CHAPTER III

EXPERIMENTAL

3.1 Apparatus and Instrument

1. High speed Labomixer with rotor speed of 2,000 rpm

   PAPENMEIER; Model TGEHKV 10

2. Brabender plasticorder torque rheometer

   BRABENDER; Model PL-2000

3. Hardness tester

   BAREISS; Model HHP-2000

4. Oxygen index tester

   CEST; Model 6170/000 – 6171/000

5. Balance with 4 decimals

   METTLER; Model E400

6. Universal testing machine

   LLOYD; Model L-1000R

7. Scanning electron microscope

   JEOL; Model JSM-5410 LV

3.2 Materials Used

1) Polymer

Suspension PVC, industrial grade, TPC Co., Ltd. with K 66

(a molecular weight index for PVC polymers).

2) Two plasticizers were studied :

2.1) Bis-2-ethylhexyl phthalate or dioctyl phthalate (DOP) [4, 16] :

$C_{24}H_{38}O_4$

Molar mass 390.6 g/mol

Density (ρ) 985 kg/m^3

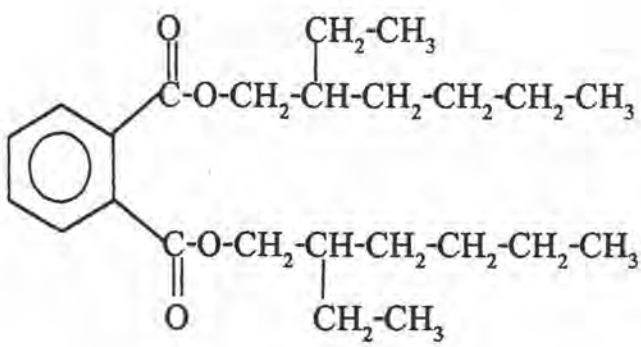

Figure 3.1: Structure of DOP [16]

Plasticizers used in the vinyl industry are mainly diesters of dibasic acids and monohydroxy alcohol and the most common are phthalates.

2.2) Chlorinated paraffin with 52 % chlorine (CP) : $C_{18}H_{30}Cl_8$

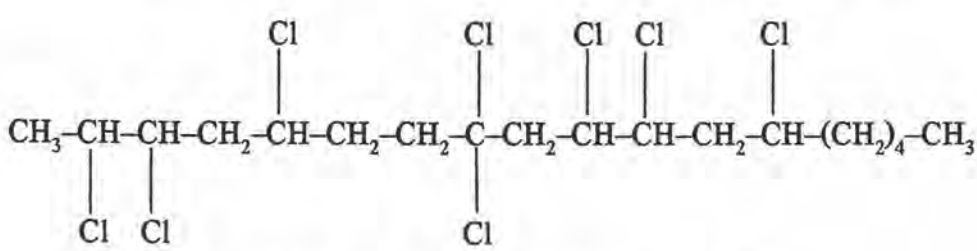

Figure 3.2: Structure of chlorinated paraffin [20]

Molar mass	530	g/mol
Density (ρ) @25c	1.25	g/ml
Viscosity @25c	1.6	Pa.s
Reflextive Index	1.508	
Carbon chain length	C18	
Volatility @180c,4hr	1.4	%w/w

3) Immersion oils

3.1) Motor oil (Lubricant oil which is a hydrocarbon with C atom 25up: low and high viscosity type)

This is deasphalted and dewaxed petroleum oil as base-oil to blend and use for motor oil.

Function: Base oil, solvent refined mineral.

Chemistry: lubricating oil (petroleum), C atom 25 up.

Appearance: A clear, bright, oily liquid: free of foreign matter. (slight petroleum odor)

Type	**Appearance**	**Viscosity (@40°C (cSt))**	**Specific gravity (@15°C)**
Low viscosity	Clear amber liquid	11	0.850
High viscosity	Pale yellow colored liquid	550	0.905

3.2) Silicone oil (lubricating oil that is polydimethyl liquid: low and high viscosity type)

Chemical name is polydimethyl siloxane or dimethicone, is clear water-white, tasteless, odorless and neutral liquid which has chemical structure as follow:

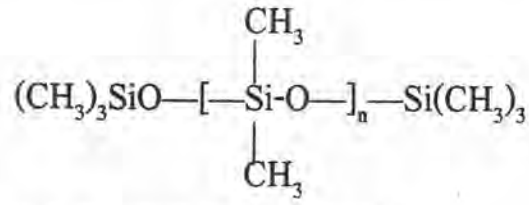

Figure 3.3: Structure of silicone oil[21]

Type	Appearance	Viscosity (@ 25°C (cSt))	Specific gravity
Low viscosity	Colorless transparent liquid	100	0.97
High viscosity	Colorless transparent liquid	1000	0.97

4) Other materials: stabilizer system (lead stabilizer), calcium carbonate: industrial grade.

3.3 Preparation of Samples

3.3.1 Dry blend samples

PVC (100 phr) lead stabilizer (3 phr) and calcium carbonate (30 phr) were blended at starting temperature of about 37°C in a high speed Labomixer with 2000 rpm until the temperature reached 80°C, various amounts of plasticizer blends (DOP and chlorinated paraffin) were added.

The mixture was continuously blended until the temperature rose to 120°C, the system was cooled and the speed was decreased to 1000 rpm. After cooling down to

around 40~50°C, the sample was discharged from the Labomixer and called "dry blend".

3.3.2 Milled sheet samples

The dry blend in section 3.3.1) was placed on a hot two-roll mill at 150°C, to mill them homogeneously for 5 min. The thickness of milled sheet was adjusted to about 1 mm. After a further 2 min., the sheet was removed from the two-roll mill. After cooling, the milled sheet was faintly yellow and flexible (called "milled sheet") and was cut to dimension of 20 cm. x 20 cm., the same size as stainless steel pressed mold.

3.4 Migration of plasticizer and hardness measurement after oil ageing

Six of the milled sheets with 1 mm. thickness in section 3.3.2) were hot pressed at 160°C (preheated 2 min., heated 3 min. and cooled down 5 min.) to form a sheet with thickness about 6 mm. The pressed sheet were allowed to stand overnight and were cut to dimension of specimen 50 mm. x 50 mm. x 6 mm. and kept them again for one night before weighing and measuring of hardness according to ASTM D-2240.

Three specimens of each formulations were immersed in four different hot oil baths: high and low viscosity motor oils and silicone oils for 2 hours, 4 hours, 24 hours and 48 hours at 70°C and 120°C. Then these specimens were removed from the oil bath and wiped with tissue. They were left overnight at ambient temperature before weighing to record the weight loss and specimens which were immersed in oils for 4 and 48 hours would be kept another 5 days to measure hardness according to JIS K-6301. The experiment of each formulation was repeated for immersion 4 and 48 hours and the results were obtained by average.

3.5 Determination for degree of fusion (Compatibility)

The dry blend in section 3.3.2) of each formulation was processed in a Barbender plasticorder torque rheometer according to ASTM D-2538. Fifty grams of dry blend was discharged in the hot mixing chamber at 140°C. After 15 min., the temperature and torque at the fusion peak were recorded. This method enabled products to be compared and compound formulations and temperature settings to be worked out. The procedure was repeated for all formulation studied.

3.6 Determination of flammability

Three of the milled sheets with 1 mm. thickness in section 3.3.2) were hot pressed at 160°C (preheated 2 min., heated 3 min. and cooled down 5 min.) to form a sheet with thickness about 3 mm. After cooled down and let them stand overnight, these pressed sheets were cut to dimension of specimen 6 mm. x 150 mm. x 3 mm. and kept them again for one night before testing of oxygen index (OI) method (ASTM D-2863).

Clamp the specimen in the holder vertically in the approximate center of the open glass column. Select the desired initial concentration of oxygen based on the difficulty of ignition and time of burning and purge the system with the constant flow for 30 seconds before igniting. After 20 seconds removing the ignition flame and start the time from zero again to determine the minimum concentration of oxygen which can burn the specimen for 3 minutes or longer. After that the result was repeated with a new specimen.

3.7 Mechanical Properties: Tensile Strength

The milled sheet with 1 mm. thickness in section 3.3.2) of each formulation was hot pressed at 160°C (preheated 2 min., heated 3 min. and cooled down 5 min.) to

form a sheet. After cooled down and let them stand overnight, these pressed sheets were cut to dog-bone shape in accordance with the dimension shown in Figure 3.4.

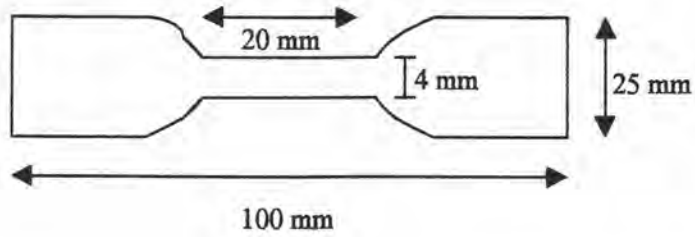

Figure 3.4: Schematic representation and dimension of dumbbell shape specimen[22]

And kept them again for one night before measuring the tensile property. Tensile test was carried out on a LLOYD universal testing machine, at 23°C, at a crosshead speed of 200 mm./min. according to ASTM D-638.