CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1.	Natural rubber STR 5 L	: Thai Hua Rubber
2.	Zinc oxide	: Hsien and Industrial
3.	Stearic acid	: P.T. Cisadane Raya Chemicals
4.	Sulphur	: Utids Enterprise
5.	MBTS (2-2'- dithiobisbenzothaiazole)	: Cosan Co.,Ltd.
6.	DPG (diphenylguanidine)	: Cosan Co.,Ltd.
7.	Carbon black (N330)	: Siam Luck trading Co.,Ltd.
8.	Silica (Tokusil UR-T)	: Siam Luck Trading Co.,Ltd.
9.	2μ-calcium carbonate (Omyacarb 2)	: Surin Omya
10.	Uncoated nanocalcium carbonate	: B.R.S. Intertrade Ltd.,Part.
11.	Coated nanocalcium carbonate	: B.R.S. Intertrade Ltd.,Part.
12.	Toluene	: Shell (Thailand)

3.2 Equipments

1.	Two roll mill	: LabTech Engineering, Thailand
2.	Compression molding machine	: LabTech Engineering, Thailand
3.	Universal testing machine	: LLOYD LR 5K
4.	Hardness testing machine Shore A	: Societa per azioni Milano, Italia
5.	Mooney viscometer	: Shimadzu SMV-201, Japan
6.	Curelastometer	: Nichigoshoji II F, Japan
7.	Transmission Electron Microscope	: Hitachi TEM H-600A, Japan

3.3 Procedure

3.3.1 Characterization of Calcium Carbonate

The particle sizes of the fillers (nanocalcium carbonate and 2μ-calcium carbonate) were determined by transmission electron microscope (TEM). The nanoparticles were dispersed, using ultrasonic bath for 10 min.

3.3.2 Preparation of Natural Rubber / Nanocalcium Carbonate Composites

Natural rubber STR 5L (100 phr) was masticated for 5 min by the two-roll mill. Then activator additives such as zinc oxide (5 phr), stearic acid (1phr) and nanocalcium carbonate (uncoated surface) (0, 10, 20, 30, 40, 50 phr) was added. The rubber compound was mixed by cut-and-fold technique for 10 min until the surface of compound was smooth. The viscosity was measured by Mooney viscometer. The compound were mixed with MBTS (1 phr), DPG (0.5 phr) and sulphur (2.5 phr). The cure time of the sheet was determined by using curelastometer. The rubber compounds were then pressed into a preheated mold of compression molding at temperature of 150°C and pressure of 150 kg/cm^2.

After pressing, the rubber sheet of the specified size (Table 3.1) was transferred to a water cooled press for 5 min. The sheet was cut into the standard specimens according to the ASTM test method. The rubber sheet filled with fillers (2μ-calcium carbonate, nanocalcium carbonate (coated surface) and carbon black) were prepared in the same manner. The summarized procedure for preparation of natural rubber/ filler composite is shown in Figure 3.1. The compositions of compound formulation are shown in Table 3.2.

Table 3.1 Mould Dimensions

Properties	Mould dimension (mm) length x width x thickness
Tensile	160 x 160 x 2
Hardness	25 x 25 x 6

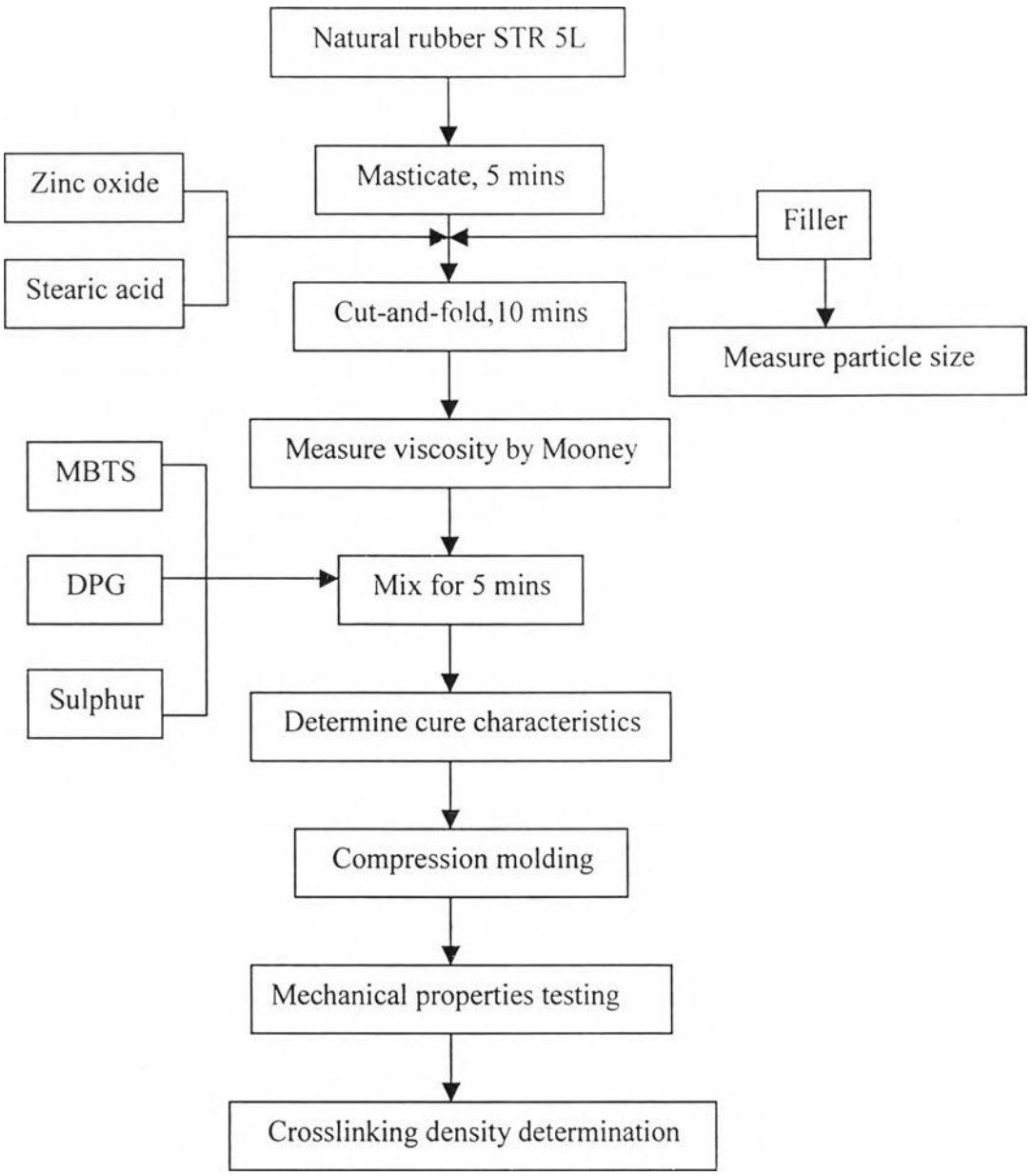

Figure 3.1 The Overall Schematic Experiment Process.

Table 3.2 Compound Formulation.

Ingredients	Part per hundred of rubber (phr)		
Natural rubber STR 5L	100	100	100
ZnO	5.0	5.0	5.0
Stearic acid	1.0	1.0	1.0
Sulphur	2.5	2.5	2.5
MBTS	1.0	1.0	1.0
DPG	0.5	0.5	0.5
2μ-calcium carbonate	10,20,30,40,50	0	0
Nanocalcium carbonate (Uncoated surface)	0	10,20,30,40,50	0
Nanocalcium carbonate (Coated surface)	0	0	10,20,30,40,50

3.4 Determination of Viscosity

Mooney viscometer (Shimadzu SMV-201) with large rotor was used to measure the viscosity. The test temperature was 100°C. The Mooney viscosity was determined according to ASTM D1646-87 and reported as (ML 1+4) in Mooney Unit. The Mooney viscosity curve is shown in Figure 3.2.

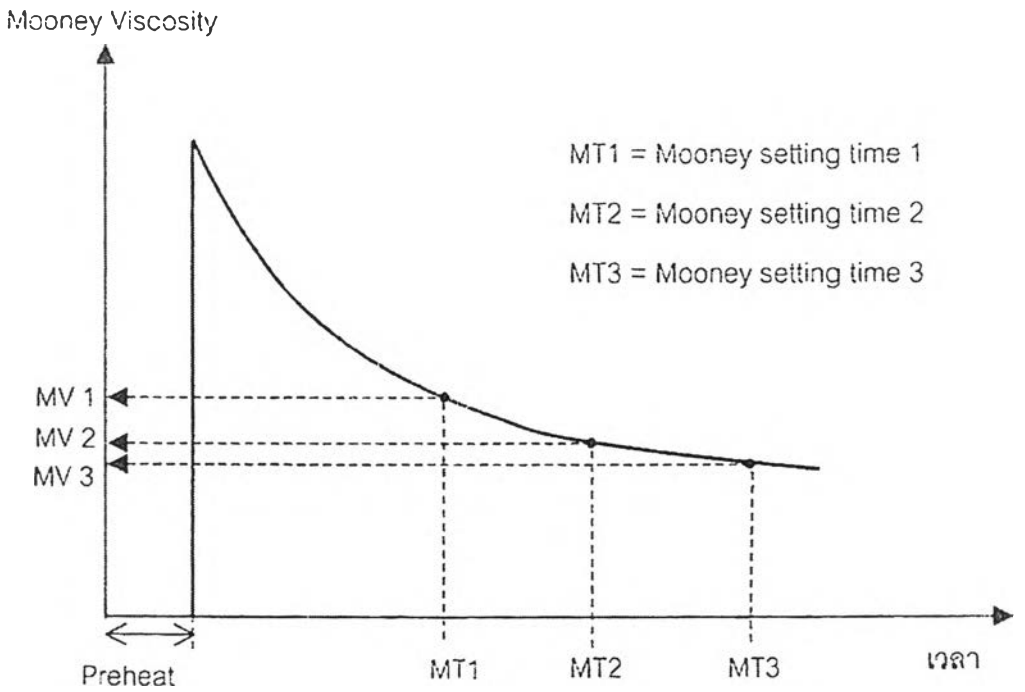

Figure 3.2 The Mooney Viscosity Curve of Rubber [18].

3.5 Determination of Cure Characteristics

Cure characteristics of rubber compounds were determined by using the Moving Die Rheometer (Nichigoshoji II F) at temperature of 150°C. In Figure 3.3, the main cure characteristics obtained are scorch time (t_2), optimum cure time (t_{90}), minimum torque (M_L), maximum torque (M_{HF}) and cure rate index (CRI) calculated from equation (3.1).

$$\text{Cure rate index (CRI)} = \frac{100}{(t_{90} - t_2)} \qquad (3.1)$$

where t_{90} = cure time

t_2 = scorch time

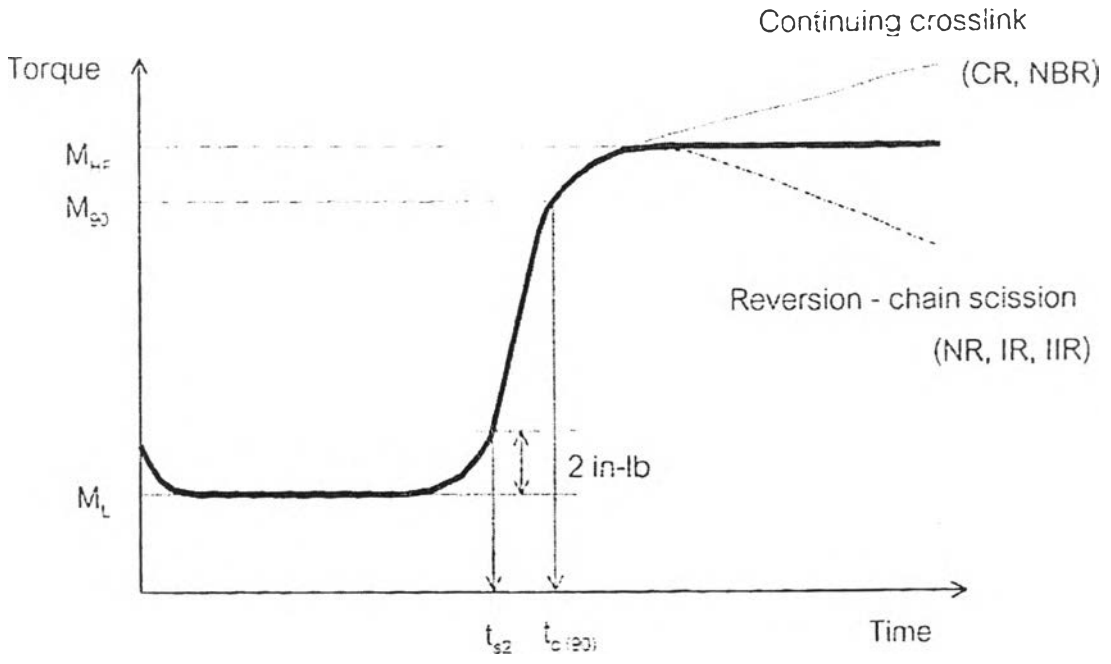

Figure 3.3 The Cure Curve of Rubber [18].

where M_L = minimum torque, lb_f *in

M_{HF} = maximum torque, lb_f *in attained during specified period of time when no plateau or maximum torque is obtained.

t_{s2} = time taken for a two-unit rise above the minimum, min.

$t_{c(90)}$ = time taken for attaining 90% of the maximum torque, min.

3.6 Mechanical Testing.

3.6.1 Tensile Properties (ASTM D 412)

Tensile strength, modulus and elongation at break were measured by using the Universal testing machine (LLOYD LR 5K) with a cross head speed of 500 mm/min. Vulcanized sheets were cut into the dumb-bell shape with the dimensions as shown in Figure 3.4.

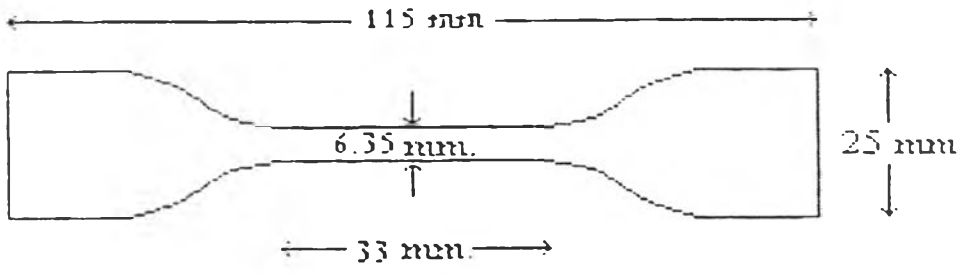

Figure 3.4 Dimensions of the Tensile Specimen [15].

3.6.2 Hardness (ASTM D2240)

The hardness of vulcanized sheets was measured at room temperature by using Societa per azioni Milano with Shore A hardness scale. The cylindrical shape samples (16±0.3 mm in diameter and 6 mm in thickness) were used for the test. The measurement was made according to ASTM D2240.

3.7 Determination of Specific Gravity

The vulcanized rubber specific gravity was determined by using Electronic Densimeter, MD-200S adapted from Archimedes' principle. The determination of (relative) density value was based on the density of water at 4°C: 1 g/cm^3 [19].

3.8 Crosslinking Density

Specimen of approximately 15 mm in diameter and 2.5 mm in thickness was allowed to swell in toluene. The density of each specimen was determined according to section 3.7. Specimen was then placed in a vial containing toluene. The vial was always kept covered to

prevent evaporation. Periodically, over a period of 1-3 days, the specimens were removed from the toluene, blotted dry on a paper towel, and then weighed quickly and accurately. The swelling index, which is defined as the grams of solvent per gram of rubber hydrocarbon, is calculated according to the Flory-Huggins Theory.