CHAPTER III

EXPERIMENTAL

3.1 Materials

Materials used in this research as tabulated in Table 3.1 are obtained from various sources.

Table 3.1 Materials and sources

Materials	Trade name	Sources of Suppliers
Poly(vinyl chloride)	SG – 610	Thai Plastic Co., Ltd.
Butyltin stabilizer	JF – 50R	Sankyo Co., Ltd.
Acrylic copolymer	PA – 20	Kaneka Co., Ltd.
Oleochemical derivative	G – 161	P.P.P. Chemical and Lube Co., Ltd.
Complex ester	G – 70S	P.P.P. Chemical and Lube Co., Ltd.
Acrylonitrile butadiene styrene	ABS	Henkel Co., Ltd.
Di – octyl phathalate	DOP	South City Chemical Co., Ltd.
Calcium carbonate	$CaCO_3$	Peerash Co., Ltd.
Magnesium silicate	Talcum	Polymer Innovation Co., Ltd.
Aluminium silicate	Kaolin	Centasia Co., Ltd.

3.2 Apparatus

1. Two – Roll Mill: MODELCR 820 SER.NO.7182
2. Compression Molding Machine: MODEL : GT7014 – 10
3. Mixer: MODEL MX - T31GN SER. NO950728
4. Oven: TYPE 1511530000202 No#T31GN DIN12880
5. Hardness Tester: MATSUZAWA DXT Rockwell ASTM D-785
6. Universal Tester: TESTROMETRIC MICRO 350
7. Heat Deflection Temperature Tester: ROSAND ASTM D-648
8. Scanning Electron Microscopy: JOEL Model JSM-5300

3.3 Experimental procedure

Various compounds were prepared by varying the amount of fillers such as $CaCO_3$, talcum and kaolin (Table 3.2). The components were weighed as parts per hundred by weight of the PVC resin. The compounds were mixed by a mixer for 3 minutes and blended on a two-roll mill at 200°C [21] to give 0.3 mm thick the compound specimens were tested for, mechanical and thermal properties namely, tensile strength, tear strength, impact strength, heat deflection temperature (HDT), and shrinkage. They were compared with those of commercial formulation.

Table 3.2. Formulations of PVC compounds

Sample No.	$CaCO_3$ (phr)	Talcum (phr)	Kaolin (phr)
S1	5.0	-	-
S2	10.0	-	-
S3	20.0	-	-
S4	-	5.0	-
S5	-	10.0	-
S6	-	20.0	-
S7	-	-	5.0
S8	-	-	10.0
S9	-	-	20.0
S10	2.5	2.5	-
S11	5.0	5.0	-
S12	10.0	10.0	-
S13	2.5	-	2.5
S14	5.0	-	5.0
S15	10.0	-	10.0
S16	-	2.5	2.5
S17	-	5.0	5.0
S18	-	10.0	10.0
S19	2.5	1.5	1.0
S20	5.0	3.0	2.0
S21	10.0	6.0	4.0
S22	1.0	2.5	1.5
S23	2.0	5.0	3.0
S24	4.0	10.0	6.0
S25	1.0	2.5	1.5
S26	2.0	5.0	3.0
S27	4.0	10.0	6.0

Remark - Other raw materials were fixed in the formulations S1-S27 as follows: PVC 100 phr, stabilizer 1.8 phr, impact modifier 7 phr, processing Aid 2.0 phr, internal lubricant 0.55 phr, external lubricant 0.65 phr.

3.3.1 Mixing Procedure

The compositions were prepared by mixing in a small high-speed mixer for 3 minutes and all of the ingredients were blended on a two-roll mill at 200°C for 10 minutes to make sheets having about 0.3 mm thick for tensile test. The nip between the rolls were adjusted to facilitate the mixing. The sheets of 0.30 mm thick were molded on a molding machine at 200°C and 200 kg/cm^2 for 13 minutes to make the PVC sheets with about 3 mm thickness for impact and HDT tests.

3.4 Mechanical Measurement

Mechanical properties of the rigid PVC opaque sheets were measured by the following ASTM test methods.

3.4.1 Tensile Testing

Tensile strength of the samples was determined by ASTM D638-97 [22]. The samples were tested at 25°C in a controlled humidity atmosphere of 65 %. The samples were cut into a dumbbell-shape, as illustrated in Figure 3.1

W	: Width of narrow section	6 mm.
L	: Length of narrow section	33 mm.
Wo	: Width of overall min	19 mm.
Lo	: Length of overall min	115 mm.
G	: Gage length	24 mm.
D	: Distance between grips	64 mm.
R	: Radius of fillet	14 mm.

Ro : Outer radius 25 mm.

T : Thickness 0.30 mm.

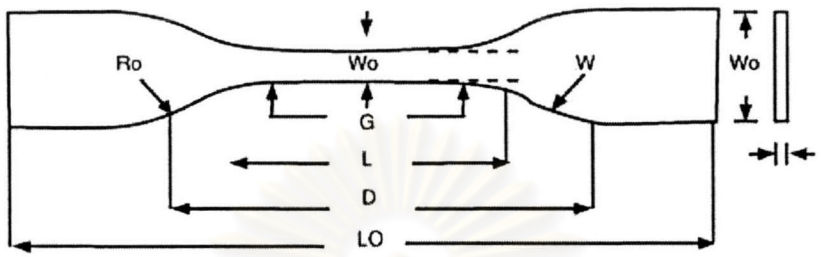

Figure 3.1 Schematic dimension of tensile test specimen

3.4.2 Impact Testing; Izod Impact

Impact strength is the resistance to shock loads and was determined by test method ASTM D618 [22]. The Izod value is a useful comparison of various types or grades of a filler. The part in the base of a pendulum testing machine was clamped, and cantilevered upward with the notch facing the direction of impact. The pendulum was released and the force consumed in breaking the specimen was calculated from the height of the pendulum reached on its follow-through in Figure 3.2.

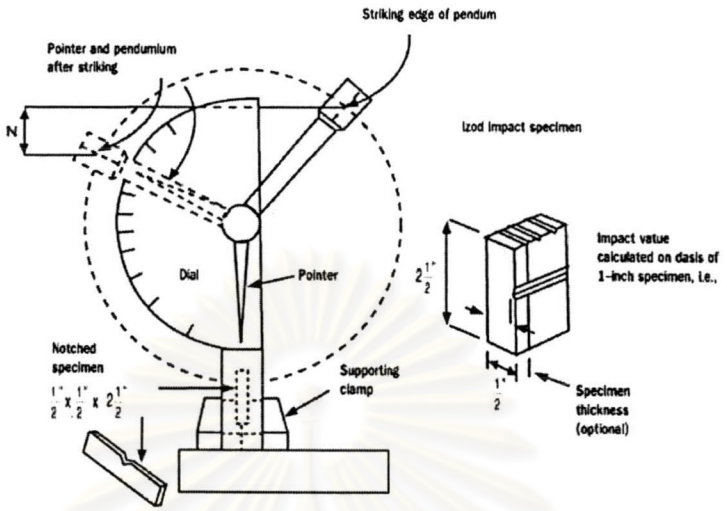

Figure 3.2 Schematic dimension of Izod Impact test machine and specimen

3.4.3 Tear Testing

The tear strength was carried out using a TESTROMETRIC MICRO350 universal tester according to ASTM D1938. The test specimen dimension is shown in Figure 3.3.

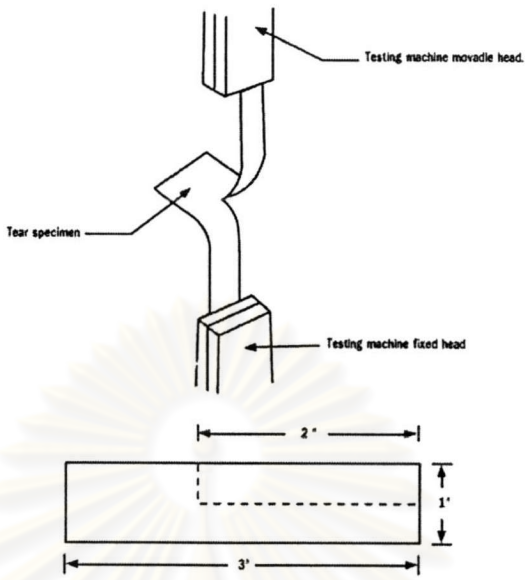

Figure 3.3 Schematic dimension of universal tester and specimen dimension

3.5 Physical Measurement

3.5.1 Shrinkage Testing

The shrinkage value is a useful comparison of various types or grades of fillers, which are added into the rigid PVC opague sheet.

Shrinkage of the specimen was determined as per JIS K 6734-1975. The specimens were prepared by cutting the sheet size 120x120 mm and 100 mm already mark points in both dimensions were measured as illustrated in Figure 3.4. After that the specimen was placed in oven at 100°C for 10 minutes and percent shrinkage value was calculated as follow:

% shrinkage = (Specimen area before – Specimen area after)X100

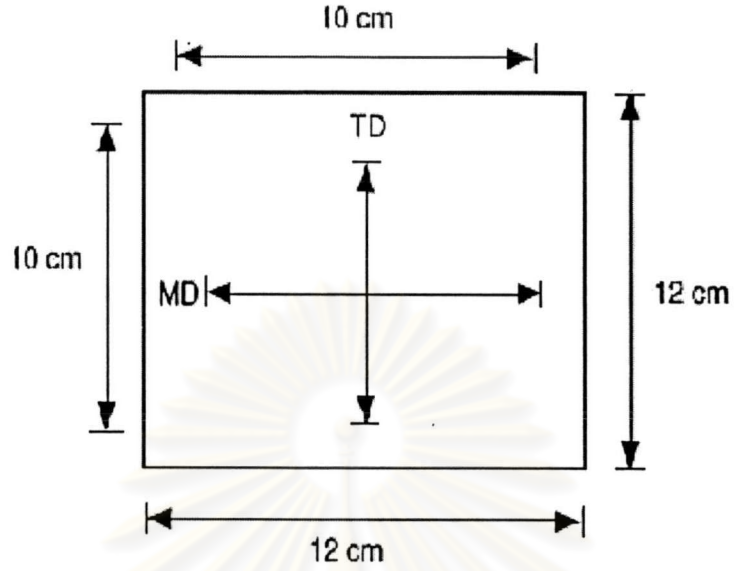

Figure 3.4 Schematic dimension of shrinkage test specimen

3.6 Thermal Measurement

3.6.1 Heat deflection Temperature

Heat deflection temperature (HDT) or heat distortion [24] was measured to study the effects of various types and concentration of filler according to ASTM D648. The specimen was placed on the supports. A load was given on the center of test machine. The temperature in the chamber was raised at the rate of 2 °C ±0.2 °C/min. The temperature at which the specimen was bent or softened was detected as the deflection temperature.

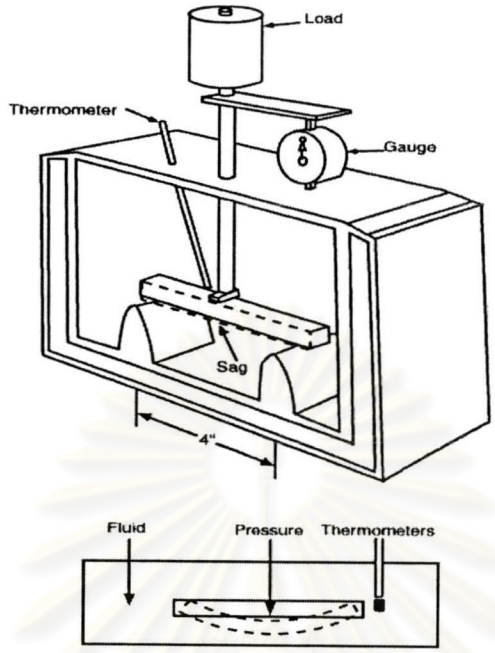

Figure 3.5 Schematic dimension of HDT test machine and specimen.

3.7 Morphology of the Fractured Surface

The scanning electron microscopy (SEM) [23, 24] was used to investigate the fracture surface of the rigid poly(vinyl chloride) opaque sheet containing different modified fillers. Samples for SEM were mounted on an SEM stubs using a double-sided adhesive tape and the fractured specimens were coated with gold.