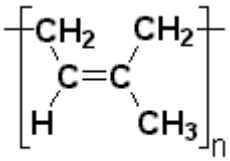
MATERIALS AND METHODS

Materials

1. Natural Rubber (NR); STR5L, S.M.P. RUBBER, Thailand.



cis-polyisoprene

Figure 13 Structure of Natural Rubber

2. Fillers
   - Natural zeolite; 69.8% SiO_2, Kasetcenter, Thailand.
   - Calcium carbonate ($CaCO_3$); C.P. Chemistry Industry, Thailand.
   - Rice Husk Ash (RHA); Phichit Power Plant.
   - Carbon black; N330, reinforcement filler, C.P. Chemical Industry, Thailand.
   - Ground Rubber; Siam United Rubber, Thailand.
3. Reagents
   - ZnO, an activator, Global Chemistry, Thailand.
   - Stearic Acid, an activator, Imperial Industrial Chemical, Thailand.
   - 2,2,4-Trimethyl-1,2-dihydroquinoline polymer (TMQ), an antioxidant, Eliokem, USA.

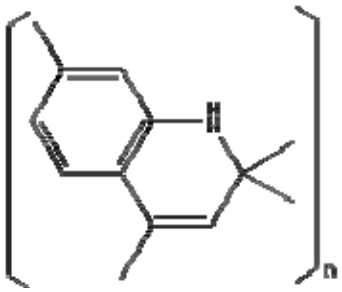


Figure 14 Structure of 2,2,4-Trimethyl-1,2-dihydroquinoline polymer (TMQ)

- Rubber oil, a processing aid, PSP Specialties, Thailand.
- N-cyclohexyl-2-benzothiazole sulphenamide (CBS), an accelerator, Flexsys, Germany.

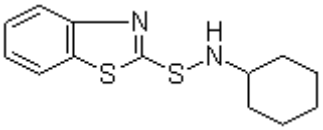


Figure 15 Structure of N-cyclohexyl-2-benzothiazole sulphenamide (CBS)

- Sulfur, a crosslink agent, Sahapaisal Industry, Thailand.

4. Equipments
   - Internal Mixer, Brabender plasticorder
   - Two-roll-mill, model LRM 150, Labtech
   - Moving Die Rheometer (MDR), Tech PRO
   - Compression molding, G30H-15-CX, Wabash, USA
   - Tear strength, 5569, Instron
   - Hardness, Cogenix, Wallace
   - Din abrasion, Materialprufung, Zwick
   - Electronic Densimeter, MD-200S, Mirage
   - Oven, Binder

Methods

1. Preparation of rubber compounds and vulcanizates

The mixing was carried out in both an internal mixer and a two-roll mill. All other ingredients, except the curatives (sulfur), were mixed with the natural rubber in Brabender plasticorder. Mixing was done at temperature of 70 ^{o}C, and 40 rpm for 9 min. There after, the curatives were added and mixed with the compounds on the two-roll-mill at 70 ^{o}C for 5 min. Then the mixes were sheeted out and kept at room temperature for 24 hr before testing.

Prior to vulcanize the mixes, a vulcanization time, the time at which the rheometer torque increased to 90% of the total torque change on the cure curve, was determined by means of Moving Die Rheometer (MDR). The mixes were compression molded using a hydraulic hot press at 150 °C under pressure 15 MPa.

The experiment was mainly divided into 2 steps:

Step 1 Study the effect of vulcanization system on mechanical properties of vulcanizates.

The mechanical properties of the vulcanizates cured with conventional vulcanization (CV) and efficient vulcanization (EV) were compared with those of rubber mat's formulation (control) obtained from the industry. The compound formulations are given in Table 3.

Table 3 Formulations of rubber compound with various vulcanization systems

Ingredient	phr		
	Control	CV	EV
Natural Rubber	100	100	100
Carbon Black (N330A)	61	61	61
$CaCO_3$	159	159	159
ZnO	7	7	7
Stearic acid	2.5	2.5	2.5
Antioxidant	3	3	3
Ground Rubber	285	285	285
Rubber oil	100	100	100
CBS	2.5	0.5	2.5
Sulphur	2.5	2.5	0.8

Step 2 Study the effect of type and amount of fillers on mechanical properties of vulcanizates after considering the proper vulcanization system.

The compound formulations are given in Table 4.

Table 4 Formulations of rubber compound with various types and amount of fillers

Ingredient	phr		
	$CaCO_3$	RHA	Natural Zeolite
Natural Rubber	100	100	100
Carbon Black (N330A)	61	61	61
Filler	Variable (100, 159, 200, 250)		
ZnO	7	7	7
Stearic acid	2.5	2.5	2.5
Antioxidant	3	3	3
Ground Rubber	285	285	285
Rubber oil	100	100	100
CBS	2.5	2.5	2.5
Sulphur	0.8	0.8	0.8

2. Characterization of Fillers

Physical properties and surface area of fillers ($CaCO_3$, RHA and natural zeolite) were determined by the Brunauer-Emmett-Teller (BET) on Autosorb-1 method. Particle size analyzer (wet sieve) was used to measure the particle size of the fillers. The composition of $CaCO_3$, RHA and natural zeolite was measured by X-ray fluorescence spectrometry (XRF).

3. Mechanical Properties

3.1 Hardness

Shore A hardness of the specimens was measured using hardness tester. The specimens about 6 mm in thickness were placed on test platform. The durometer was held in a vertical position with point of the indenter at least 12 mm from any edge

of the specimens. Five measurements were taken as the hardness value of the test sample.

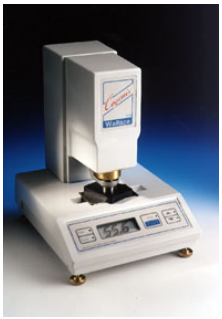


Figure 16 Hardness Tester

3.2 Abrasion

The density of the specimens with 12 mm thickness was determined by Densimeter. The specimens were weighed before and after test friction by DIN abrasion. The expression of loss volume is shown as following:

$$\text{Volume loss} = m_1\text{-}m_2 / D$$

where: m_1 = weight of the specimen before testing
m_2 = weight of the specimen after testing
D = density of the specimen

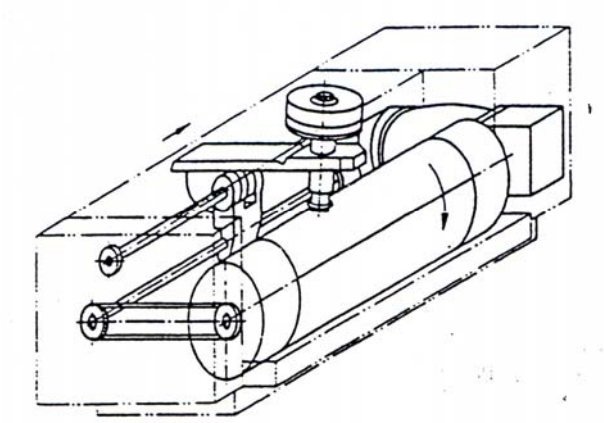


Figure 17 Abrasion Tester

3.3 Compression Set

The original thickness of the specimens was measured. The test specimens were placed between the plates of the compression device with the spacers on each side, allowing sufficient clearance for the bulging of the rubber when compressed at 70^{o}C for 22 hour (Fig 18).

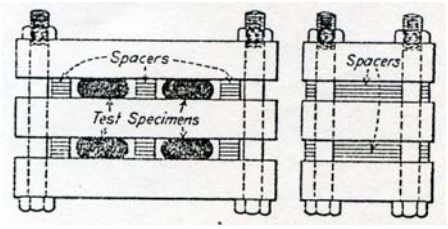


Figure 18 Device for Compression Set Test under constant deflection

The specimens then rested on poor thermally conducting surface, such as wood, for 30 min before making the measurement of the final thickness.

The expression for the calculation of the compression set:

$$C_B = [(t_o - t_i)/ (t_o - t_n)] \times 100$$

where: C_B = compression set expressed as percentage of the original deflection

t_o = original thickness of specimen

t_i = final thickness of specimen

t_n = thickness of the spacer bar used (4.5mm)

3.4 Tear Strength

Figure 19 Universal Testing Machine

The vulcanizates were cut into tear specimens by using the punching machine. Testing was carried out on universal testing machine in accordance with ASTM D624-98.

The crosshead speed of 500 mm/min was used with a full scale load cell at 1 kN. At least 5 specimens were used for each measurement.

4. Thermal Aging

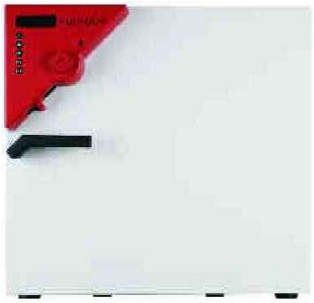


Figure 20 Oven

Accelerated thermal-oxidative aging testing was followed in the present investigation. The thermal aging experiment was performed in an oven at temperature 70 ^{o}C for 10 days according to ASTMD 573-99.

After the thermal exposure, the specimens were evaluated by mechanical properties. The results were compared with the specimens before testing.

5. Fluid Resistance

Fluid resistance experiment was performed in fluid (H_2O and cow's urine) at room temperature for 70 hours that was done according to ASTM D471-98.

After the immersion in fluid, the mechanical properties of the specimens were undertaken. The obtained results were then compared with the specimens before testing.

6. Mechanical Properties after Installation in Cow's Corral

The appropriate rubber compound formulations were manufactured as rubber mat with 30 mm thickness by the factory. The manufactured rubber mats were then installed in cow's corral. After 2 months, hardness and thickness of the installed

rubber mats were measured at 2 different regions (i.e. irregular and regular contact by cows).