

CHAPTER III

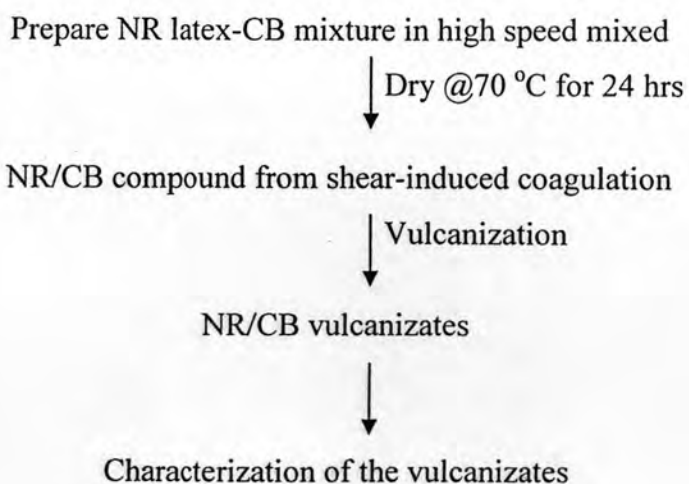
EXPERIMENTALS

3.1 Materials

Natural rubber latex was provided by Pan Asia Biotechnology Co., Ltd., The latex was characterized as Appendix A. The type is concentrated high ammonia (HA, 07%), having ~60% dry rubber content (DRC). Carbon black grade N220 (Intermediate super abrasion furnace, ISAF), N330 (High abrasion furnace, HAF) and N550 (Fast extruding furnace, FEF) were supplied by Thai Tokai Carbon Product Co., Ltd. Dried natural rubber, STR 5L, was supplied by Thai-Hua Chumporn Rubber Co., Ltd. DelacNS accelerator, N-tertiarybutyl-2-benzothiazole sulfonamide (TBBS), was supplied by Win Biz Co., Ltd. Zinc oxide (ZnO) white seal grade was provided by Uthid Enterprise Co., Ltd. Stearic acid was supplied by Siam Luck Trading Co., Ltd. Sulfur, used in powder form, was acquired from Shiraishi Calcium (Thailand) Co., Ltd.

3.2 Procedure

Working diagrams for this study:



3.2.1 Preparation of natural rubber/carbon black compound from shear-induced coagulation process

Concentrated NR latex was poured into a beaker and stirred by mechanical stirrer (Eyela Z1100) for evaporating ammonia until the latex pH decreased to 9.0 as determined by H+P Labortechnik (CG843). Filter the latex with gauze for separation of impurity. Demineralized water was added to dilute the latex in order to dilute the latex from 60% DRC to 30% with the purpose to reduce the latex viscosity. CB of the required amount was placed into a lab-scale high speed intensive mixer (Lab Tech LMX 5), followed by demineralized water. The high speed mixer was operated at 2,800 rpm for 2 minutes to obtain slurry. The latex was then mixed with the CB slurry and stirred at 2,800 rpm for 3 minutes to induce the coagulation of the latex-CB mixture. The solidified mixture was dried in an oven (Binder model FD115) at 70°C for 24 hour and at 100°C for 1 hour to remove water and ammonia retained in the samples.

3.2.2 Vulcanization of Natural rubber/carbon black composites

The formulation for sulfur cure is given in Table 3.1. The compound was mixed by a two-roll mill (Nishimura) at the temperature of 70°C. Before adding curing chemicals, a small quantity of NR compound was removed for Mooney viscosity analysis (see Mooney Viscosity section below). The cure characteristics of rubber compounds were determined with a rotorless curemeter (see Cure Characteristic section below). The curing was carried out in a compression molding machine with 15 MPa at 155°C for 90% cure time (T90) obtained from the curemeter add 1 minute. The vulcanized composite sheets with 2 mm. thick were obtained and stored in open air at 24°C for 24 hours for further test.

Table 3.1 Compound formulation for NR/CB compound obtained from shear-induced coagulation

Ingredient	Quantity (phr ^a)
NR/CB compound	100/CB variable (20, 30,40 and 50 phr)
ZnO	3
Stearic acid	1
DelacNS (TBBS)	1
Sulfur	1.75

^aphr is part per hundred parts of rubber by weight

Table 3.2 Compound formulation for solid NR

Ingredient	Quantity (phr ^a)
STR 5L	100
ZnO	3
Stearic acid	1
DelacNS (TBBS)	1
Sulfur	1.75
Carbon black (N330, N550)	Variable (20, 30, 40 and 50 phr)

3.2.3 Characterization of NR/CB compound

Mooney Viscosity

The NR/CB compound (before vulcanization) was cut into cylinder shape, diameter 5.5 cm about 25 g. It was then preheated at 100°C for 1 minute after the platens were closed before measuring its Mooney viscosity by using a Mooney viscometer (Monsanto Mooney MV2000). The rheological property was studied after rotating the disc for 4 minute with speed 1 s⁻¹ at 100°C by large rotor in accordance with ASTM D1646. The Mooney viscosity was then determined and expressed as ML1+4 (100°C)

Scanning Electron Microscope (SEM)

The NR/CB compound was passed through a two-roll mill for 10 cycles and then sheeted with nip size approximately 2 mm. The compound was frozen in

liquid nitrogen and rapidly fractured by using pliers. The cross-section of compound was assessed by using a scanning electron microscope (JSM-6480LV, JEOL) after coating the compound with sputtered gold for about 5 minute.

3.2.4 Characterization of NR/CB vulcanizates

Cure characteristic

Rotorless curemeter (Ektron EKP2000P) was run to determine the cure properties, e.g., cure time (T90), scorch time (T2), maximum torque (MH) and minimum torque (ML) of each NR/CB composite at 155°C.

Mechanical Properties

After the vulcanization, the tensile properties of rubber were measured on a tensile tester (Tech Pro, tensiTECH) according to ASTM D412 at a cross-head speed of 500 mm/minute of 1 kN at 24°C. The values reported for each sample were based on the average of five measurements. Dumbbell-shaped test pieces were punched out from a cutting die with the dimension according to ASTM D412. Tear properties were tested on the same machine according to ASTM D624 (die C) in the different dimension die. Tensile properties were measured along the grain direction whereas the tear properties were measured perpendicular to the grain direction. Hardness was measured using Durometer hardness system (Shore A) according to ASTM D2240. Measurements are made of five different points distributed over the specimen.