Sample Preparation

The rubber compound formula used in this study is presented in Table 3.3 to Table 3.6. The compounds were prepared by melt-mixing technique. All fillers were dried at 100 °C for 24 hours, immediately before use. First the NR was masticated for 10 minutes on a temperature controlled two-roll mill (Lab Tech LRM 110) at room temperature. The masticated NR, fillers and vulcanising system were mixed at room temperature in a Brabender mixer at rotor speed using 40 round per minute (rpm), but the



**Table 3.4** Formulation of rubber compound with fillers in volume fraction\*

<b>Material</b>	<b>Mix 1</b>	<b>Mix 2</b>	<b>Mix 3</b>	<b>Mix 4</b>	<b>Mix 5</b>	<b>Mix 6</b>	<b>Mix 7</b>	<b>Mix 8</b>
NR	100	80	80	80	80	80	80	80
Gypsum	-	20	-	-	-	-	-	-
N330	-	-	20	-	-	-	-	-
N220	-	-	-	20	-	-	-	-
N110	-	-	-	-	20	-	-	-
VN2	-	-	-	-	-	20	-	-
Hi-Sil255	-	-	-	-	-	-	20	-
Hi-Sil927	-	-	-	-	-	-	-	20
Zinc oxide	-	-	-	-	-	-	-	-
Stearic acid	-	-	-	-	-	-	-	-
MBTS	-	-	-	-	-	-	-	-
Sulfur	-	-	-	-	-	-	-	-
Total volume	100	100	100	100	100	100	100	100

\* In this thesis, the volume fraction is calculated only from the volume of NR and the volume of each filler and does not include the volume of the other additives such as zinc oxide, stearic acid, MBTS and sulfur. (See in Appendix C).

**Table 3.5** Formulation of rubber compound with mixed filler in part per hundred rubber (phr).

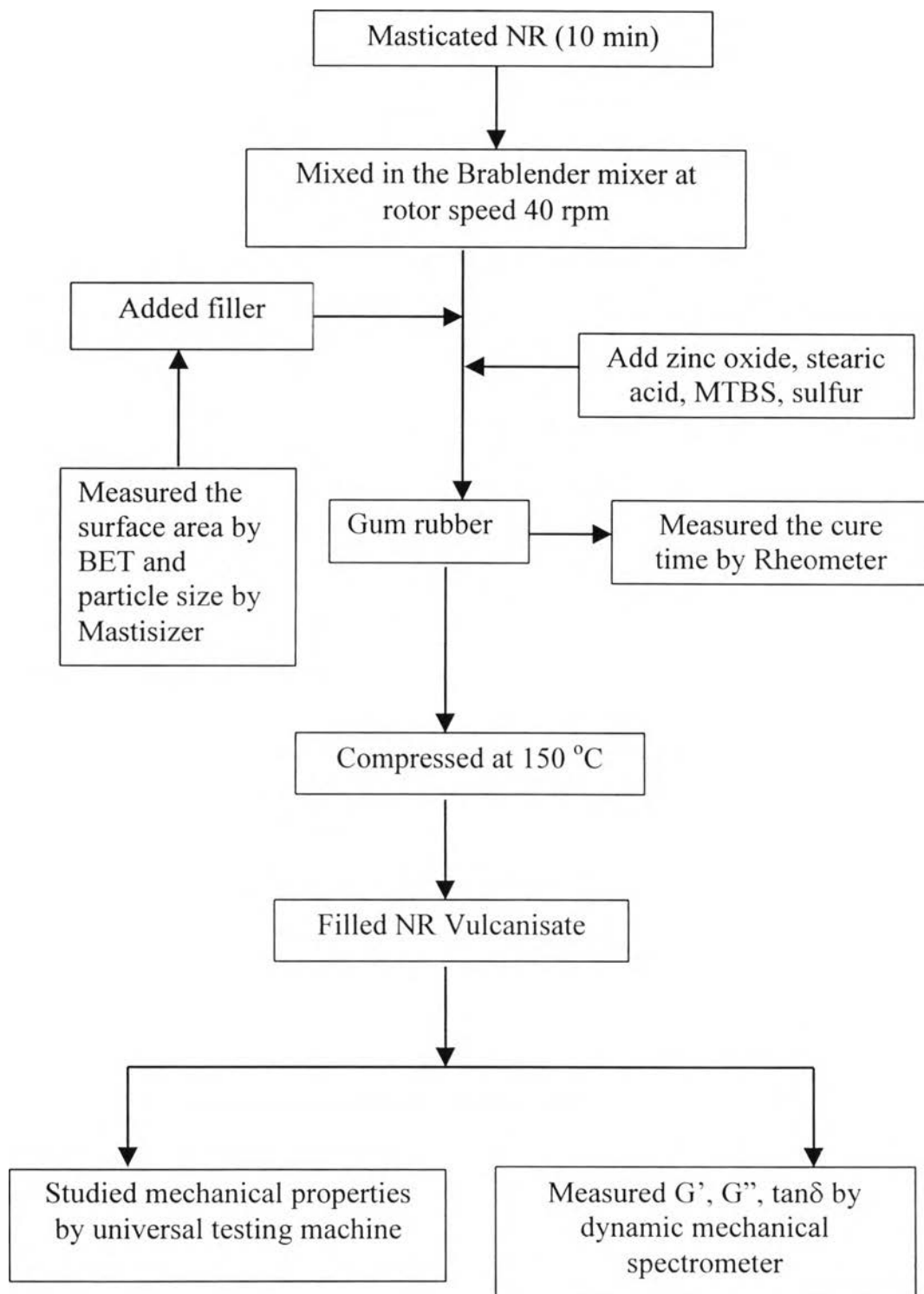
<b>Material</b>	<b>Mix 9</b>	<b>Mix 10</b>	<b>Mix 11</b>	<b>Mix 12</b>
NR	100	100	100	100
Gypsum	10.7	22.4	5.3	10.7
N330	-	-	-	-
N220	-	-	-	-
N110	38.1	27.3	38.1	27.3
VN2	-	-	-	-
Hi-Sil255	-	-	-	-
Hi-Sil927	-	-	4.5	9.3
Zinc oxide	5	5	5	5
Stearic acid	2	2	2	2
MBTS	1.8	1.8	1.8	1.8
Sulfur	3	3	3	3



**Table 3.6** Formulation of rubber compound with mixed filler in volume fraction.\*

<b>Material</b>	<b>Mix 9</b>	<b>Mix 10</b>	<b>Mix 11</b>	<b>Mix 12</b>
NR	80	80	80	80
Gypsum	4	8	2	4
N330	-	-	-	-
N220	-	-	-	-
N110	16	12	16	12
VN2	-	-	-	-
Hi-Sil255	-	-	-	-
Hi-Sil927	-	-	2	4
Zinc oxide	-	-	-	-
Stearic acid	-	-	-	-
MBTS	-	-	-	-
Sulfur	-	-	-	-
Total volume	100	100	100	100

\* In this thesis, the volume fraction is calculated only from the volume of NR and the volume of each filler and does not include the volume of the other additives such as zinc oxide, stearic acid, MBTS and sulfur. (See in Appendix C).



**Figure 3.1** A diagram of filled NR compound preparation.