

## CHAPTER IV

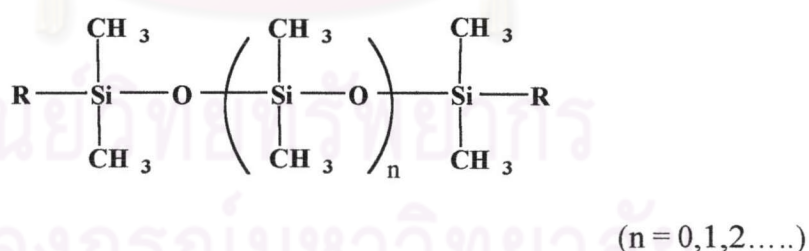
### EXPERIMENTAL WORK

This chapter explained the characteristics of the materials used in this research as well as all essential experimental techniques.

#### 4.1 MATERIALS

##### 4.1.1 Polydimethylsiloxane [14]

Siloxane polymers, PDMS in this case, are well known due to their highly flexible backbone, which renders it one of the lowest glass transition temperature ( $T_g$ ) i.e.  $-127\text{ }^\circ\text{C}$ [15]. Linear PDMS is a highly viscous liquid, whereas the crosslinked polymers are an elastomer. These inherent characters have led PDMS to find a wide range of industrial applications. Many commercially available silicone resins contain solvents, fillers, low molecular weight crosslinkers, catalysts and other additives. These unknown components may cause unexpected problems due to chemical and physical interactions. The chemical structure of polydimethylsiloxane is presented in Figure 4.1



R = reactive end groups

**Figure 4.1** The chemical structure of polydimethylsiloxane

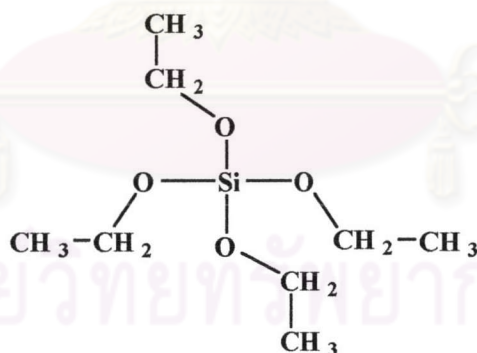
In this work, four types of commercial RTV silicone resins as a function of crosslinker concentration were investigated. Locally available resins; RTV 585, RTV 300, RTV 3480 and RTV 4503 were studied. Typical characteristics of each silicone resin types are shown in Table 4.1

**Table 4.1 Fundamental characteristics of commercial silicone resins**

Characteristics	RTV 585	RTV 300	RTV 3480	RTV 4503
Appearance	viscous liquid	viscous liquid	viscous liquid	viscous liquid
Colour	beige	white	pale gray	white
Density, 25°C (g/cm <sup>3</sup> )	1.22	1.10	1.33	1.17
Viscosity, 25°C (mPa.s)	45,000	50,000	35,000	40,000
Cost (Baht/kg)	630	600	900	800

#### 4.1.2 Curing Agents

A silicone resin was formed linkages at room temperature with an aid of a curing agent i.e chemical crosslinking. Organometallic (tin) catalyst was utilized as a curing agent in the present work. It is a clear or slightly amber liquid. The curing agent was varied at different concentrations to investigate its effects on the resulting silicone properties. The chemical structure of tetraethoxysilane is shown in Figure 4.2.

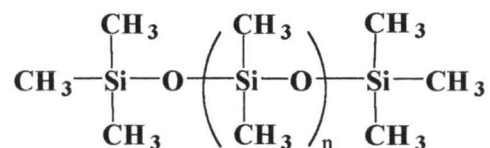


**Figure 4.2 The chemical structure of tetraethoxysilane**

#### 4.1.3 Modifying Agents [16]

Silicone oil, was utilized as a modifying agent in this work, are medium viscosity polydimethylsiloxane polymers manufactured to yield linear polymers. A suitable proportion of silicone oil added to the mixture in order to soften the silicone elastomer, and to render the gel composition of more lifelike. Linear

polydimethylsiloxane polymers characteristically have the following typical chemical composition as shown in Figure 4.3. The characteristics of silicone oil that used in this work are shown in Table 4.2



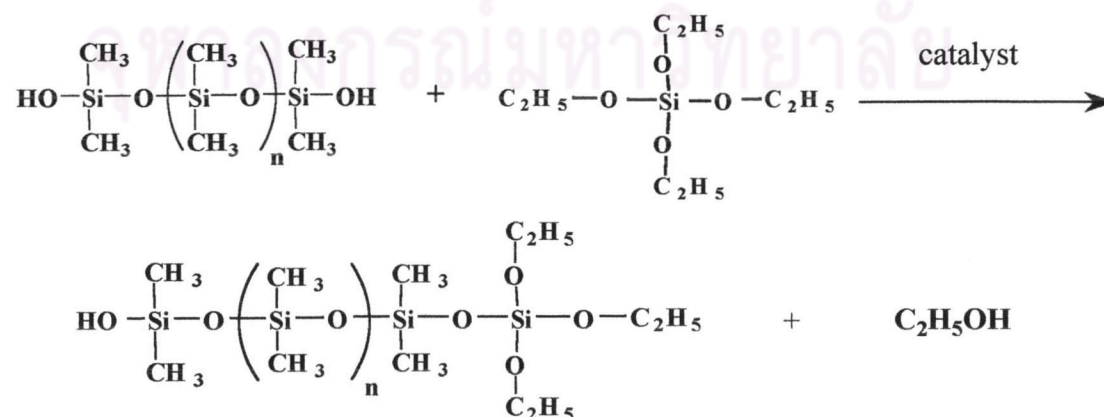
**Figure 4.3** The chemical structure of methyl silicone oil

**Table 4.2** Fundamental characteristics of methyl silicone oil

Characteristics	methyl silicone oil
Appearance	colorless transparent liquid
Viscosity, 25°C (mPa.s)	350
Density, 25°C (g/cm <sup>3</sup> )	0.968
Cost (Baht/kg)	300

#### 4.1.4 Formulation of silicone elastomer

Silicone resins used in the present are silanol-terminated polydimethylsiloxane prepolymer. They were crosslinked with tetraethoxysilane in the presence of organometallic as a catalyst. The formulation of silicone elastomer is shown in Figure 4.4. From the reaction, a condensation by-product of cure is a small amount of alcohol.

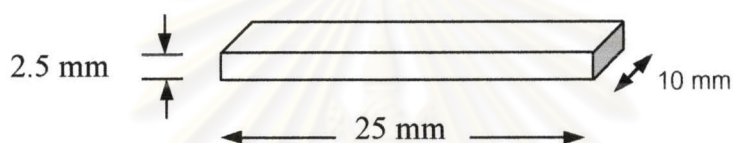


**Figure 4.4** Condensation curing system

## 4.2 SAMPLE CHARACTERIZATION

### 4.2.1 Physical Aging

All silicone elastomer samples in this work were aged according to the room temperature condition ( $27 \pm 2$  °C). They were aged for periods up to four weeks. Then, the samples were left to observe tensile properties for every week. They were cut into strip-shaped samples. The silicone sheet is 10 mm in width, 25 mm in length and  $2.5 \pm 0.2$  mm in thickness. Figure 4.5 showed the shape of the test of aging effect sample.



**Figure 4.5** The shape of the test of aging effect sample

### 4.2.2 FT-IR Spectroscopy

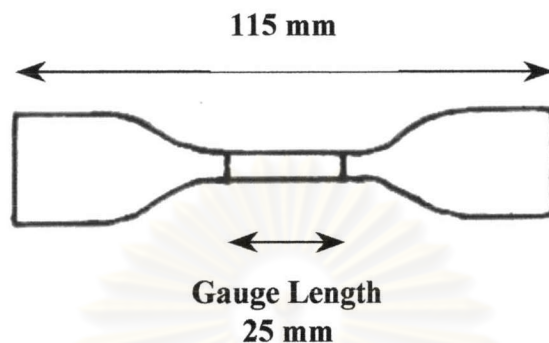
Types of silicone resins are characterized by Fourier Transform Infrared Spectroscopy (FT-IR) technique. All spectra of commercial silicone resins were recorded with a “Bruker Vector 33 spectrometer” instrument. The spectra were measured with 64 scans at a resolution of  $4\text{ cm}^{-1}$  and recorded in the range of  $4000$  to  $400\text{ cm}^{-1}$ . It was observed using a deuterated triglycinesulfate detector (DTGS).

### 4.2.3 Tensile Measurements

One of the most widely used testing methods for mechanical properties of polymer is the tensile or stress-strain test. This test is usually done by measuring the force that generates as the specimen is elongated at a constant rate of extension. The force and the extension of specimens can be converted to stress and strain by calculations using the original dimensions and the increase in length.

Test specimens were prepared according to ASTM D412, as illustrated in Figure 4.6. The tensile properties of specimens were determined by using a universal

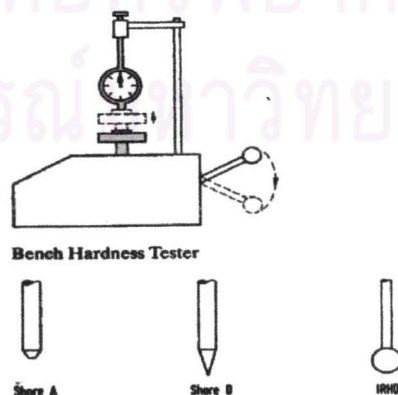
testing machine (LLOYD 2000R) outfitted with an extensometer and a load cell of 1 kN. The crosshead speed was 500 mm/min. Reported tensile properties were based on the mean of at least three samples for each composition. The results of the maximum tensile strength, percent elongation at break and modulus of elasticity were reported.



**Figure 4.6 Standard Dies for cutting dumbbell specimen**

#### 4.2.4 Hardness Measurements

Hardness is defined as the resistance of a material to deformation, particularly permanent deformation, indentation or scratching. This test method is based on the penetration of a specific type of indenter when forced into the material under specified conditions. Test specimens were prepared using ASTM D2240 (shore A) and were examined by using a durometer hardness testing (PTC instruments Model 408 Type A). Figure 4.7 shows a durometer hardness tester and type of an indenter. Five measurements of hardness at different position on the specimen of at least 6 mm. (0.25 in.) apart were performed and the arithmetic mean was determined.



**Figure 4.7 Durometer hardness tester and indenter [17]**

#### 4.2.5 Rheological Measurements

Rheological measurements were conducted using a computer-controlled Haake RS75 Rheometer in a cone and plate geometry. The bottom plate is a fixed surfaced which is movable (up and down). The top plate is a cone of 3.5 cm in diameter with a 0.017 radian ( $1^\circ$ ) cone angle and gap 0.052 mm. The curing process in thermosets can be followed by time sweep of a dynamic oscillation measurement performed at a specific frequency of 1 Hz. The strain amplitude was set to be  $2.5 \times 10^{-2}$  [18]. The storage modulus ( $G'$ ), and loss modulus ( $G''$ ) were continually monitored as a function of time. All experiments were conducted at 25 °C

#### 4.2.6 Tear Strength Measurements

Tear strength is important in the performance of general rubber products and this method measures the resistance to tearing action. Test specimens were prepared following ASTM D624 (Die C), as illustrated in Figure 4.8

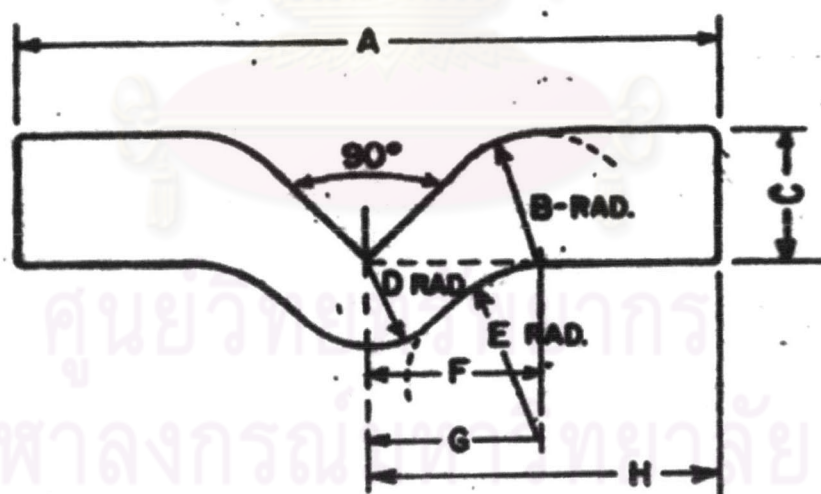


Figure 4.8 Die C for tear test specimen

Dimension	Millimetres		Inches	
	Value	Tolerance	Value	Tolerance
A	102	± 0.50	4.0	± 0.02
B	19	± 0.05	0.75	± 0.002
C	19	± 0.05	0.75	± 0.002
D	12.7	± 0.05	0.5	± 0.002
E	25	± 0.05	1.0	± 0.002
F	27	± 0.05	1.061	± 0.002
G	28	± 0.05	1.118	± 0.002
H	51	± 0.25	2.0	± 0.01

Measurements of tear strength were carried out at room temperature and were applied a steadily increasing traction force at a rate of separation of the grips of  $500 \pm 50$  mm/min. The tear strength ( $G_c$ ) is calculated through equation 4.1.

$$G_c = 2f/t \dots \dots \dots (4.1)$$

where :

$G_c$  = tear strength in force/unit thickness

f = force applied to ends of test piece

t = thickness

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