

CHAPTER III

EXPERIMENTAL

3.1 Chemicals

1. Natural rubber latex : Revertex (Thialand) Ltd.
2. Zinc dibutyl dithiocarbamate (ZBDC) : Rubber Research Institute
3. Tetramethyl thiuram disulfide (TMTD) : Rubber Research Institute
4. Titanium dioxide : Rubber Research Institute
5. Zinc oxide (ZnO) : Rubber Research Institute
6. Casein : Fluka
7. Potassium hydroxide : Meark
8. Tergital NP9 : Union Carbide
9. Ammonium hydroxide : J.T. Baker
10. Chloroform : Ajax Chemicals
11. Toluene : Carlo Erba
12. Carboxymethylcellulose : Commercial
13. Lauric acid : Commercial

3.2 Instruments and Apparatus

1. Ball mill
2. Mastersizer : Malvern Instruments Ltd.
3. Universal testing machine : Instron Corporation
: Lloyd Instruments
4. Hardness testing machine : The Shore Instrument & MFG.Co.Inc.
5. Brookfield viscometer : RVT small sample
6. Natural rubber lattice mechanical
stability apparatus : Klaxon
7. Thermogravimetric analyzer : Elmer Perkin TGA7

3.3 Procedures

3.3.1 Preparation of Compounding Ingredients

Zinc Oxide (ZnO), Tetramethyl Thiuram Disulfide (TMTD), Zinc Dibutyl Dithiocarbamate (ZBDC), Titanium Dioxide Dispersion (TiO₂)

All of the ingredients were prepared by ball mill under room temperature. The rate of ball mill was 84 rpm. The formulations for dispersions of compounding ingredients are shown in Table 3.1. In the case of ZBDC, it would be drying milled for 2-4 hours before mixing with bentonite clay, dispersant (vultamol), and water for another 26-36 hours.

Table 3.1 Formulations for dispersions of compounding ingredients.

	Parts by weight			
	ZnO	ZBDC	TMTD	TiO ₂
ZnO	50	-	-	-
ZBDC	-	33	-	-
TMTD	-	-	50	-
TiO ₂	-	-	-	50
Bentonite clay	1	0.5	1	0.5
Vultamol	1	1.5	1	-
Vulcastab LW	-	-	-	2
Water	48	75	48	47.5
Grinding time by ball mill	12-24 hr.	26-36 hr.	26-36 hr.	12-24 hr.

Particle size

The particle size analysis was determined by the Mastersizer. The average particle diameter and size distributions of rubber particle and ingredient dispersions are shown in Appendix A.

3.3.2 Preparation of Prevulcanized Latex

Natural rubber latex was mixed with ingredients as shown in Table 3.2 by mechanical stirrer to become a homogeneous latex at 30°C for approximately 30 minutes. Then, the compounded latex was heated to 70-80°C and maintained at this temperature until the desired degree of vulcanization had been achieved. Constant stirring (50 rpm) was maintained with special attention to keep the particles of ingredients in suspension state. Care was also taken to minimize skin formation. After the required degree of vulcanization, prevulcanized latex was cooled, strained and stored at room temperature for at least 7 days before subjecting to other tests.

Table 3.2 Formulation of prevulcanized latex

Ingredient	Quantity of dry weight (phr)
60% Natural rubber latex	100 parts by dry weight
15% Casein	0.5
25% Tergitol NP 9	0.5
10% KOH	0.75
50% ZnO	1
50% TMTD	3
33% ZBDC	1
10% Thiourea	1
50% Titanium dioxide	2.5

The prevulcanized latex was prepared and subjected to various investigations as follows:

1. Effect of prevulcanization time to degree of vulcanization

The compounded latex was heated at 70-80°C for 0, 2, 4, and 6 hours. The degree of vulcanization was determined by chloroform test. The chloroform test is characteristic of rubber latex coagulation by chloroform or carbontetrachloride in the same quantity (approximately 10 ml.) and stirred by stirring rod. The characteristic of rubber latex to coagulate within 2-3 minutes was shown as the degree of vulcanization.

2. Effect of KOH concentration

The prevulcanized latex was prepared by heating to 70-80 °C at an appropriate time. KOH concentration was varied by changing at 0.25, 0.50, 0.75 and 1.0 phr. and other ingredients were the same as shown in Table 3.2.

3. Effect of surfactant concentration

The prevulcanized latex was prepared at appropriate concentrations of KOH. The amount of surfactant was varied by changing at of 0.25, 0.5, 0.75 and 1.0 phr. and other ingredients were the same as shown in Table 3.2.

4. Effect of protective colloid concentration (casein)

The prevulcanized latex was prepared at appropriate concentrations of KOH and surfactant. The amount of protective colloid was varied by changing at 0.25, 0.50 and 0.75 phr. and other ingredients were the same as shown in Table 3.2.

5. Effect of types of surfactant

The prevulcanized latex was prepared at appropriate concentration of KOH and protective colloid. The formulations of prevulcanized latex were used as 20% ammonium laurate as surfactant and used by changing the concentrations of 0.05, 0.10, and 0.15 phr. and other ingredients were the same as shown in Table 3.2.

6. Effect of vulcanizing system , titanium dioxide and types of surfactant

The prevulcanized latex was prepared at appropriate stabilizers. The formulations of prevulcanized latex were changed as shown in Table 3.3.

Table 3.3 Formulations to study effect of vulcanizing system, titanium dioxide and types of surfactant

Ingredient	Type	Quantity of dry weight (phr)
60% Natural rubber latex	-	100
15% Casein	-	0.75
10% KOH	-	1
Surfactant	25% Tergitol NP9,	0.5
	20% Ammonium laurate	0.15
Vulcanizing system		
50% ZnO	-	0.33, 0.66, 1.00
50% TMTD	-	1, 2, 3
33% ZBDC	-	0.33, 0.66, 1.00
10% Thiourea	-	0.33, 0.66, 1.00
Filler: 50% Titanium dioxide	-	1.25, 2.5, 3.75

3.3.3 Preparation of Vulcanized Sheets

The prevulcanized latex was formed into a film by pouring off it on a glass plate to stand at room temperature for 48 hours. The film was peeled from the glass plate surface, heat-treating at 120 °C for 30 minutes, and then subjected to the measurement of mechanical properties.

3.4 Mechanical Stability Time Testing (MST) (ASTM D 1076)

The prevulcanized latex was warmed to 36-37°C. The warmed latex was immediately strained through a 180- μ m stainless steel sieve and 80 \pm 0.5 g of the strained latex was weighed out and placed into the test bottle. The test bottle was placed in the container platform for stirring. The position of the test bottle shall be such that the axis of the stirrer was concentric with the axis of the stirrer shaft which was concentric with the axis of the bottle, and that the bottom of the agitator disk was 12.7 \pm 2.5 mm from the bottom inside of the bottle. Subsequently, the latex was stirred at 14,000 \pm 200 rpm until the end point was reached. The end point was determined every 15 seconds, by dipping a stainless stirring rod into the latex and touched on the surface of water for spreading it. The first appearance of small pieces of coagulated rubber that deposited in the water was the end of the test. This end point should be confirmed by the presence of increased amount of coagulated rubber in a water surface deposit after 15 seconds additional agitation.

3.5 Mechanical Testing

Mechanical properties of the vulcanized rubber were measured by following the ISO and the ASTM test methods as follows:

ISO 37 : Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties

The tensile strength and the elongation were determined. The dumb-bell test pieces (type 1) shall have the outline shown in Figure 3.1. The test length shall be 25 ± 0.5 mm. for type 1. And dies for preparation of dumb-bells have the dimensions shown in Figure 3.2.

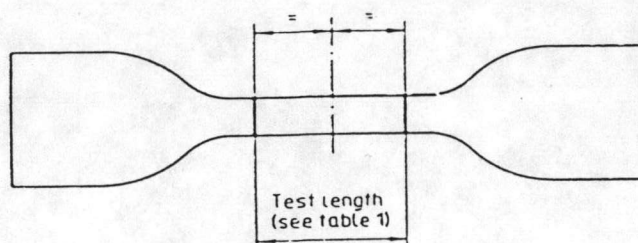


Figure 3.1 Shape of dumb-bell test pieces

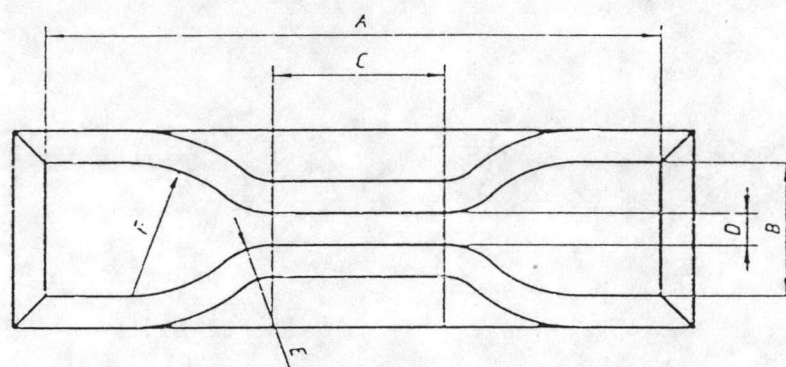


Figure 3.2 Dimensions of dies for dumb-bell test pieces

Dimension	Type 1 (mm.)
A Overall length (minimum)	115
B Width of ends	25.0 ± 1.0
C Length of narrow portion	33.0 ± 2.0
D Width of narrow portion	$6.0 + 0.4$
E Transition radius outside	14.0 ± 1.0
F Transition radius inside	25.0 ± 2.0

The tensile testing condition

Temperature	25 °C
Humidity	50 %
Crosshead speed	500.0 mm./min.
Full scale load range	5.0 kN

ASTM D 2240: Standard test method for rubber property-Durometer hardness

In this experiment, vulcanized sheets were used for the type A durometer. The shape of the indenter is shown in Figure 3.3.

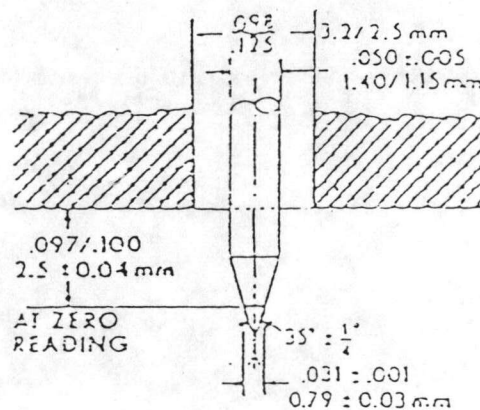


Figure 3.3 Indenter for type A durometers

The test specimen was at least 6 mm.in thickness unless it was known that results equivalent to the 6 mm. values were obtained with a thinner specimen. The minimum requirement for thickness of the specimen depended on the extent of penetration of the indenter into the specimen. The lateral dimensions of the specimen were measured to at least 12 mm. from any edge. The surface of the specimen was

The viscosity of solder masks (samples) were determined by Brookfield viscometer models RVT small sample by pouring the samples into the container that was maintained at 25 ± 2 °C. A spindle no.14 was carefully inserted into the samples, in such a way as to avoid air being trapped. The spindle was placed vertically in the samples and in the center of the container. The rotational frequency of spindle was slowly adjusted to appropriate the pointer to be indicated in the range of 5-95. Viscosity of solder masks were expressed in millipascal seconds (centipoises), using the appropriate factor obtained from Appendix D.

3.7 Determination of Thermal Properties of Solder Mask

The initial degradation temperature was measured using the thermogravimetric analyzer (TGA) of Elmer Perkin, model TGA7. Data were recorded at a scanning rate of 20 °C/min. against a baseline scan. Temperature range of the scan was 100 to 600° C with the nitrogen pressure 40 psi. and weight of specimens used was 1-5 mg.