

## CHAPTER III

### EXPERIMENTAL

#### 3.1 Materials

1. High Impact Polystyrene (HIPS)
  - HIPS resin : TPI
  - Flame retardant grade : DOW, ATO CHEM
2. Tetrabromobisphenol A (BA 59)
  - : Great Lakes Chem Corp.
3. Decabromodiphenyl oxide (DE 83-R)
  - : Great Lakes Chem Corp.
4. Octabromodiphenyl oxide (DE 79)
  - : Great Lakes Chem Corp.
5. Polydibromostyrene (PDBS-80)
  - : Great Lakes Chem Corp.
6. Antimony Trioxide ( $\text{Sb}_2\text{O}_3$ )
  - : MIKUNI Smelting and Refining Co., Ltd.
7. Chlorinated Polyethylene (CPE)
  - : Dow Chemical Ltd.

### 3.2 Apparatus

1. Oxygen index testing ; CEAST 6170/000-6171/000  
(ASTM D2863-91)
2. Single screw extruder machine ; ISHINAKA IRON WORK
3. Injection molding machine ; NISSEI
4. Universal testing machine ; INSTRON 4301
5. Impact testing machine ; CEAST 6545/000
6. Melt flow index testing machine ; CEAST 6841/000
7. Scanning Electron Microscope ; Model JSM-6400
8. Two Roll Mill ; NISHIMURA NO. 89-324

### 3.3 Preparation of flame retardant HIPS

The HIPS compositions were prepared using the components (listed in Table 3-1 to 3-4) in the following manner. Flame retardant, synergist and chlorinated polyethylene were combined with the HIPS resin according to the formulation indicated in Table 3-1 to 3-4 and were dry-blended by shaking them together for 2 minutes in a plastic bag and passed through a single screw extruder. The barrel and melt temperatures were kept below 200 °C.

The final blends were molded on a NISSEI injection-molding machine with the following processing conditions :

- cycle time 61 seconds.
- injection time 15 seconds.
- curing time 45 seconds.
- cycle start 1 seconds.
- melt temperature 210 °C.
- mold temperature 40 °C.
- injection pressure, 450-psi hydraulic oil, 800 psi on the plastic in the barrel.
- hold pressure, 8000 - psi pressure in the barrel, 500-psi mold-cavity pressure.

**TABLE 3-1** HIPS compound containing DE 83-R and flame retardant additives.  
(Unit - part per hundred)

FORMULATIONS	1	2	3	4	5	6	7	8	9	10	11
HIPS resin	100	100	100	100	100	100	100	100	100	100	100
DE 83-R (Decabromodiphenyl oxide)	12	12	12	12	12	13.5	13.5	13.5	15	15	15
Sb <sub>2</sub> O <sub>3</sub>		3	3	4	5	3	4	5	3	4	5
CPE			10	10	10	10	10	10	10	10	10

**TABLE 3-2** HIPS compound containing DE 79 and flame retardant additives.  
(Unit - part per hundred)

FORMULATIONS	12	13	14	15	16	17	18	19	20	21	22
HIPS resin	100	100	100	100	100	100	100	100	100	100	100
DE 79 (Octabromodiphenyl oxide)	12	12	12	12	12	13.5	13.5	13.5	15	15	15
Sb <sub>2</sub> O <sub>3</sub>		3	3	4	5	3	4	5	3	4	5
CPE			10	10	10	10	10	10	10	10	10

**TABLE 3-3** HIPS compound containing BA 59 and flame retardant additives.  
(Unit - part per hundred)

FORMULATIONS	23	24	25	26	27	28	29	30	31	32	33
HIPS resin	100	100	100	100	100	100	100	100	100	100	100
BA 59 (Tetrabromobisphenol A)	12	12	12	12	12	13.5	13.5	13.5	15	15	15
Sb <sub>2</sub> O <sub>3</sub>		3	3	4	5	3	4	5	3	4	5
CPE			10	10	10	10	10	10	10	10	10

**TABLE 3-4** HIPS compound containing PBDS-80 and flame retardant additives.  
(Unit - part per hundred)

FORMULATIONS	34	35	36	37	38	39	40	41	42	43	44
HIPS resin	100	100	100	100	100	100	100	100	100	100	100
PBDS-80 (Polydibromostyrene)	12	12	12	12	12	13.5	13.5	13.5	15	15	15
Sb <sub>2</sub> O <sub>3</sub>		3	3	4	5	3	4	5	3	4	5
CPE			10	10	10	10	10	10	10	10	10

### 3.4 Determination of flammability and mechanical property

All of specimens were prepared according to standard ASTM procedures and test condition indicated in Table 3-5.

Table 3-5 Testing conditions for HIPS compounds.

Properties	Unit	Test method	Test condition
Flammability : Limiting Oxygen Index	%	ASTM D-2863	100 x 7 x3 mm.
Melt flow index	g/10 min	ASTM D-1238	200 °C, 5 Kg
Izod impact strength	kg-cm/cm	ASTM D-256	1/4" at 23 °C
Tensile strength	kg/cm <sup>2</sup>	ASTM D-638	23 °C
Tensile modulus	kg/cm <sup>2</sup> × 10 <sup>4</sup>	ASTM D-638	23 °C
Flexural strength	kg/cm <sup>2</sup>	ASTM D-790	23 °C
Flexural modulus	kg/cm <sup>2</sup> × 10 <sup>4</sup>	ASTM D-790	23 °C
Vicat softening temperature	°C	ASTM D-1525	1/8 " at 1 kg

### 3.4.1 Flame Properties Test of HIPS

Flame retardancy was measured as Limiting Oxygen Index (LOI), which is a method for the determination of the relative flammability of polymeric materials. This test conformed to ASTM D-2863-91[15]. A material in the form of thin strip with the dimension of 100 x 7x 3 mm is clamped vertically and ignited at the top, so that it burns in a candle-like manner in an upward flowing mixture of oxygen and nitrogen. The oxygen concentration of the gas is then adjusted until the minimum level for sustained burning is reached. LOI is especially useful for preliminary flame retardant studies or comparative evaluations. The main advantages of LOI are that numerical data are obtained and the LOI value usually shows a linear relationship with the concentration of flame retardant added [16]. In this study, LOI values were determined of each formulation in an oxygen index apparatus is shown in Figure 3-1.

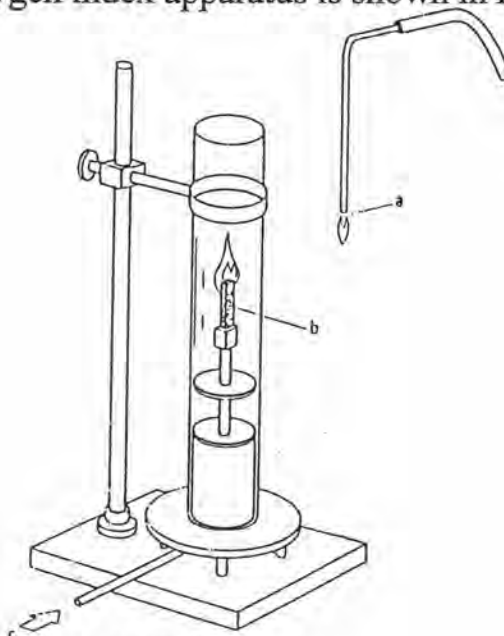


Figure 3-1 Oxygen index apparatus.

a: pilot flame, b: burning specimen, c:  $N_2 / O_2$  supply

Another method for flammability testing is UL-94 standard [16]. UL-94 contains test procedures for both horizontally and vertically positioned solid plastic test specimens in the form of rods. There are two methods ;

HB test : The test specimen is clamped with its longitudinal axis horizontal and a Bunsen burner flame is applied to its free end for 30 sec, the burning rate of the test specimen should not exceed a maximum value dependent on its thickness or that specimen extinguishes itself after removal of the flame.

V test : There is double Bunsen burner flame in the vertical dimension and lists the criteria for each V classification as Table 3-6.

Table 3-6 UL-94 vertical burn test

Rating *	V-0	V-1	V-2
Max individual burn time	≤ 10	≤ 30	≤ 30
Total of 5 specimens	≤ 50	≤ 250	≤ 250
Glow time after second ignition	≤ 30	≤ 60	≤ 60
Ignites cotton	NO	NO	Yes

\*Rating criteria are reported in seconds.

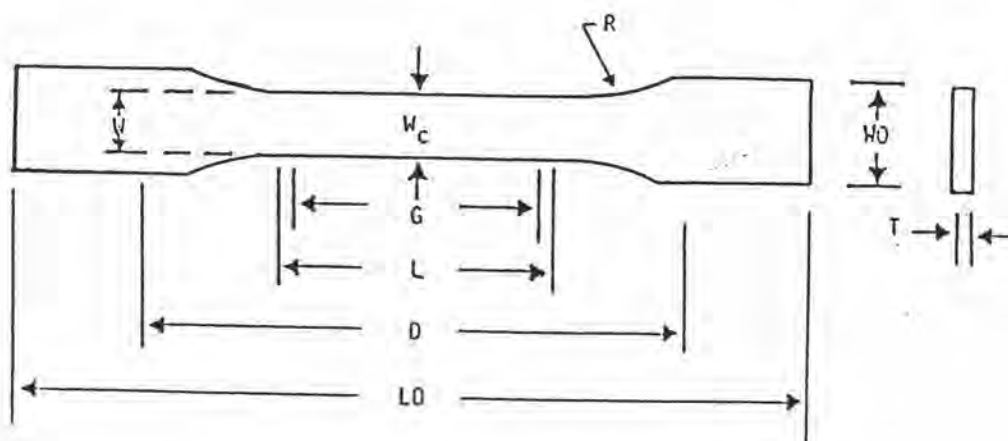


### 3.4.2 Mechanical property testing

Mechanical properties of the thermoplastic were measured by following the ASTM test methods as follows :

ASTM D638 : Standard test method for tensile properties.

The test specimens (type I ) dimension is shown in Figure 3-2.



W : 13 mm     $W_0$  : 19 mm    G : 50 mm    R : 76 mm  
 L : 57 mm    L : 165 mm    D : 115 mm

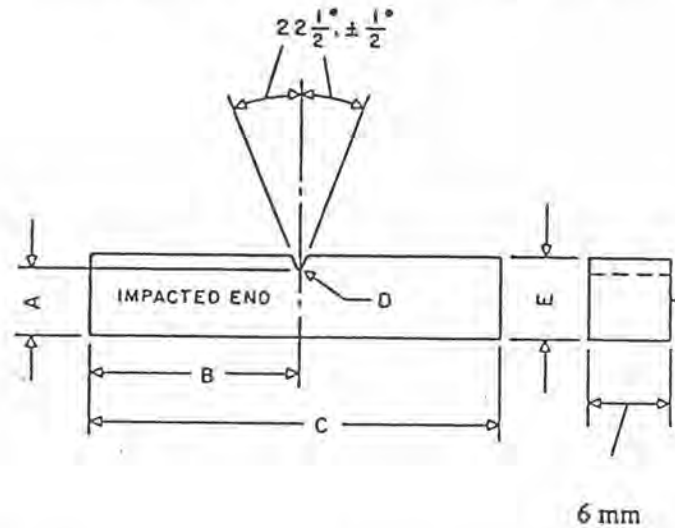
Figure 3-2 Schematic of tensile test specimen (type I).

The tensile testing conditions were as follows :

Temperature :                    23.0 °C  
 Relative humidity :            50.0 %  
 Distance between grips :    115 mm  
 Gage length :                    50 mm

ASTM D256 : Standard test method for impact resistance.

The test specimens dimension for Cantilever Beam (Izod Type) test is shown in Figure 3-3.



Unit : mm

A :  $10.16 \pm 0.05$

D :  $0.25 \pm 0.05$

B : 32.00 max., 31.50 min.

E :  $12.70 \pm 0.15$

C : 63.50 max., 63.30 min.

**Figure 3-3** Schematic of Izod type test specimen.

The machine parameters and testing conditions of the impact test are listed below :

Temperature :	23.0	°C
Relative humidity :	50	%
Depth of specimen :	10.16	mm
Pendulum capacity :	11.0	J