

CHAPTER III

EXPERIMENTAL

3.1 Materials

1. High-ammonia natural rubber (HANR) latex
: Thai Rubber Latex and Cooperation (Thailand) Co., Ltd
2. Titanium (IV) oxide (TiO_2)
: Analytical grade; Riedel-de Haen and Degussa P25
3. Sulfur
: Chemical grade; Kijphaiboon Co.Ltd.
4. Zinc oxide
: Chemical grade; Kijphaiboon Co.,Ltd
5. Zinc diethyl dithiocarbamate (ZDEC)
: Chemical grade; Kijphaiboon Co.,Ltd
6. Potassium Laurate
: Chemical grade; Kijphaiboon Co.,Ltd
7. Potassium hydroxide
: Chemical grade; Kijphaiboon Co.,Ltd
8. Toluene
: Analytical grade; Carlo Erba
9. Methanol
: Analytical grade; Merck
10. Tetrahydrofuran
: Analytical grade; Lab-Scan

3.2 Equipments

1. Ultraviolet light
: Phillip , UVB 290-315 nm, 40 watt
2. UV Simulator Machine
: Q. Panel Company, 160 watt
3. Hot Plate
: Corning
4. Analytical Balance
: Mettler , model AB204-S
5. Centrifuge
: MSE, model Centaur 2
6. Mechanical Stirrer
: IKA Labortechnik, model RW 20DZM.n
7. Fourier Transform Infrared Spectrophotometer (FT-IR)
: Perkin Elmer, Spectrum One
8. Scanning Electron Dispersive X-ray Microscopy (SEM-EDX)
: JEOL, model JSM-5800LV
9. Gel Permeation Chromatography (GPC)
: Waters, model 150-CV, PLgel column, RI detector
10. Tensile Tester
: LLOYD, model LLOYD500, UK
11. Mastersizer S
: Malvern, model Mastersizer S, UK

3.3 Procedure

3.3.1 Purification and Determination of Gel Content of Natural Rubber

HANR latex 5 mL was poured into a glass mold and covered with aluminum foil. The latex was dried in the dark at ambient temperature for 2 days. The dried

latex sheet was dissolved in toluene. The solution was kept without stirring in the dark at ambient temperature for 3 days. The gel fraction was collected as a bottom fraction by vacuum filtration. The solution fraction of rubber was collected as a sol fraction, which was later precipitated with an excess amount of methanol. The dried solid rubber obtained after precipitation was called fractionated rubber.

3.3.2 Oxidative Degradation of Natural Rubber Solution in the Presence of TiO₂ under Accelerated Condition

Rubber solution (5.0×10^{-3} g/mL) was prepared by dissolving 1.0 g of fractionated rubber in 200 mL toluene, which was stirred for 2 days in the dark at ambient temperature. TiO₂ powder was added into 100 mL beaker containing 30 mL rubber solution and a magnetic stirrer to obtain 0.5, 1, 10, 20 %w/w. The solution was kept stirring continuously while exposed to ultraviolet light for a desired period of time (15-120 min). The solution was then centrifuged and filtered to separate TiO₂ powder. Solid rubber was obtained by precipitating the rubber solution in an excess amount of methanol and dried under vacuum for 24 h.

3.3.3 Oxidative Degradation of Unvulcanized Natural Rubber Sheet Filled with TiO₂ under Accelerated Condition

TiO₂ powder (0.5, 1, 5, 10 and 20 g) was mixed with 100 g HANR latex by stirring for 2 h. The mixture was poured into a glass mold and covered with aluminum foil. The latex was dried in the dark at ambient temperature for 2 days. The rubber sheets were then incubated in a UV simulator machine and exposed to sunlight for a desired period of time. The irradiated rubber sheet was dissolved in 200 mL toluene. The solution was kept without stirring in the dark at ambient temperature for 2 days. The solution was then vacuum filtered to remove TiO₂ powder along with the gel fraction. After the removal of TiO₂, the solid rubber was obtained by adding an excess amount of methanol. The rubber was then dried under vacuum for 24 h.

3.3.4 Molecular Weight Determination of Natural Rubber by Gel Permeation Chromatography

15 mg of irradiated solid rubber was dissolved in 5 mL of tetrahydrofuran (THF) and shaken for 24 h. Rubber solution was then subjected to molecular weight determination using gel permeation chromatography fitted with PL gel column. The column set was calibrated using polystyrene standard. Tetrahydrofuran was used as the solvent with a flow rate of 1 mL/min at 30°C.

3.3.5 Preparation of Vulcanized Natural Rubber Sheet Filled with TiO₂

Pre-vulcanized natural rubber latex was prepared from concentrated HANR latex having 60% dry rubber content (DRC). Ingredients used for sulfur pre-vulcanization were obtained in dispersion form. Each ingredient was prepared by grinding its original form in water for 24 h in a ball mill. The formulation is shown in Table 3.1

Table 3.1 Formulation for sulfur pre-vulcanization

Ingredient	Composition (phr)
60 % HANR	100
10 % Potassium hydroxide	0.50
20 % Potassium laurate	0.25
50 % Sulfur	0.75
50 % Zinc diethyl dithiocarbamate (ZDEC)	1.00
50 % Zinc oxide (ZnO)	1.00

Sulfur, ZDEC and ZnO in the form of 50% dispersion were added to a 1-L beaker containing HANR latex at 60°C under continuous stirring for 2 h. 25% TiO₂ dispersion was then added into the latex mixture to get 0.5, 3, 5 and 10 %w/w. The pre-vulcanized latex was filtered to remove non-dispersed material and left overnight in the dark at ambient temperature.

A test tube with 2-cm diameter was dipped into the pre-vulcanized latex for 45 sec to obtain compounded rubber sheet with the thickness of 0.1-0.2 mm. The compounded rubber sheet was dried in the dark at ambient temperature. The vulcanized sheets were prepared by heating the compounded sheet at 100°C for 1 h. The vulcanized sheets were exposed to UV for a desired period of time (4-24 h). Photooxidative degradation was studied by monitoring mechanical properties.

3.3.6 Mechanical Testing

The tensile strength and elongation at break of vulcanized rubber sheets were measured using a tensile tester according to ASTM D 412-87. The condition for tensile testing is illustrated in Table 3.2. A dumbbell test piece (type C) has a dimension as shown in Figure 3.1.

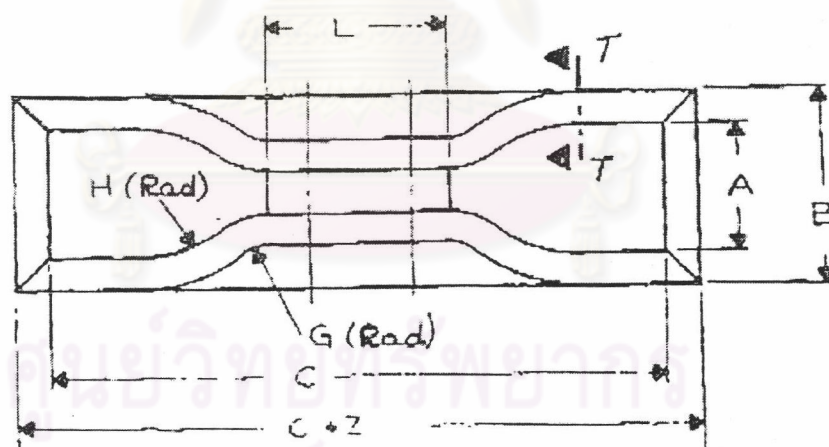


Figure 3.1 Schematic of tensile specimen

Table 3.2 Dimension of dumbbell test pieces (type C) prepared for mechanical testing

Dimension	(mm)
(A) Width of ends	25 ± 1
(C) Overall length (minimum)	115
(G) Transition radius outside	14 ± 1
(H) Transition radius inside	25 ± 2
(L) Length of narrow portion	33 ± 2

Table 3.3 The condition for tensile testing

Temperature	25 °C
Humidity	60%
Crosshead speed	500.00 mm/min
Gauge length	20.0 mm

ศูนย์วิทยทรัพยากร
จุฬาลงกรณ์มหาวิทยาลัย