

CHAPTER 3

EXPERIMENTAL

3.1 Apparatus and Chemical Substances.

3.1.1 Apparatus.

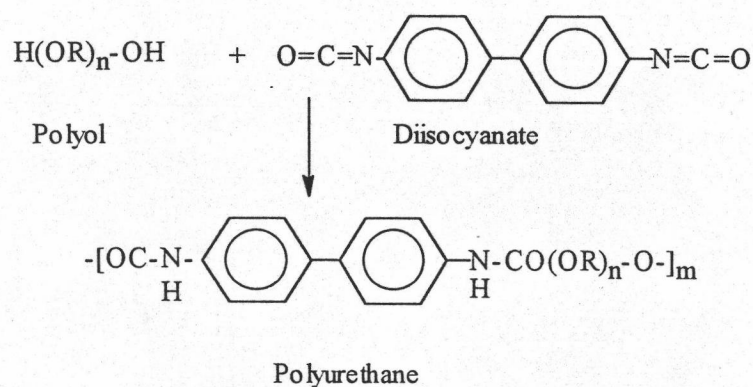
1. Flammability testing (ASTM D 2863-87), Stanton Redcroft FTA module, England.
2. Smoke density apparatus (ASTM E 662-79), NBS Smoke Chamber, England.
3. Thermogravimetric and differential thermal analyzer, Stanton Redcroft STA 780, England.

3.1.2 Chemical Substances.

1. Polyether linear polyol, ICI Chemical Ltd.
Average hydroxy number (K_{OH}) = 575.
2. 4,4-diphenylmethanediisocyanate(MDI), ICI Chemical Ltd.
Average isocyanate number (K_{NCO}) = 30.6.
3. Silicone oil, Dow Corning 200 fluid 100 Cs.
4. Trichlorofluoromethane, Aldrich Chemical Ltd.
5. Catalyst SFC, Dibutyl dilaurate ($C_{32}H_{64}O_4Sn$), Fluka Chemie AG.
6. Dimethoxymethyl phosphate (DMMP), ICI Chemical Ltd.
7. Chlorinated paraffin (C70), ICI Chemical Ltd.
8. Dechlorane plus (DECH), Occidental Chemical Co.
9. Dibromoneopentylglycol (DBNPG), Aldrich Chemical Ltd.
10. Decabromodiphenyl oxide (DBDPO), Aldrich Chemical Ltd.
11. Monobutyltin oxide (MBTO), Acima.

12. Monooctyltin oxide (MOTO), Acima.
13. Dibutyltin oxide (DBTO), Aldrich Chemical Ltd.
14. Dioctyltin oxide (DOTO), Alfa.
15. Dibutyltin maleate (DBTM), P & R Laboratory Supplies Co.
16. Dibutyltin diacetate (DBTA), Aldrich Chemical Ltd.
17. zinc hydroxystannate (ZHS), Alcan Chemical Ltd.
18. zinc stannate (ZS), Alcan Chemical Ltd.

3.2 Synthesis of polyurethane.



3.2.1 Additive free polyurethane.

The MDI (51.1 g.) was weighed and put into a plastic beaker. The polyol (34.5 g.), surfactant (0.3 g.), catalyst (1.0 g.) and blowing agent (13.2 g.) were also put into a plastic bottle, and shaken together. This mixture was then poured into the rapidly stirred isocyanate MDI. When the foam began to rise, the beaker was removed from the stirrer and the foam was allowed the fully rise and set. The height of the set foam was recorded.

3.2.2 Polyurethane containing tin and/or halogenated additive.

The polyurethane foams were prepared, using the ingredients as listed in table 3-1 to 3-2, in the following manner. The isocyanate MDI was weighed into a plastic beaker and, if required, the solid tin compound, and/or the halogenated compound were added. These were then incorporated into the MDI using a shear mixer. The polyol, surfactant, catalyst and blowing agent were weighed into a plastic bottle. If required, the liquid organotin or dimethoxy methyl phosphate were added. The plastic bottle containing the polyol and other compounds was shaken and then poured into the isocyanate MDI. After that, these ingredients were stirred with a electric paddle. When the foam began to rise, the beaker was removed from the stirrer and placed on the work bench. The height of the set foam was measured.

Remark : Only good quality foams, being comparable in height and appearance to the additive free foams were used for the fire property tests and smoke evolution.

Table 3-1 Amount of ingredients producing polyurethane foam treated one additive

	DMMP	Tin	RX
Polyol P230	32.9	31.7	30.5
Silicone oil	0.3	0.3	0.3
Catalyst SFC	1.0	1.0	1.0
Blowing agent	13.2	13.2	13.2
Isocyanate	48.6	46.8	45.0
Flame retardant			
-DMMP	4	-	-
-Tin compound	-	7	-
-Halogenated cpd.	-	-	10

Table 3-2 Amount of ingredients producing polyurethane foam containing halogenated and tin additives.

	DMMP/RX	1g Tin/RX	3g Tin/Rx	7g Tin/Rx
Polyol P230	28.9	30.1	29.3	27.7
Silicone oil	0.3	0.3	0.3	0.3
Catalyst SFC	1.0	1.0	1.0	1.0
Blowing agent	13.2	13.2	13.2	13.2
Isocyanate	42.6	44.4	43.2	40.8
Flame retardant DMMP+Halogenated compound	4 : 10	-	-	-
-Tin+Halogenated compound	-	1 : 10	3 : 10	7 : 10

3.3 Test method

3.3.1 Fire property test of polyurethane.

Fire retardancy was measured as Limiting Oxygen Index (LOI), which is a method for the determination of the relation flammability of polymeric materials. This test confirmed to ASTM D2863-70, a material in the form of either a rod or a sheet 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. In this study, LOI values were determined by using 10 x 10 x 120 mm. strips of the foam, 3 samples in a Stanton Redcroft FTA Apparatus. The Stanton Redcroft FTA model LOI Apparatus is shown in figure 3-1. The fire performance data obtained in this study are summarised in table 4-1, 4-2 and 4-3 respectively.

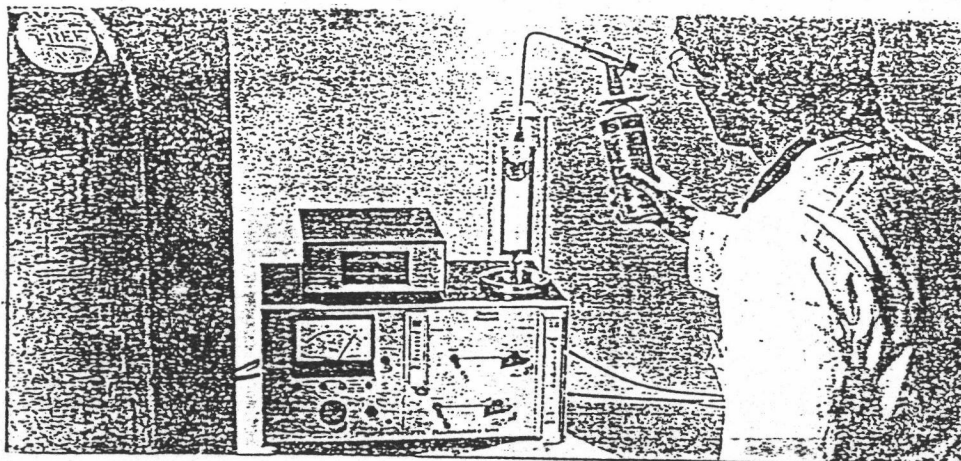


Figure 3-1 LOI apparatus

3.3.2 Smoke density measurement²⁴

It has been reported that emission can present a greater or more immediate danger than toxic gases since smoke itself is toxic and more important. It reduces visibility and could prevent a rapid escape from the hazardous situation. It has been estimated that approximately 80% of the victims of fire are not harmed by flames, but die as a result of smoke inhalation. Hence, the emission of smoke and toxic gases was one of the major hazards of fires. Smoke evolution test is mainly based on the use of a photoelectric cell to measure the optical density of the smoke. This test conforms to ASTM E662-79. The apparatus, NBS smoke chamber, is shown in figure 3-2. In this chamber, a horizontal light beam passed through the smoke evolution. Then smoke density determination was obtained by Stanton-Redcroft FIB unit. In the test, three samples, 70 x 70 x 25 mm, were examined and the mean value of optical density was recorded in figures 3-1 to 3-5. The values of maximum corrected smoke density per gramme (D_{mc}/g) in each case were shown in table 3-1, 3-2, 3-4.

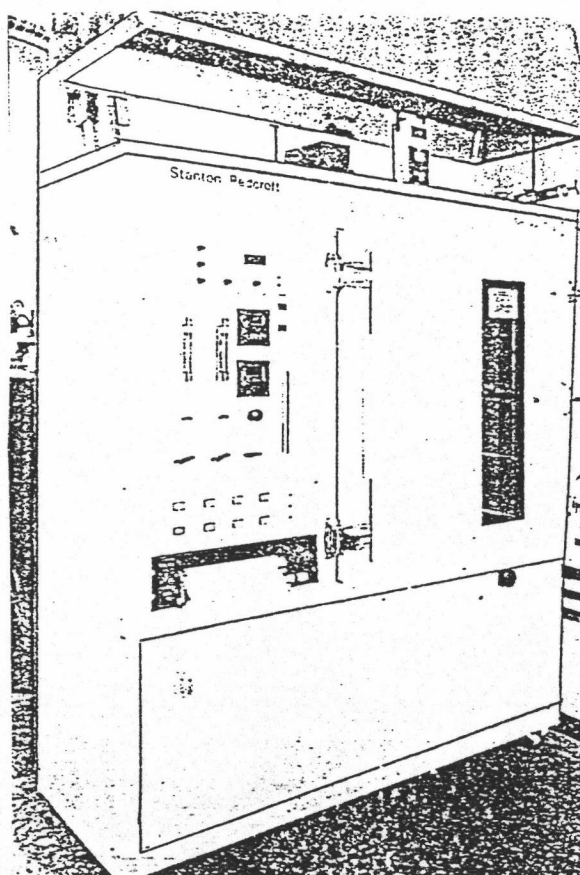


Figure 3-2 Smoke Chamber

3.4 Mechanistic studies

The burning characteristics of polymer can be generally examined by thermogravimetric analyser. This equipment indicates action mode of additive, solid phase or gas phase retardation.

For this study, simultaneous thermogravimetry (TG), derivative thermogravimetry (DTG), and differential thermal analyser (DTA) provides a useful insight into the thermal degradation and combustion processes which occur within the burning polymer.

Two main processes may be observed; initial degradation wherein the readily combustible material is burnt to yield a char; and char oxidation in which the char produced from the initial combustion is then burnt off. Furthermore, the weight of the residue remaining after combustion can indicate the presence or formation of non-combustible material.

The effect of additive on the burning characteristics of the polymeric material may be inferred by comparison between the additive-free, and the with-additive plots, changes in the plot profile of the initial degradation step would indicate that the fire retardant properties arise from a condensed phase reaction, i.e. polymer breakdown had been modified, this usually arises due to the formation of cross-links and results in a reduction in weight loss during the initial combustion, and hence an increase in char formation. If however, the initial degradation profile is unchanged, a vapour phase fire retardant mechanism is implied. A change in the char oxidation profile often indicates the formation of a more stable component within the char.

Comparison of the DTG_{max} values to assess the effect that additives have on the thermal stability of the polymer and/or the char produced. Fire retardants may have no effect on increase or decrease in the thermal stability of a polymer or its char.

In this study, The samples weighed about 10 mg were performed on a Stanton-Redcroft STA780 instrument with a computer enhancement facility (CETA). The heating rate was $10^{\circ}\text{C}/\text{min}$ from ambient temperature to 1000°C . The atmosphere was air, with a flow rate of $20\text{ cm}^3/\text{min}$. The results are summerized in table 4-5 to 4-6 and plotted in figure 4-10 to 4-22.