CHAPTER IV

RESULTS AND DISCUSSION

4.1 Physical properties and flammability of commercial HIPS

The addition of a flame retardant into HIPS normally affects some properties of HIPS. Comparison of HIPS without a flame retardant to the one with a flame retardant produced by ATO and DOW indicated that there are several different properties as shown in Table 4-1. Obviously, LOI is raised to 25. Eventhough other properties are also different from HIPS resin, they may be caused by several additives. For examples, izod impact strength and melt-flow rate can be improved by adding suitable additives [12]. Vicat softening temperature as well as the other properties indicated in Table 4-1 depend on the native properties of the base resin. However, vicat softening temperature is very important that must be brought into consideration for electrical application. In this research, only one type of HIPS resin was used and no other additives except antimony trioxide and chlorinated polyethylene were added.

Accordingly, the flame retardancy will be considered in concurrent with melt-flow rate, izod impact strength and vicat softening temperature.

Table 4-1 Properties of HIPS without flame retardant and commercial HIPS with flame retardant.

Properties	HIPS	HIPS with a flame retardant	
		by ATO	by DOW
Melt-flow rate (g/10 min)	3.57	6.85	4.27
Izod impact strength (Kg.cm/cm)	12.3	9.52	10.11
Tensile strength (Kg/cm^2)	218	243	207
Elongation (%)	64	30	60
Flexural strength (Kg/cm^2)	384	302	267
Flexural modulus ($Kg/cm^2 x10^4$)	1.98	1.65	1.76
Vicat softening temperature (°C)	93.2	101.9	98.3
Flammability, UL-94	HB	V-0 at 1/16 in.	V-0 at 1/16 in.
LOI	18.8	25.5	25.0

4.2 HIPS compound containing DE 83-R and flame retardant additive

Table 4-2 shows the physical and mechanical properties of HIPS resin containing DE 83-R as a flame retardant. By the addition of 12 phr. of DE 83-R. LOI of HIPS was dramatically raised from 18.8 to 22.0 while all the values of other properties decrease except melt-flow rate and vicat softening temperature. When 3 phr. of antimony trioxide (formulation 2) was added. LOI was raised even more to 23.5 while melt-flow rate and vicat softening temperature are about the same as that of Formulation 1 but izod impact strength was decreased. In Formulation 3, 10 phr. of CPE was added and it was found that the melt-flow rate increases from 4.7 to 6.04 g/10 min. The function of CPE is to inhibit the dripping of burning material and can also improve the flowability of the resin. Furthermore, LOI has been raised from 23.5 to 24.0 as well. Consequently, this indicates that the combination of the flame retardant, antimony trioxide and CPE enhance the ignition-resistance as shown by LOI. From the Formulation 1 to 3, the LOI values were all below 25 which could not reach V-0 rating in UL-94 test.

Thus, the amount of flame retardant and synergist were varied in order to obtain their appropriates proportion which will provide LOI of 25 while maintain other acceptable properties. Comparing Formulation 3 to ll, the ignition resistance, as measured by LOI, tended to increase with increasing the amount of flame retardant and synergist. While the melt-flow rate and vicat softening temperature were maintained about the same, but the impact resistance seemed to drop. Formulation 7 which contains 13.5 phr DE 83-R, 4 phr Sb_2O_3 and 10 phr CPE is better than the others because it shows the acceptable LOI with the best impact strength (Figure 4-2).

TABLE 4-2 Properties of HIPS compound containing DE 83-R (Unit-part per hundred).

FORMULATIONS	**0**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**
HIPS resin	100	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_2O_3			3	3	4	5	3	4	5	3	4	5
CPE				10	10	10	10	10	10	10	10	10
PROPERTIES												
Melt-flow rate (g/10 min)	3.57	4.85	4.70	6.04	6.06	6.12	6.21	6.06	6.25	6.37	6.24	6.36
Izod impact strength (kg. cm/cm)	12.3	10.55	9.85	8.54	8.49	8.32	8.49	8.16	8.11	7.94	7.78	7.62
Tensile strength (kg/cm^2)	218	215	214	200	195	194	205	207	205	204	201	200
Elongation (%)	64	59	42	67	67	65	73	57	65	63	66	66
Flexural strength (kg/cm^2)	384	284	279	257	253	253	254	256	259	255	257	256
Flexural modulus ($kg/cm^2 x 10^4$)	1.98	1.60	1.62	1.43	1.41	1.41	1.41	1.44	1.43	1.41	1.43	1.43
Vicat softening temp (^{o}C)	93.2	95.0	95.6	94.2	94.1	94.6	94.4	94.7	94.6	94.8	95.0	94.3
LOI	18.8	22.0	23.5	24.0	24.0	24.5	24.5	25.0	25.0	25.5	25.5	26.0

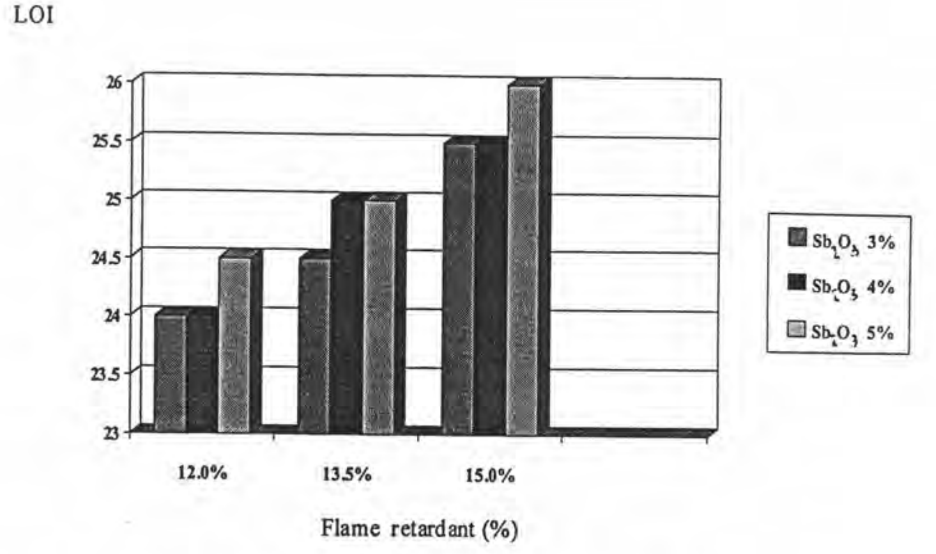


Figure 4-1 Effect of the percentage of DE 83-R and synergist on LOI.

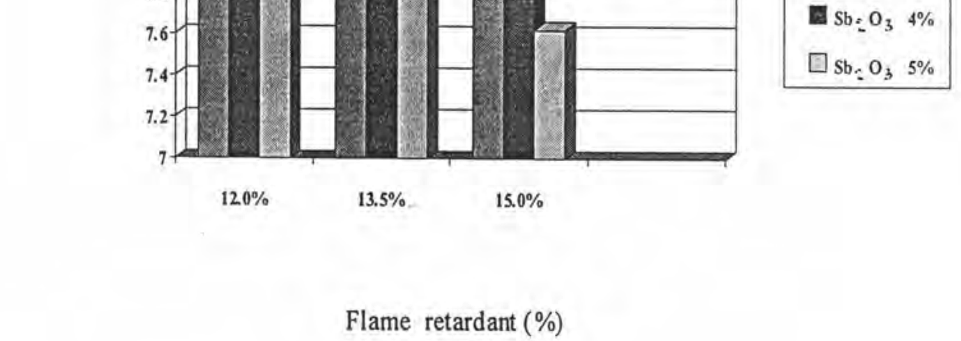


Figure 4-2 Effect of the percentage of DE 83-R and synergist on Izod impact.

4.3 HIPS compound containing DE 79 and flame retardant additive

In the same manner, Formulations 12 to 22 (Table 4-3) have been prepared using DE 79 as a flame retardant. The similar trend of physical and mechanical properties as in the case of DE 83-R has been obtained, except vicat softening temperature which is lower than the base resin comparing to HIPS resin with DE 83-R Figure 4-3 to 4-4 show the effect of the percentage of DE 79 and synergist on LOI and izod impact strength. Formulation 19 which contains 13.5 phr DE 79, 5 phr Sb_2O_3 and 10 phr CPE is better than the others because it shows the acceptable LOI with the best impact strength .

TABLE 4-3 Properties of HIPS compound containing DE 79 (Unit-part per hundred).

FORMULATIONS	**0**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**	**21**	**22**
HIPS resin	100	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_2O_3			3	3	4	5	3	4	5	3	4	5
CPE				10	10	10	10	10	10	10	10	10
PROPERTIES												
Melt-flow rate (g/10 min)	3.57	6.74	6.55	8.14	8.20	9.01	8.21	8.98	9.09	8.37	9.06	9.20
Izod impact strength (kg. cm/cm)	12.3	10.17	9.90	9.47	8.92	8.21	8.81	8.76	8.65	8.32	8.23	8.16
Tensile strength (kg/cm^2)	218	221	201	198	197	193	191	188	172	186	184	192
Elongation (%)	64	64	61	56	54	64	69	63	61	54	64	50
Flexural strength (kg/cm^2)	384	295	286	263	265	263	266	269	254	251	255	256
Flexural modulus ($kg/cm^2 x10^4$)	1.98	1.60	1.59	1.42	1.41	1.41	1.41	1.40	1.43	1.41	1.44	1.45
Vicat softening temp (^{o}C)	93.2	88.6	88.6	90.6	90.9	90.1	87.4	90.1	90.1	90.2	90.3	90.3
LOI	18.8	22.5	24.0	24.0	24.0	24.5	24.5	24.5	25.0	25.0	25.0	25.0

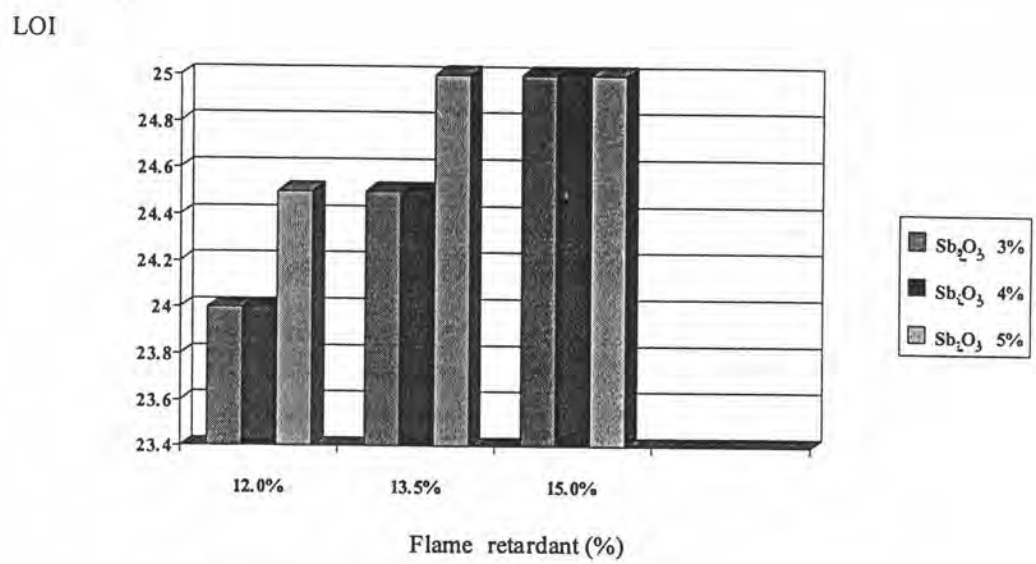


Figure 4-3 Effect of the percentage of DE 79 and synergist on LOI.

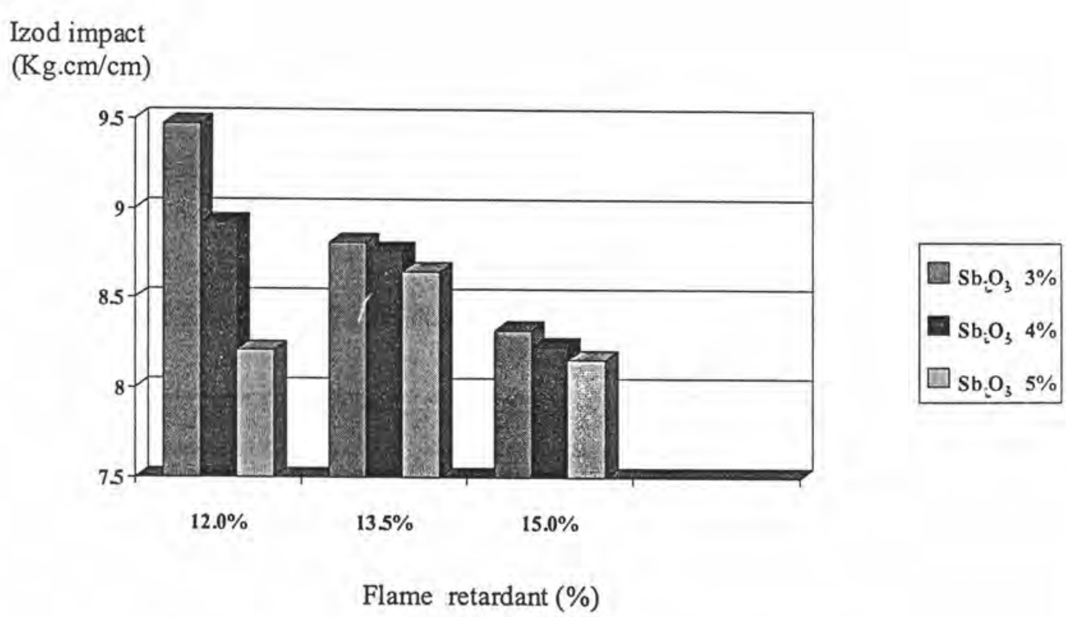


Figure 4-4 Effect of the percentage of DE 79 and synergist on Izod impact.

4.4 HIPS compound containing BA 59 and flame retardant additive

When BA 59 is used, Formulations 23 to 33 (Table 4-4), the similar trend of physical and mechanical properties is still observed. However, vicat softening temperature is much lower than the base resin comparing to HIPS resin with the first two types of flame retardants. Figure 4-5 to 4-6 show the effect of the percentage of BA 59 and synergist on LOI and izod impact strength. Formulation 32 which contains 15 phr BA 59, 4 phr Sb_2O_3 and 10 phr CPE is better than the others because it shows the acceptable LOI with the best impact strength.

TABLE 4-4 Properties of HIPS compound containing BA 59 (Unit- part per hundred).

FORMULATIONS	0	23	24	25	26	27	28	29	30	31	32	33
HIPS resin	100	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_2O_3			3	3	4	5	3	4	5	3	4	5
CPE				10	10	10	10	10	10	10	10	10
PROPERTIES												
Melt-flow rate (g/10 min)	3.57	8.94	9.86	10.05	10.04	10.20	10.20	10.25	10.30	10.30	10.40	10.50
Izod impact strength (kg. cm/cm)	12.3	10.41	9.52	8.68	8.34	7.62	7.40	7.25	7.04	7.36	7.08	6.06
Tensile strength (kg/cm^2)	218	204	203	186	180	187	182	182	183	204	194	189
Elongation (%)	64	55	51	38	39	30	39	51	68	53	69	69
Flexural strength (kg/cm^2)	384	297	288	264	262	262	259	257	256	255	254	251
Flexural modulus ($kg/cm^2 x10^4$)	1.98	1.64	1.66	1.45	1.44	1.42	1.45	1.41	1.42	1.45	1.43	1.43
Vicat softening temp (^oC)	93.2	83.9	85.0	84.6	85.3	85.6	83.5	84.4	85.2	83.0	82.4	83.5
LOI	18.8	22.0	23.5	24.0	24.0	24.5	24.5	24.5	24.5	24.5	25.0	25.0

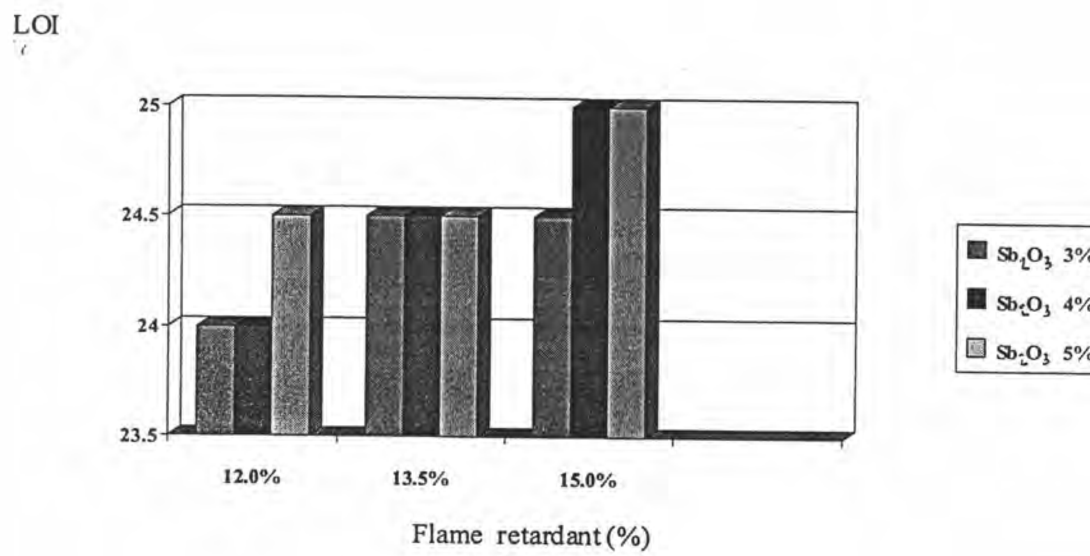


Figure 4-5 Effect of the percentage of BA 59 and synergist on LOI.

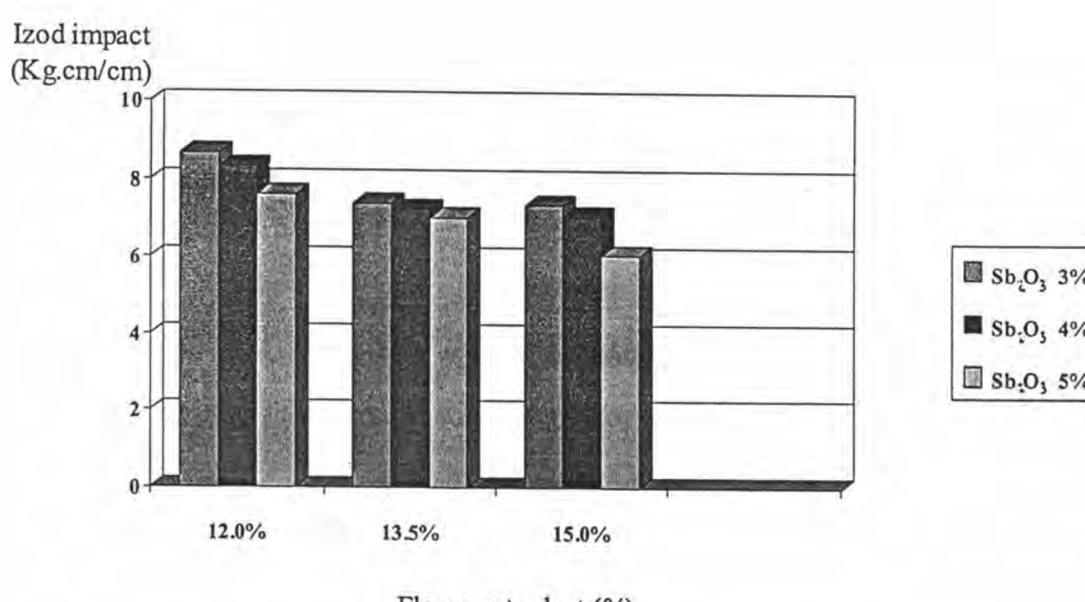


Figure 4-6 Effect of the percentage of BA 59 and synergist on Izod impact.

4.5 HIPS compound containing PBDS-80 and flame retardant additive

Formulations 34 to 44 (Table 4-5) have been prepared using PBDS-80 as a flame retardant. The similar trend of BA 59 has been observed as well except vicat softening temperature which is much higher than the base resin comparing to HIPS resin with those three flame retardants. Eventhough the melt-flow rate is also higher than the base resin similar to those three flame retardants, the increment is very small. Figure 4-7 to 4-8 show the effect of the percentage of PBDS-80 and synergist on LOI and izod impact strength. Formulation 43 which contains 15 phr PBDS-80, 5 phr Sb_2O_3 and 10 phr CPE is better than the others because it shows the acceptable LOI with the best impact strength.

TABLE 4-5 Properties of HIPS compound containing PBDS-80 (Unit-part per hundred).

FORMULATIONS	0	34	35	36	37	38	39	40	41	42	43	44
HIPS resin	100	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_2O_3			3	3	4	5	3	4	5	3	4	5
CPE				10	10	10	10	10	10	10	10	10
PROPERTIES												
Melt-flow rate (g/10 min)	3.57	3.70	3.67	4.34	4.36	4.19	4.45	4.55	4.35	4.50	4.56	4.55
Izod impact strength (kg. cm/cm)	12.3	7.40	6.96	6.58	6.31	6.26	6.04	5.39	5.39	5.17	5.11	4.68
Tensile strength (kg/cm^2)	218	211	223	203	190	200	196	198	199	201	198	200
Elongation (%)	64	73	66	61	65	72	70	64	66	63	63	61
Flexural strength (kg/cm^2)	384	292	288	270	267	270	271	265	271	274	275	271
Flexural modulus ($kg/cm^2 x10^4$)	1.98	1.65	1.67	1.48	1.48	1.53	1.50	1.45	1.53	1.53	1.51	1.54
Vicat softening temp (^oC)	93.2	97.6	97.7	97.3	97.3	97.2	97.3	97.0	96.9	96.4	96.5	96.6
LOI	18.8	20.5	23.0	24.0	24.0	24.0	24.0	24.0	24.5	24.5	25.0	25.0

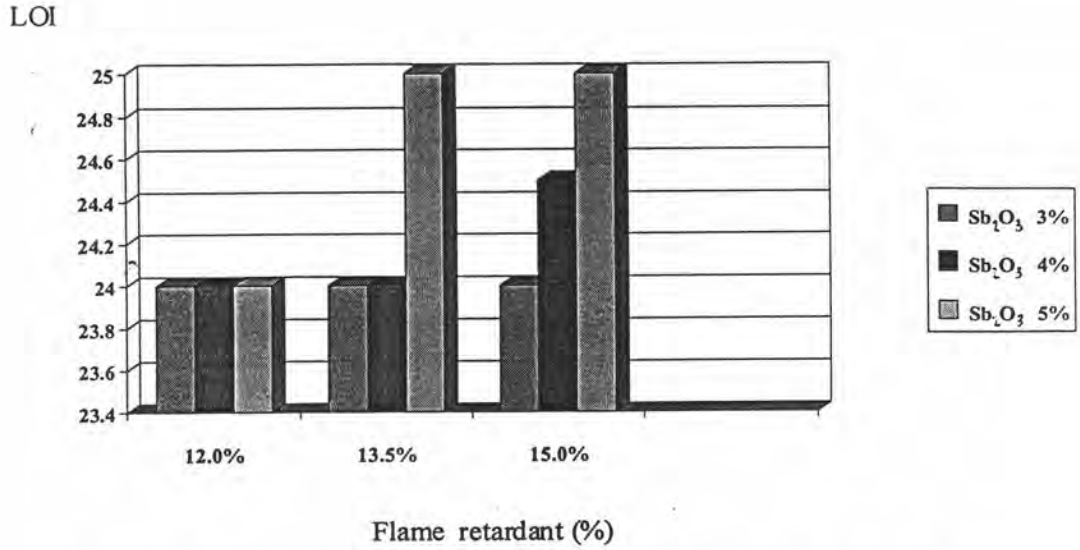


Figure 4-7 Effect of the percentage of PBDS-80 and synergist on LOI.

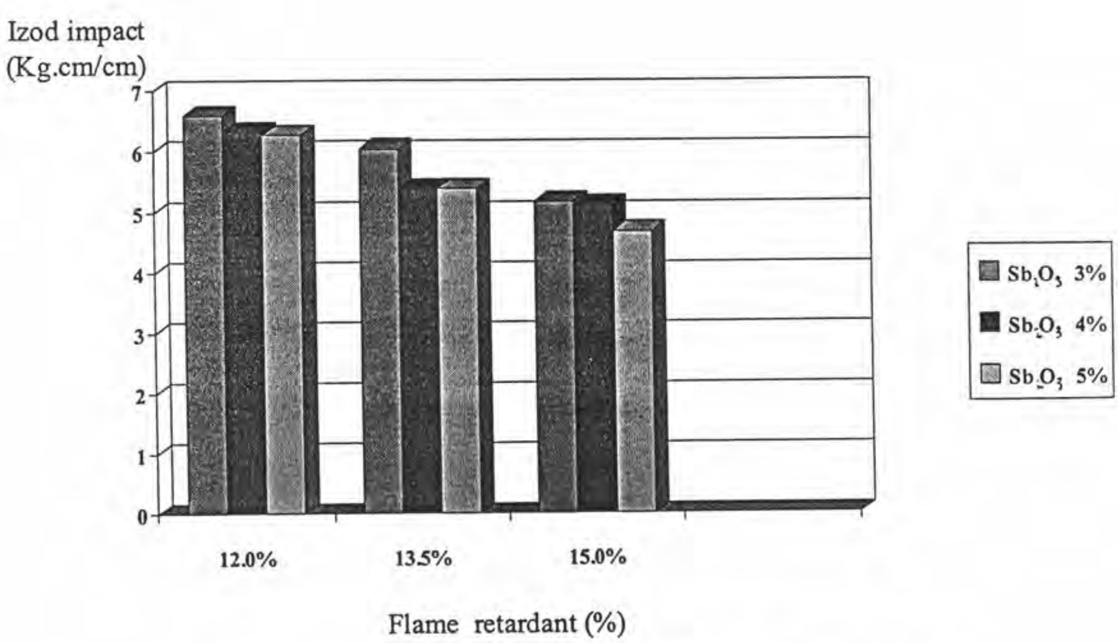


Figure 4-8 Effect of the percentage of PBDS-80 and synergist on Izod impact.

4.6 The effect of molecular structure of the flame retardants on properties of HIPS resin

The molecular structure of all flame retardants used in this research have been shown in Table 4-6. They are different in both molecular weight and percentage of bromine in the molecule. These differences affect the properties of HIPS, particularly, melt-flow rate, izod impact strength, vicat softening temperature and LOI.

Table 4-6 Molecular structures of brominated flame retardants.

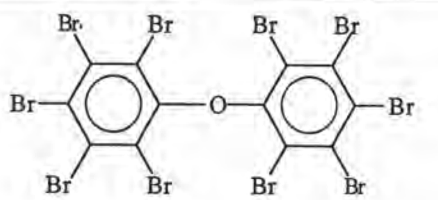
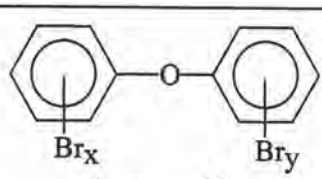
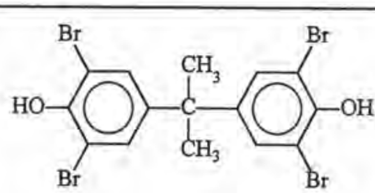
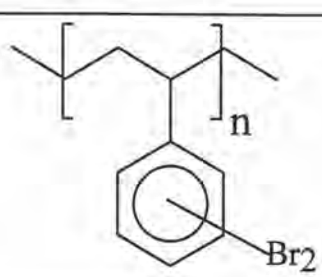
Flame retardants	Molecular structures	Bromine content(%)
Decabromodiphenyl oxide (DE 83-R) Molecular weight 959.2 Melting range 300-315 °C		83.3
Octabromodiphenyl oxide (DE 79) Molecular weight 801.4 Melting range 70-150 °C	 (x + y = 8)	79.8
Tetrabromobisphenol A (BA 59) Molecular weight 543.7 Melting range 179-181 °C		58.8
Polydibromostyrene (PBDS-80) Molecular weight 80,000 Melting range 210-230 °C		59.0

Figure 4-9 shows the effect of various brominated flame retardants on the melt-flow rate of fully formulated ignition-resistant compounds. It shows that the melt-flow rate slightly increases by increasing the amount of flame retardant. It is observed that percentage of antimony trioxide does not significantly affect such trends. The melt-flow rate of HIPS with BA 59 is higher than HIPS containing DE 79, DE 83-R, and PBDS-80, respectively. This is because of the sequence of the molecular weight as follows : BA 59 < DE 79 < DE 83-R < PBDS-80. Melt-flow rate is relatively a melt viscosity. The lower molecular weight substances tend to have a low viscosity. Thus, the lower molecular weight component will be easier to flow than the higher molecular weight.

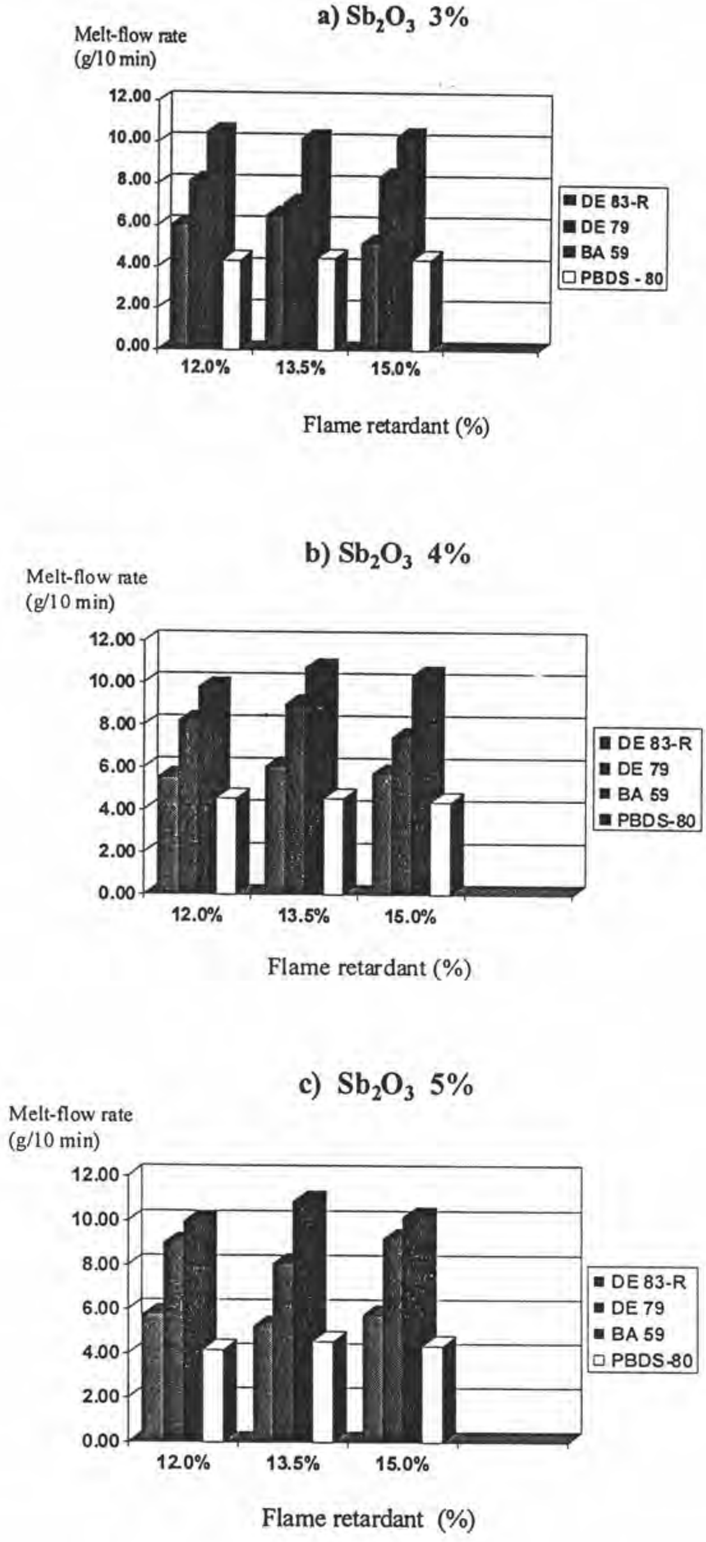


Figure 4-9 Relationship between the Melt-flow rate and the amount of flame retardant in HIPS.

Figure 4-10 shows the effect of various brominated flame retardants on the izod impact strength of fully formulated ignition-resistant compounds. It shows that the izod impact strength is decreased by increasing of the percent flame retardant and synergist. The izod impact strength of DE 79 is higher than DE 83-R, BA 59 and PBDS-80 respectively. The izod impact strength is increased by the lower molecular weight flame retardants, with the exception of BA 59.

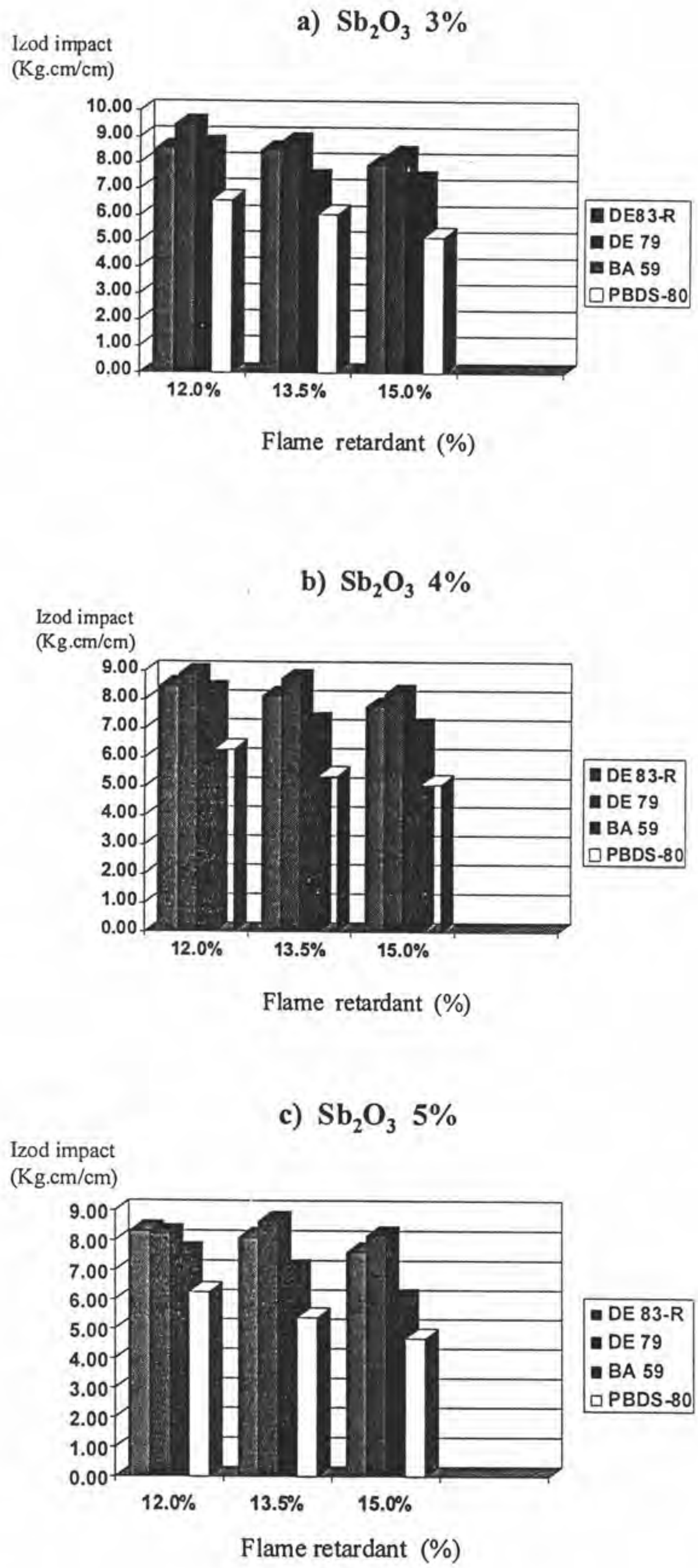


Figure 4-10 Relationship between the Izod impact and the amount of flame retardant in HIPS.

Figure 4-11 shows the effect of various brominated flame retardants on the Vicat softening temperature of fully formulated ignition-resistant compounds. From the results, Vicat is relatively independent on the percentage of flame retardant and synergist. It demonstrate that PBDS-80 give a higher value of Vicat than DE 83-R, DE 79, and BA 59 respectively. Due to their decomposition temperature, the higher decomposition temperature is, the higher vicat softening temperature will be.

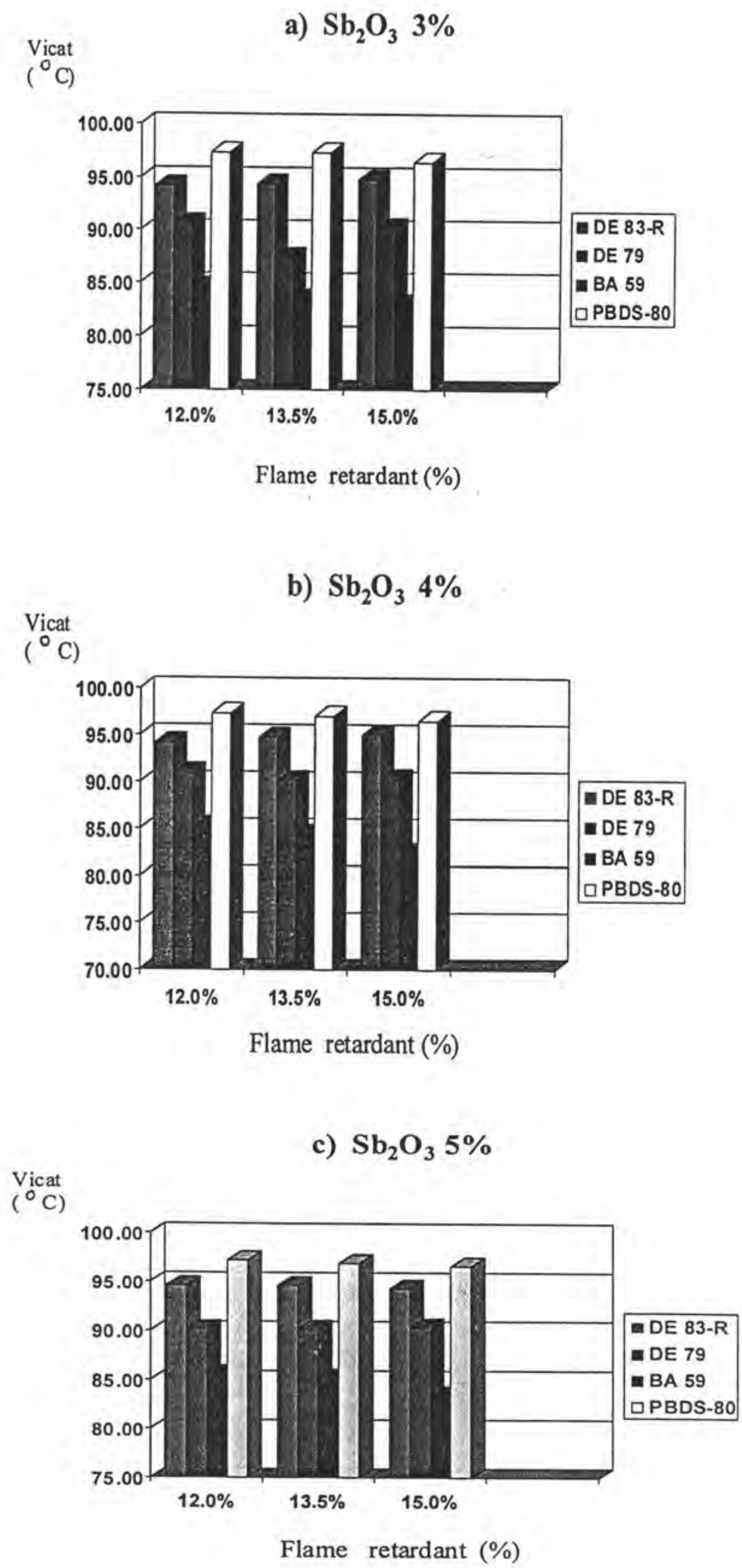
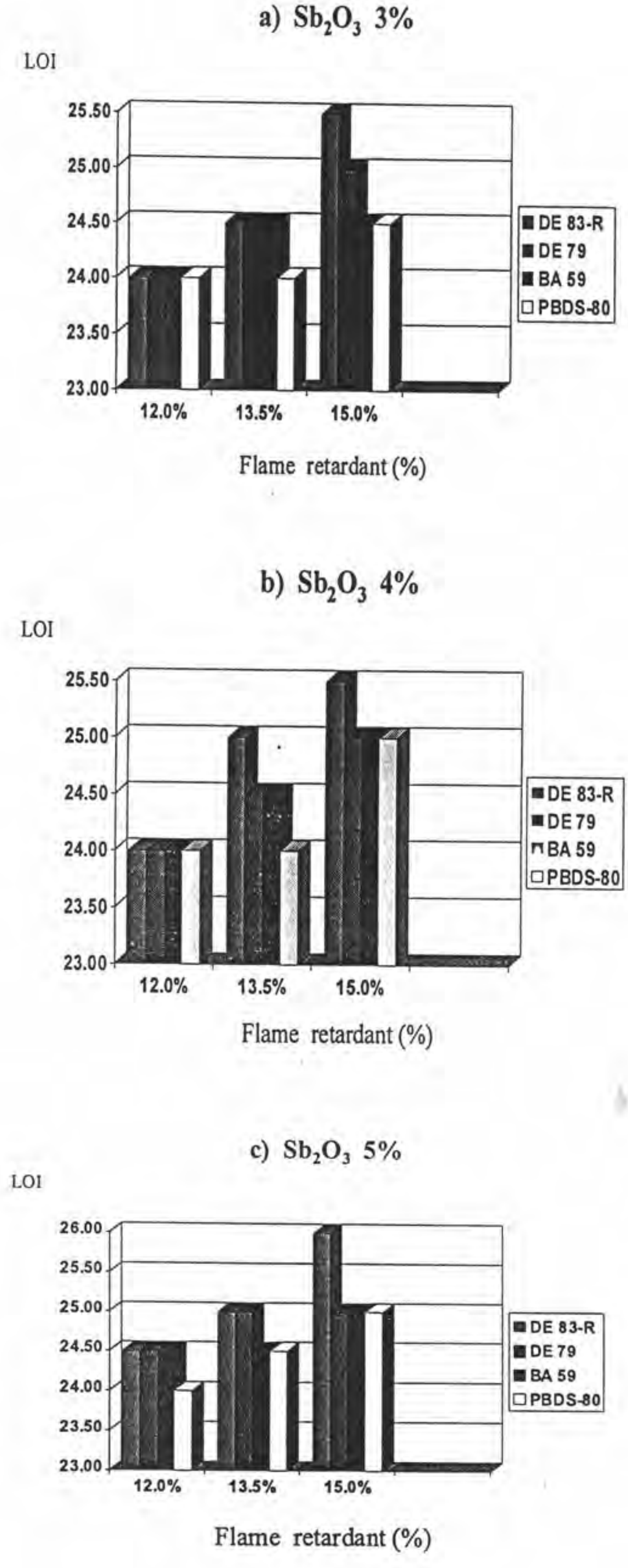


Figure 4-11 Relationship between the Vicat and the amount of flame retardant in HIPS.

Figure 4-12 shows the effect of various brominated flame retardants on the LOI of fully formulated ignition-resistant compounds. It shows that ignition-resistant, as measured by LOI, is increased by increasing the percentage of flame retardant and synergist in the compounds. DE 83-R is more effective flame retardant than DE 79, BA 59, and PBDS-80 because of LOI depends on percentage of bromine content which DE 83-R has a higher bromine content than DE 79, BA 59, and PBDS-80 respectively.



<u>**Figure 4-12**</u> Relationship between the LOI and the amount of flame retardant in HIPS.

4.7 Comparison of the most suitable formulation of each flame retardant

From section 4.2 to 4.5, the most suitable formulation of each flame retardant could be obtained. Table 4-7 exhibits the composition and properties of each of these four formulations. In this case, LOI of all four formulations one the same. Thus melt flow index, izod impact and vicat softening temperature shall be considered. Eventhough Formulation 43 shows very high vicat softening temperature, but melt flow rate and izod impact strength are very low comparing to the other there formulation. It is clearly indicated that PBDS-80 shall not be used to formulate the best flame retardant HIPS: In case of BA 59, quite low vicat softening temperature has been obtained. Therefore Formulation 32 shall not be selected as well. Between Formulation 7 and 19, melt flow rate, izod impact strength and vicat softening temperature must be brought into consideration together. As mentioned before, melt flow rate and izod impact strength can be simply improved by adding certain additives but vicat softening temperature cannot be raised. Formulation 7 containing DE83-R is accordingly the most suitable one. It should be noted that least amount of the flame retardant and Sb_2O_3 is used in this formulation.

Table 4-7 Comparison of the most suitable formulation of each flame retardant.

FORMULATIONS	7	19	32	43
HIPS resin	100	100	100	100
Flame retardant	13.5 (DE 3-R)	13.5 (DE 79)	15.0 (BA 59)	15.0 (PBDS-80)
Sb_2O_3	4	5	4	4
CPE	10	10	10	10
PROPERTIES				
Melt-flow rate (g/10 min)	6.06	9.09	10.40	4.56
Izod impact strength (kg.cm/cm)	8.16	8.65	7.08	5.11
Tensile strength (kg/cm^2)	207	172	194	198
Elongation (%)	57	61	69	63
Flexural strength (kg/cm^2)	256	254	254	275
Flexural modulus (kg/cm^2 x 10^4)	1.44	1.43	1.43	1.51
Vicat softening temperature (°C)	94.7	90.1	82.4	96.5
LOI	25.0	25.0	25.0	25.0

4.8 The effect of CPE content on the physical properties of HIPS compound containing DE 83-R and synergist

CPE content has been varied in order to see its effect on the physical and mechanical properties. This has been shown in Table 4-8. With less CPE content, the melt-flow rate is lower, but the impact strength is higher while the vicat softening temperature seems to be unchanged. Unfortunately, LOI of Formulations 45, 46, 47, and 7 are not available. However, from Formulations 2 and 3 in Table 4-2, 0Formulations 13 and 14 in Table 4-3, Formulations 24 and 25 in Table 4-4, Formulations 35 and 36 in Table 4-5 it was assumed that LOI would not be significantly different for CPE content in the range of 4-10 phr. LOI value is about 0.5-1 unit higher with the increase of 10 phr of CPE.

Table 4-8 Effect of CPE on ignition resistance of HIPS compound containing DE 83-R.

FORMULATIONS	45	46	47	7
HIPS resin	100	100	100	100
DE 83-R	13.5	13.5	13.5	13.5
Sb_2O_3	4	4	4	4
CPE		4	8	10
PROPERTIES				
Melt-flow rate (g/10 min)	4.60	4.94	5.27	6.06
Izod impact strength (kg.cm/cm)	9.36	9.28	9.03	8.16
Tensile strength (kg/cm^2)	197	198	207	207
Elongation (%)	45	58	68	57
Flexural strength (kg/cm^2)	260	256	253	256
Flexural modulus ($kg/cm^2 \times 10^4$)	1.66	1.60	1.54	1.44
Vicat softening temperature (°C)	95.8	95.7	96.7	94.7

Figure 4-13 and 4-14 show the relationship between CPE content with melt-flow rate and izod impact strength respectively.

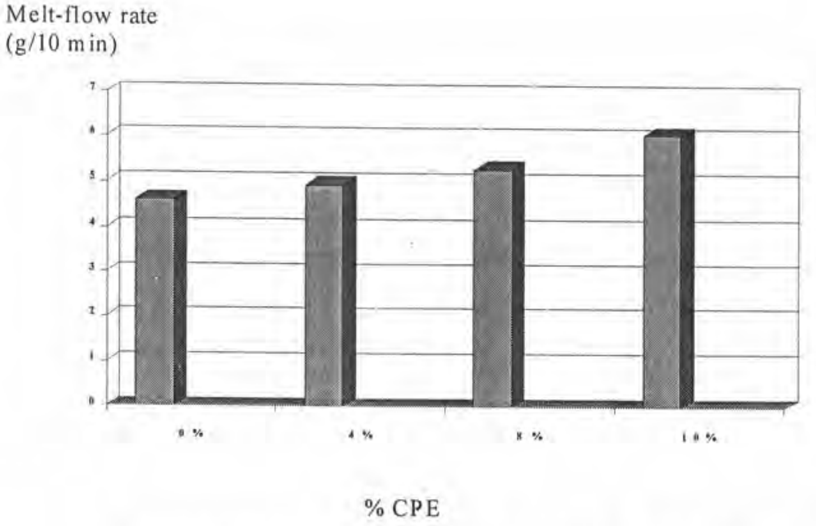


Figure 4-13 Relationship between CPE level and Melt-flow rate

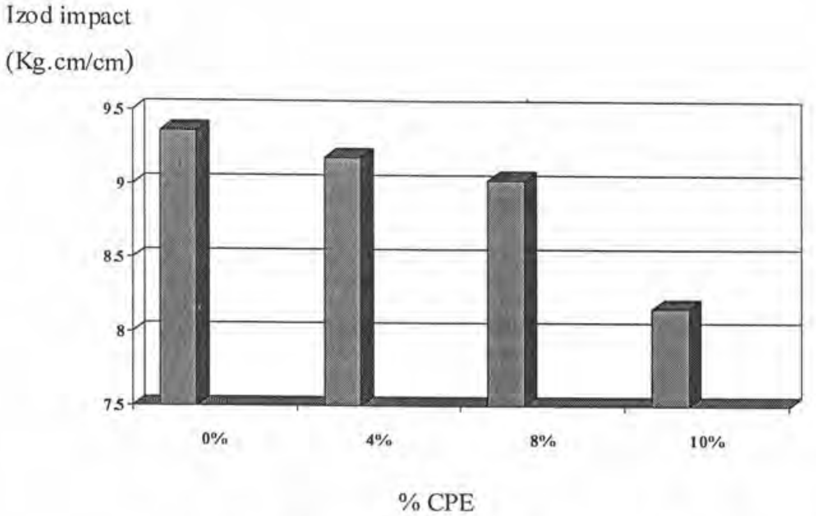


Figure 4-14 Relationship between CPE level and Izod impact strength

4.9 Determination of the dispersion of flame retardant particles in HIPS compound

The dispersion of the flame retardant in HIPS is one of the factors in determining toughness, rheology and the overall performance properties of ignition resistant HIPS. It is thus necessary to disperse the flame retardant evenly in polymer matrix.

Figure 4-15 to 4-29 exhibits the scanning electron micrographs of HIPS with and without flame retardant, Sb_2O_3, and CPE. It has been shown that all the additives can disperse well in HIPS matrix.

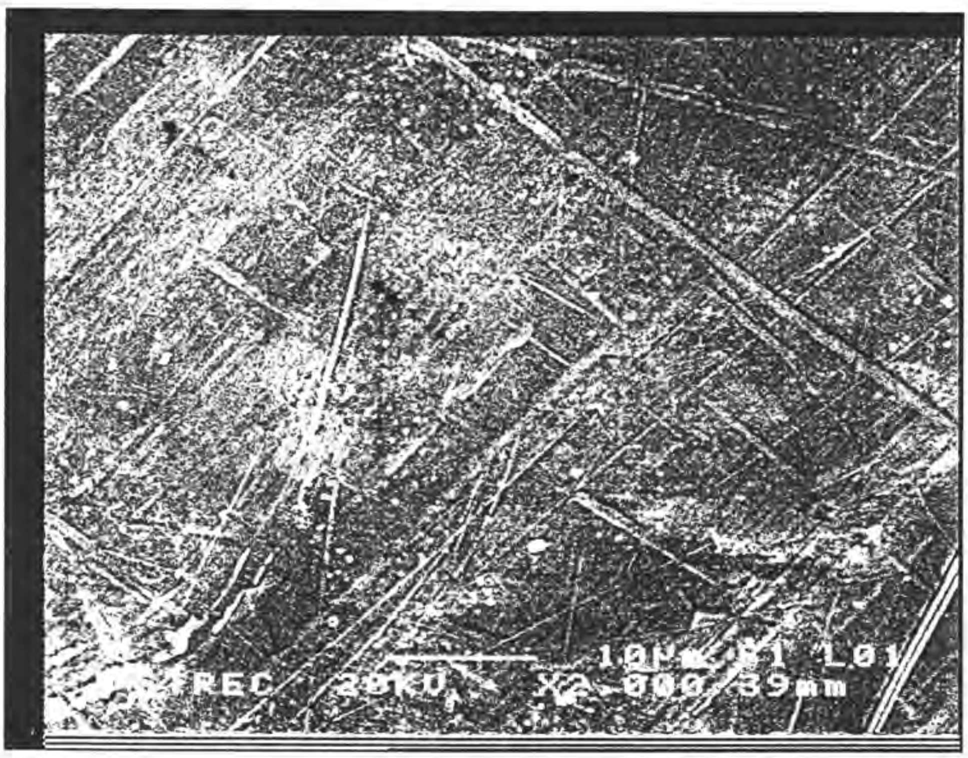


Figure 4-15 Scanning electron micrograph of HIPS resin.

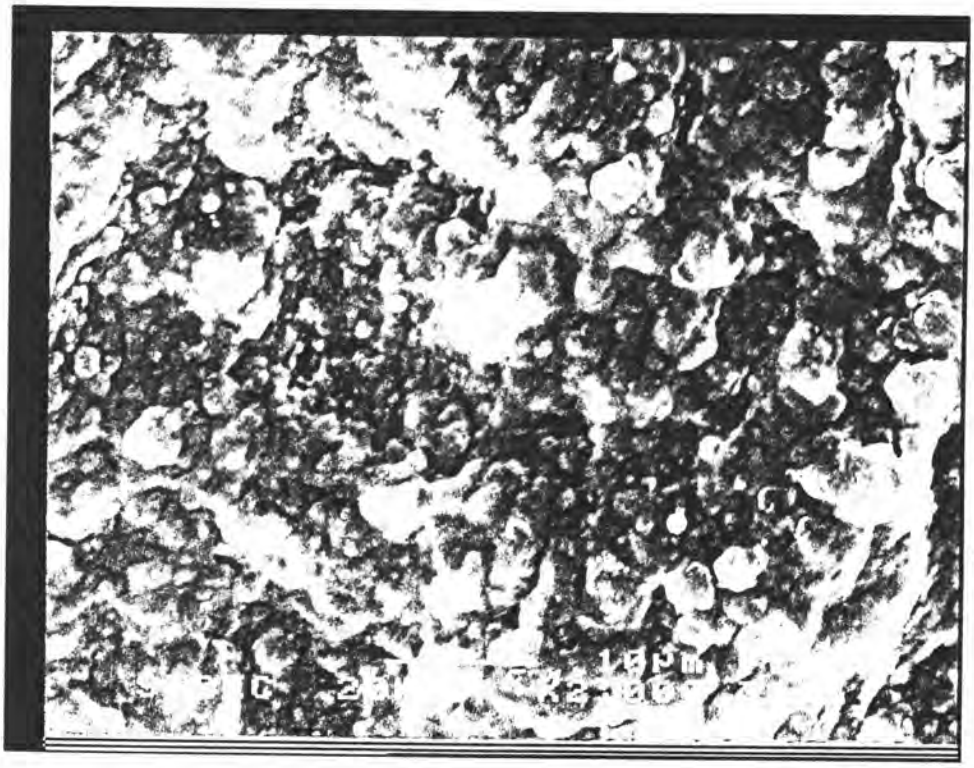


Figure 4-16 Scanning electron micrograph of commercial HIPS resin (DOW).

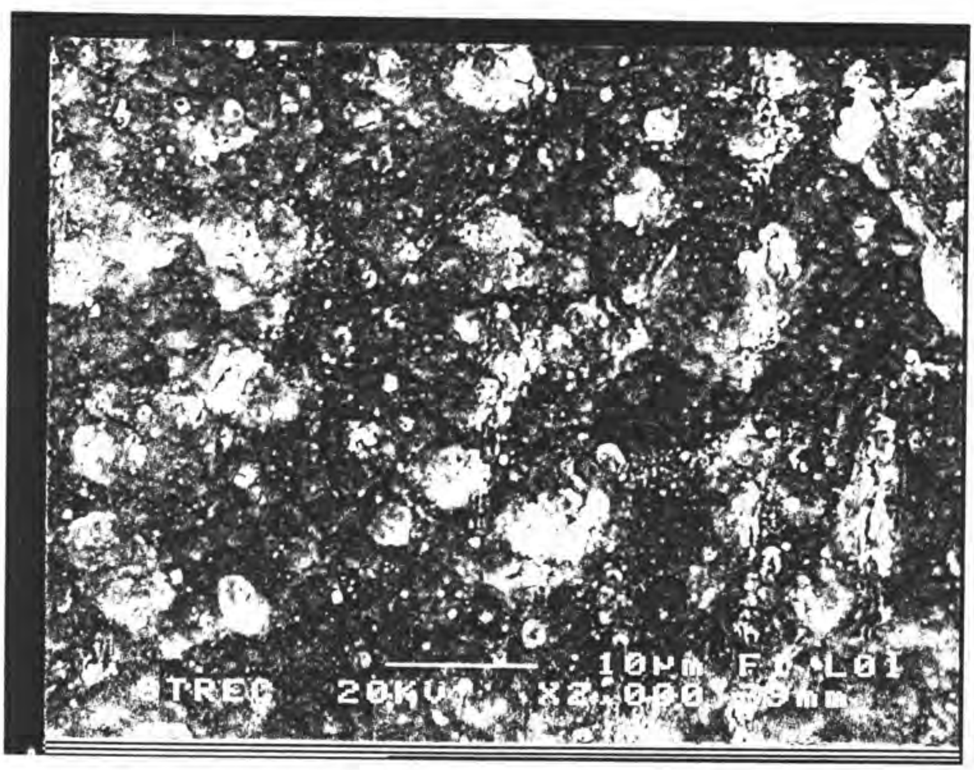


Figure 4-17 Scanning electron micrpgraph of commercial HIPS resin (ATO CHEM).

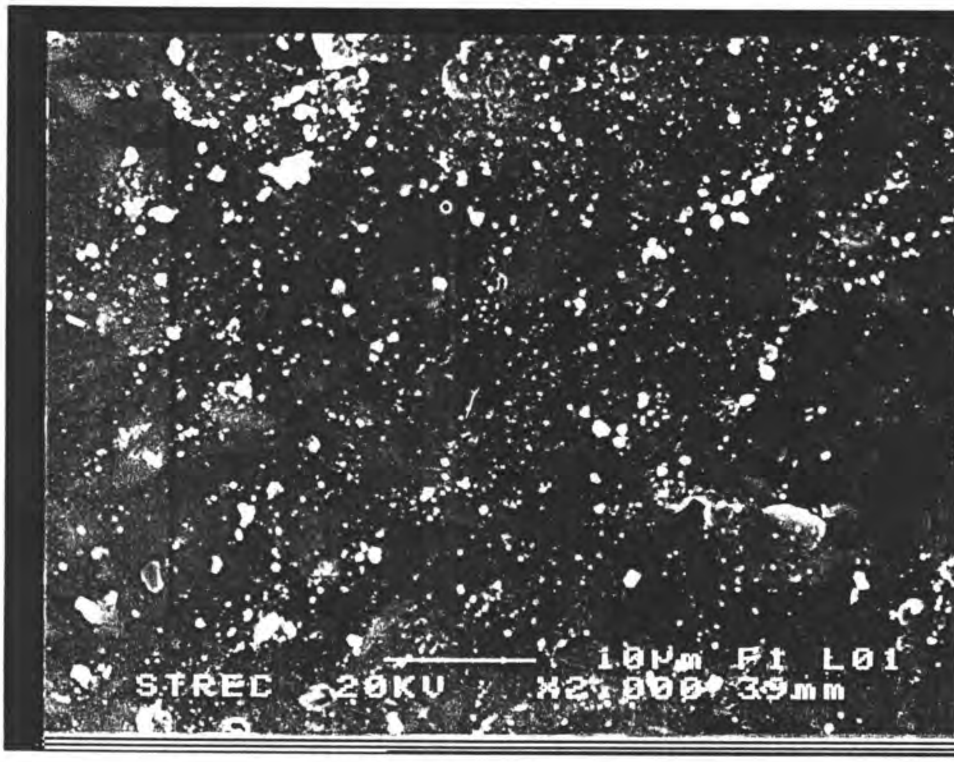


Figure 4-18 Scanning electron micrograph of HIPS with BA 59.

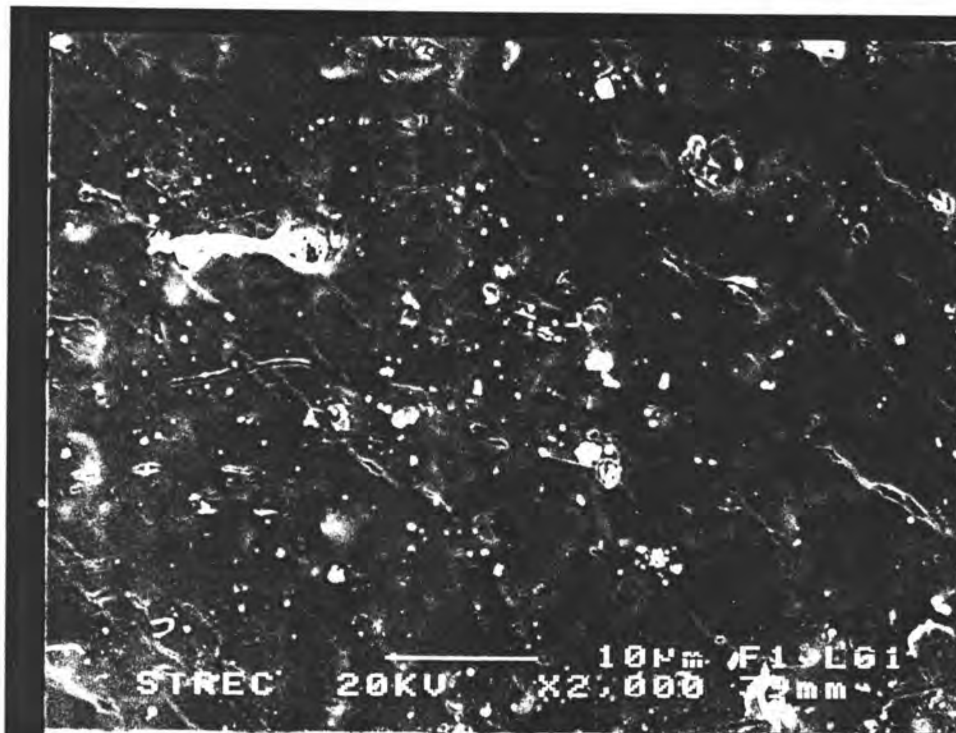


Figure 4-19 Scanning electron micrograph of HIPS with DE 83-R.

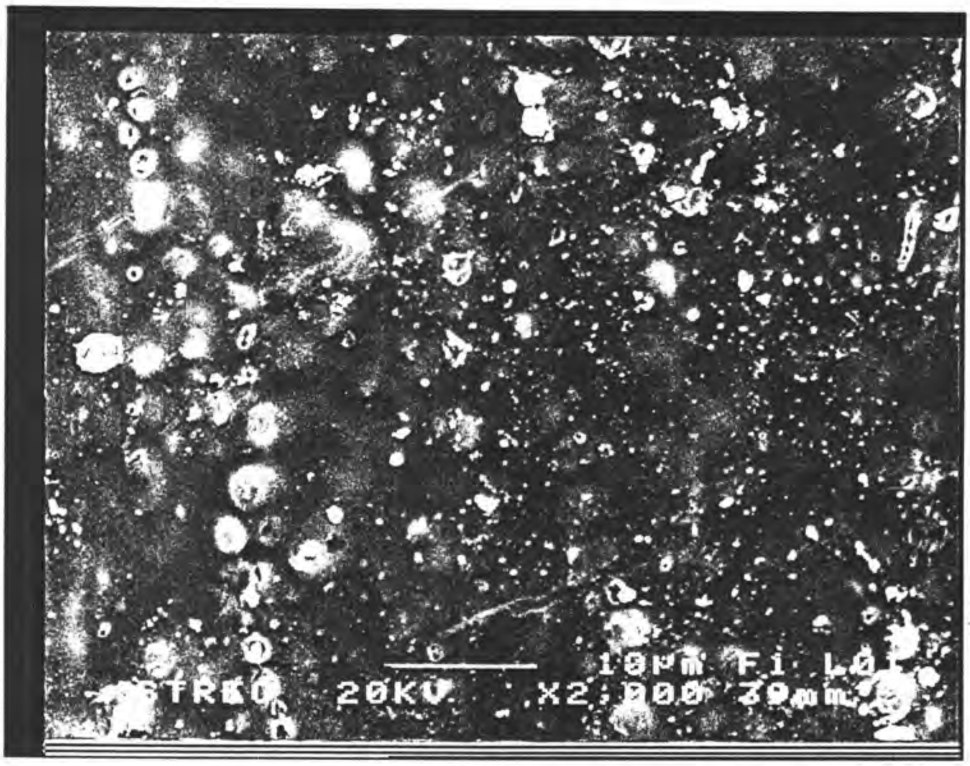


Figure 4-20 Scanning electron micrograph of HIPS with DE 79.

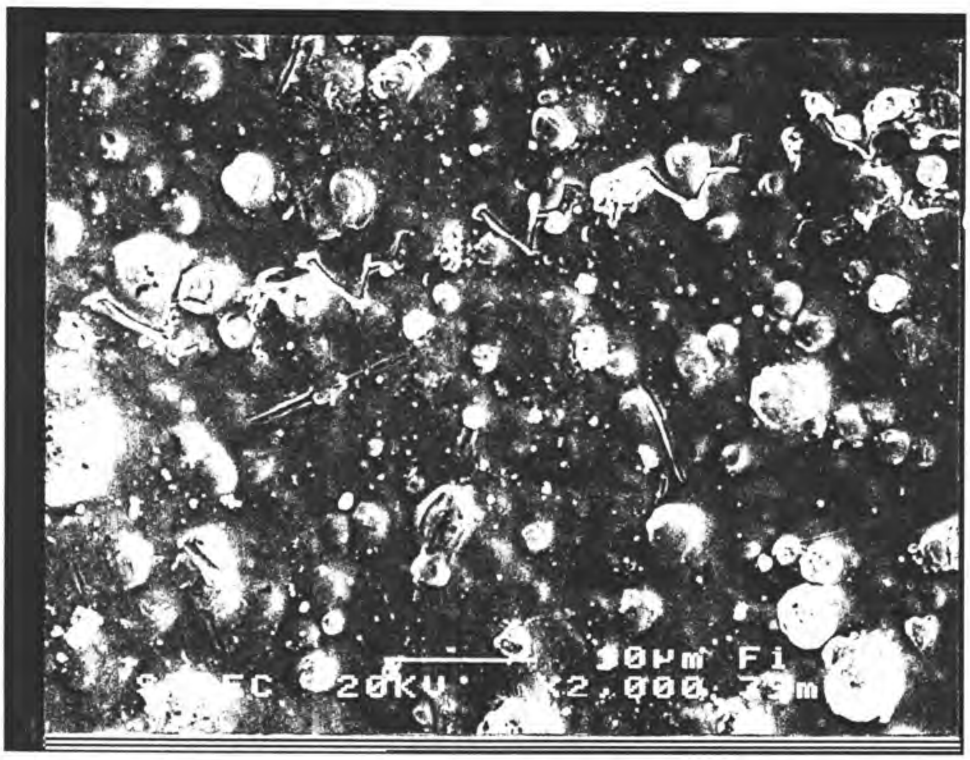


Figure 4-21 Scanning electron micrograph of HIPS with PBDS-80.

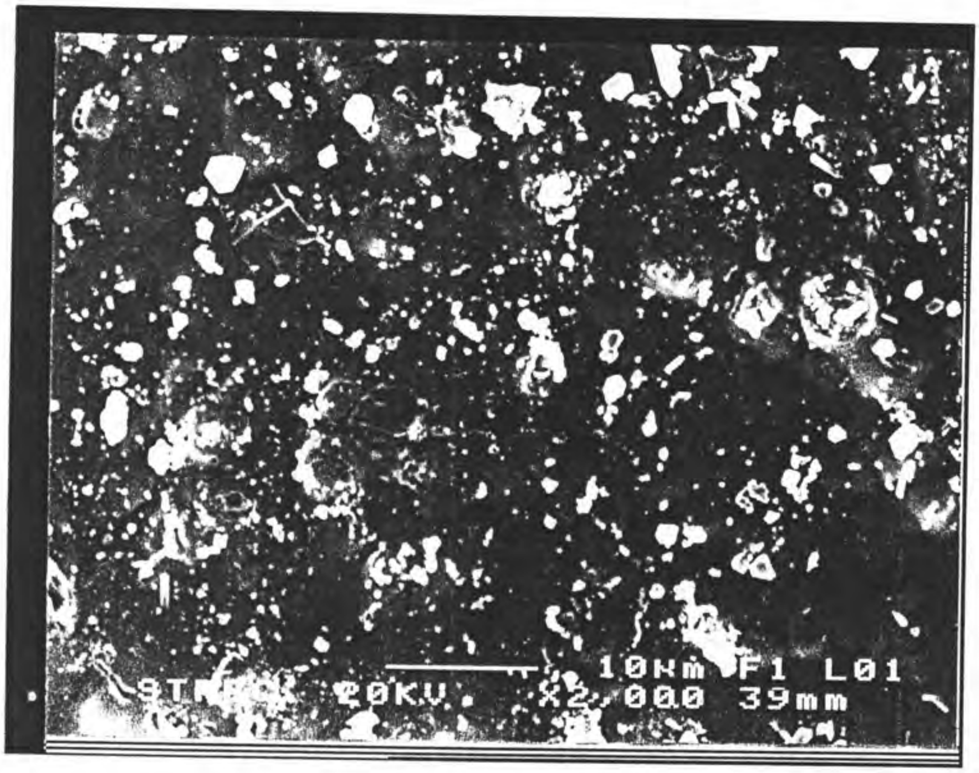


Figure 4-22 Scanning electron micrograph of HIPS with BA 59 and Sb_2O_3.

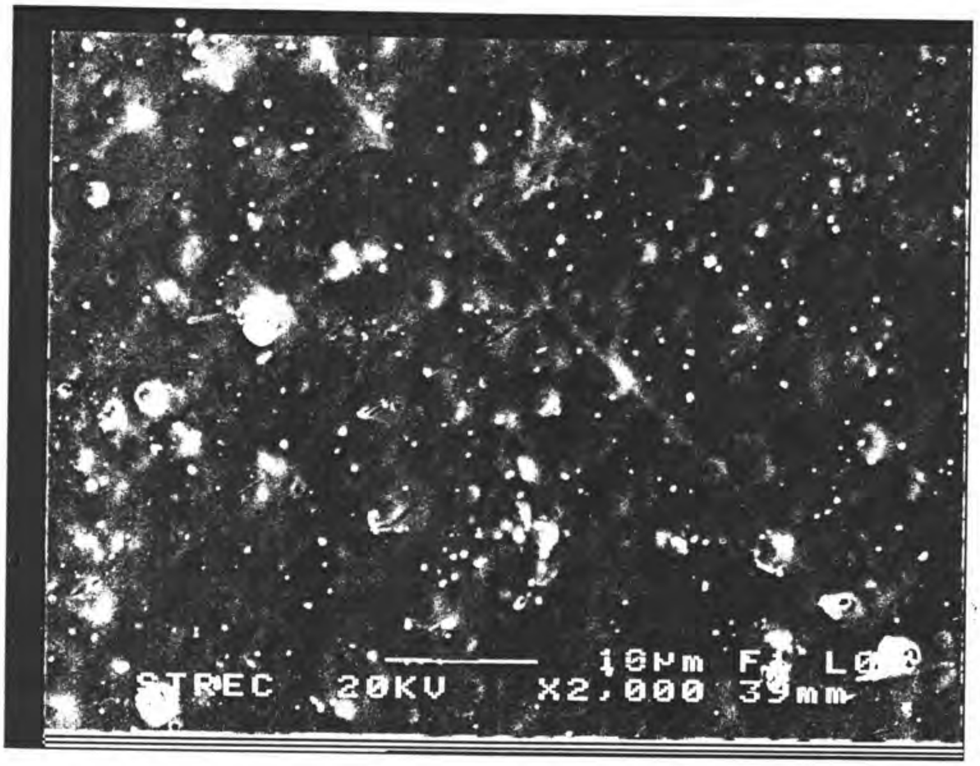


Figure 4-23 Scanning electron micrograph of HIPS with DE83-R and Sb_2O_3.

Figure 4-24 Scanning electron micrograph of HIPS with DE 79 and Sb_2O_3.

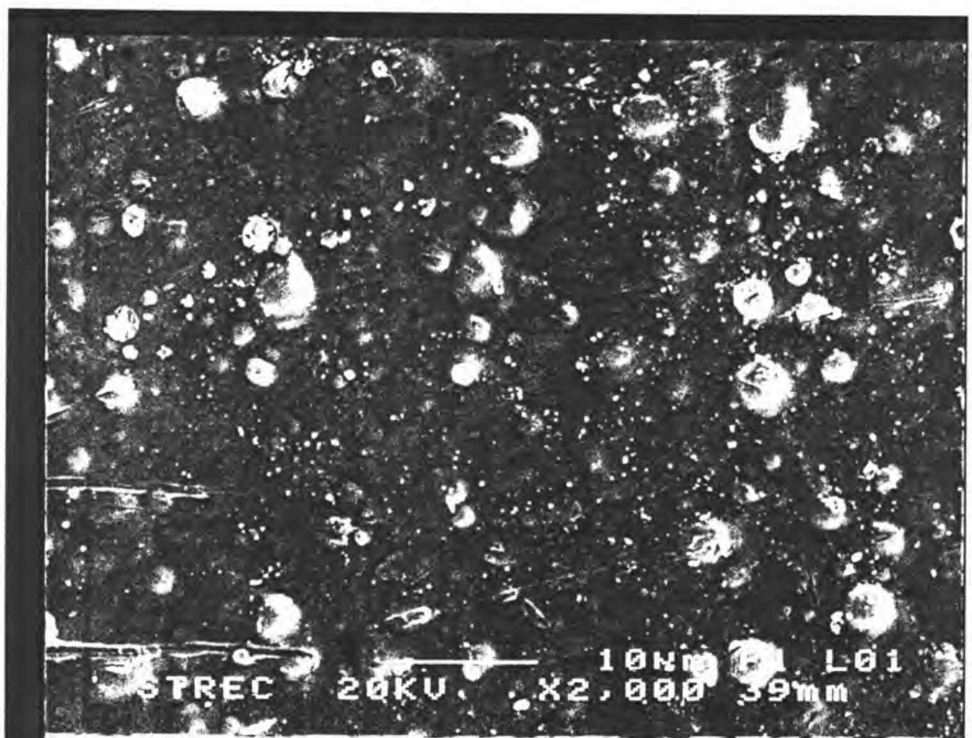


Figure 4-25 Scanning electron micrograph of HIPS with PBDS-80 and Sb_2O_3.

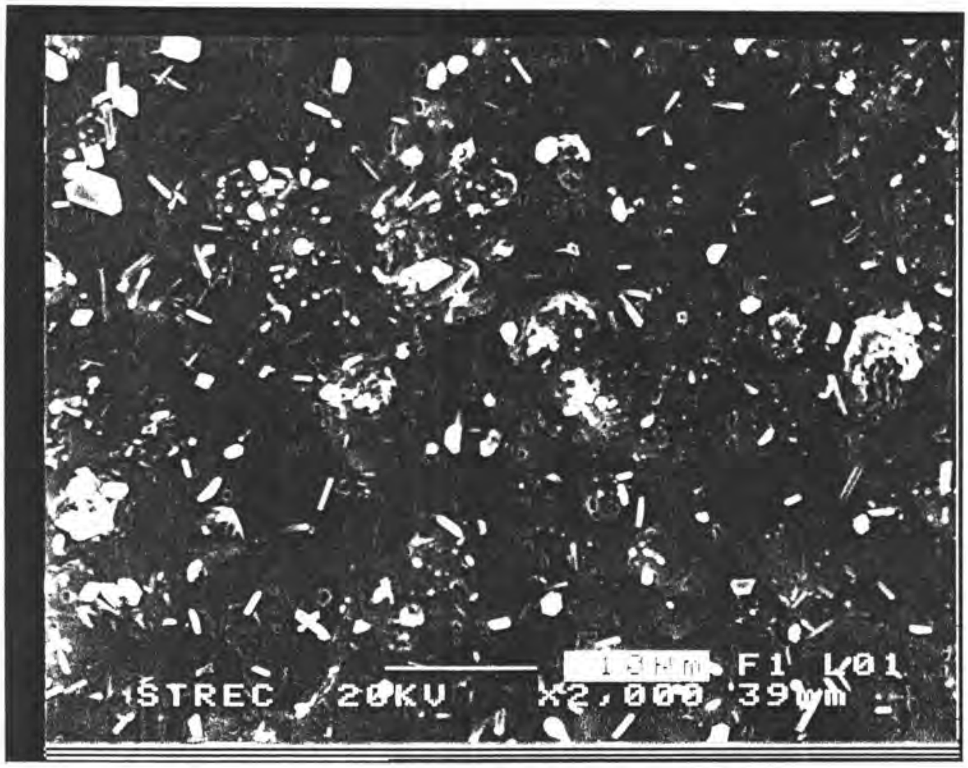


Figure 4-26 Scanning electron micrograph of HIPS with BA 59, Sb_2O_3 and CPE.

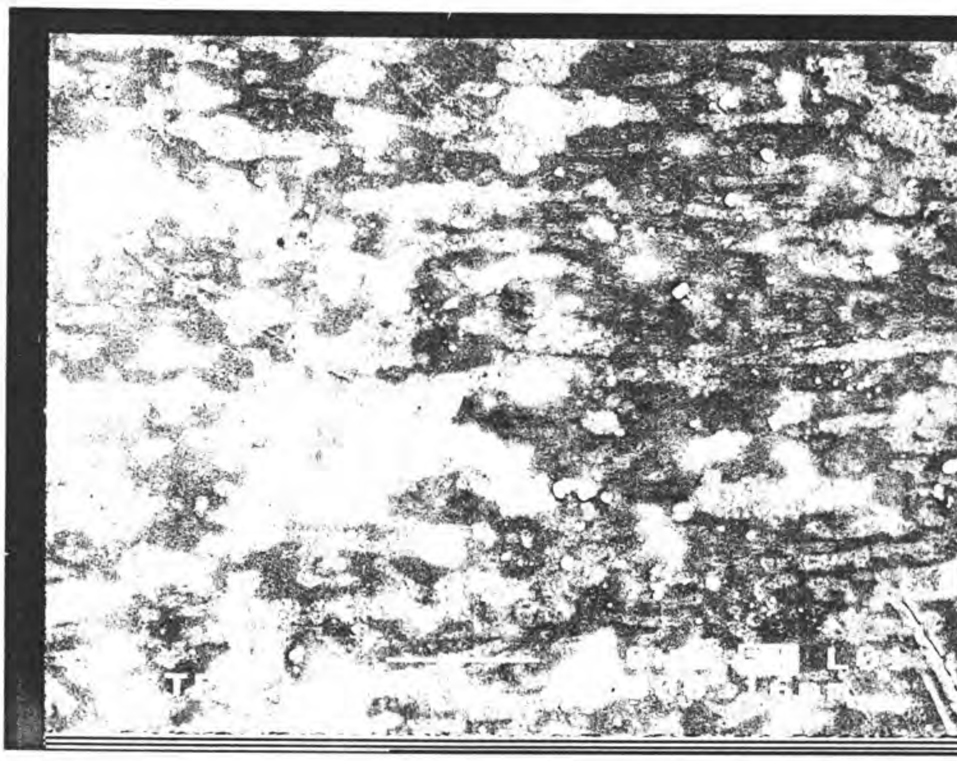


Figure 4-27 Scanning electron micrograph of HIPS with DE83-R, Sb_2O_3 and CPE.

Figure 4-28 Scanning electron micrograph of HIPS with DE 79, Sb_2O_3 and CPE.

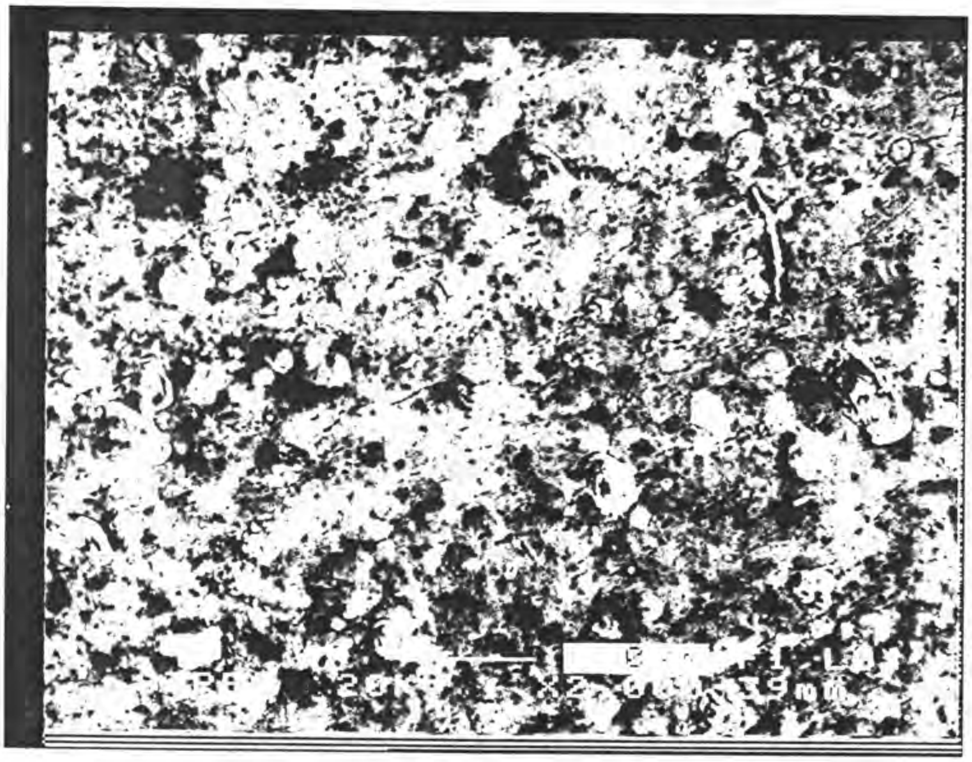


Figure 4-29 Scanning electron micrograph of HIPS with PBDS-80, Sb_2O_3 and CPE.