# 7. Experimental

## *7.1 Flame retardant treatment*

Unsized polyester fabric (150 g/m$^2$) was obtained from Wesco Fabrics. The fabrics were woven from fibre produced by South African Nylon Spinners (SANS). The flame retardant treatment process occurred in three stages.

### 7.1.1 Pre-treatment

Strips with dimensions of 50 mm wide and 200 mm long were cut. The pre-treatment consisted of weighing the strips and then placing them in an oven for a few days. A weight measurement after drying determined the amount of water bound to the fibres.

### 7.1.2 Impregnation

The fabric strips were marked for identification purposes. Solutions or suspensions of the specific flame retardant candidates were prepared a variety of solvents e.g. water, ethanol, methanol, acetone and chloroform. Solvents were chosen based on the greatest solubility for the compound under consideration. The fabric strips were submerged in the solution for a few seconds. After stirring they were removed and placed in a fume cabinet with extraction fan. The samples were then left to dry for about a day.

### 7.1.3 Post treatment

The air-dried samples were transferred to an oven and dried at 60 °C to 80 °C for a few hours. Thereafter they were weighed again to determine the mass percentage add-on of the flame retardant.

## *7.2 Evaluation of fire performance*

### 7.2.1 The bottom edge ignition test

The self-extinguishing times of the samples were tested according to the Underwriters Laboratory UL 94Vertical flame test - a common procedure specified commercially. For this test, five test specimens were evaluated and each specimen was exposed to two successive gas flame applications of 10 seconds each from the bottom edge. The chemicals used to estimate their flame retardant capabilities are listed in Table 11.

### 7.2.2 The Puddle flame retardancy test

Gouinlock, E.V *et al.* (1965) proposed a puddle test for evaluating the intrinsic flame resistance of dripping plastics. In this study, it comprised the placing a ca. one gram sample of the fabric on an inert surface. It was then ignited with a Bunsen burner held at a 45° angle. The flame length was ca. 25 mm flame and the ignition period 30 seconds. The burning behaviour as well as the mass loss of the sample was noted.

### 7.2.3 The Face ignition flame retardancy test

This test was set up as follows: A stretched fabric sample was attached to a vertical wire frame in order to keep it in place. A hole was then burned into the middle of the fabric by exposing it to a horizontal Bunsen flame. The flame was applied for ten seconds, to conform to the UL-94 flammability test. In each case the fabric was subjected to two flame applications. The fabric mass was measured before and after the flame treatments. The area of the burn-hole was also determined.

Table 11: Chemicals used to test for flame retardancy

| **Compound** | **Other Name** |
|---|---|
| 2-Furoic Acid | Furane-2-carboxylic acid |
| 4-Hydroxybenzyl alcohol | p-Hydroxybenzyl alcohol |
| Benzoyl peroxide | Dibenzoyl peroxide |
| Diethyl phthalate | Phthalic acid diethyl ester |
| Fumaric acid | 2-Butenedioic acid |
| Isophthalic acid | 1,3-Benzenedicarboxylic acid |
| Benzyl benzoate | Benzyl benzoate |
| Benzoic acid | Isopropenyl benzene |
| Benzoin | 2-Hydroxy-2-phenyl-acetophenone |
| Pentaerythritol | - |
| Phloroglucinol | 1,3,5-trihydroxybenzene |
| Pyrogallic acid | Pyrogallol |
| Benzophenone | - |
| Benzyl phenyl ketone | 2-Phenylacetophenone |
| Catechol | 1,2-Benzenediol |
| Resorcinol | 1,3-Benzenediol |
| Salicylic acid | o-Hydroxybenzoic acid |
| Terephthalic acid | 1,4-Benzenedicarboxylic acid |

### 7.2.4 Polymer dripping test

The experimental set-up to evaluate the flame retardancy of the treated polyester samples was relatively simple. Figure 2 shows the physical hardware involved for the sample-testing phase.

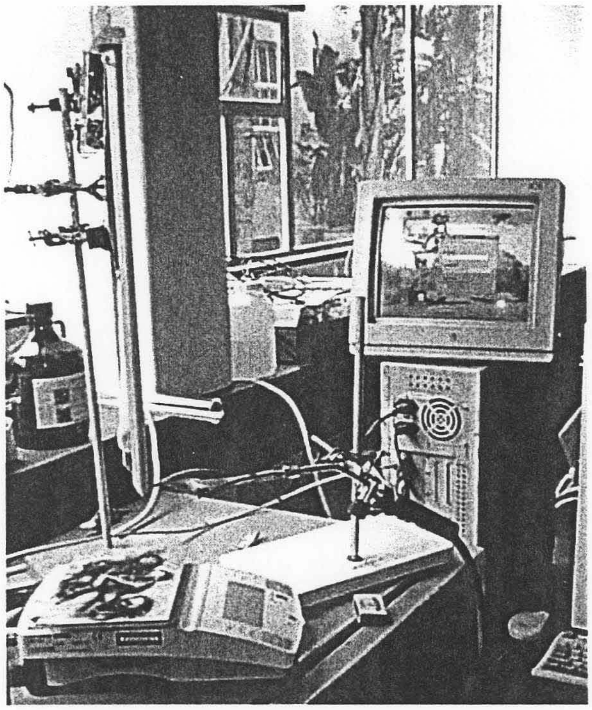

Figure 2: Experimental set-up for sample evaluation.

An OHAUS Explorer balance was used which was accurate to two decimal places and which stabilised within one second. The balance was capable of transmitting the date, time and mass to an RS232 port. It was captured on a personal computer using appropriate software.

The samples were evaluated by attaching a sample to a wire frame, which fitted into a rail to prevent the sample from rotating. The frame was attached a small 1.2V DC servomotor. A series of gears controlled the speed at which the sample was lowered into a fixed horizontal flame.

Figure 3 shows the flame and the test rig. The sample was lowered at a speed of approximately 1,8 ± 0,2 mm/s. A flat CADAC gas nozzle was used, which had almost the same width as the samples, to ensure good overall heating and melting.

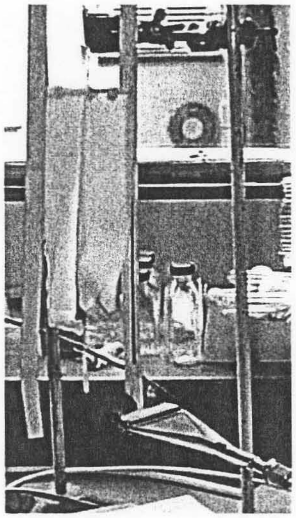

Figure 3: Sample being lowered into flame.

The molten polymer that dripped down was collected on an asbestos and stainless steel plate assembly on top of the balance load-plate. Figure 4 shows the LCD display of the balance during an actual run. The data was sampled once every second. Figure 5 shows the computer screen with the software while a sample was running.

Figure 4: Display of the balance during operation.

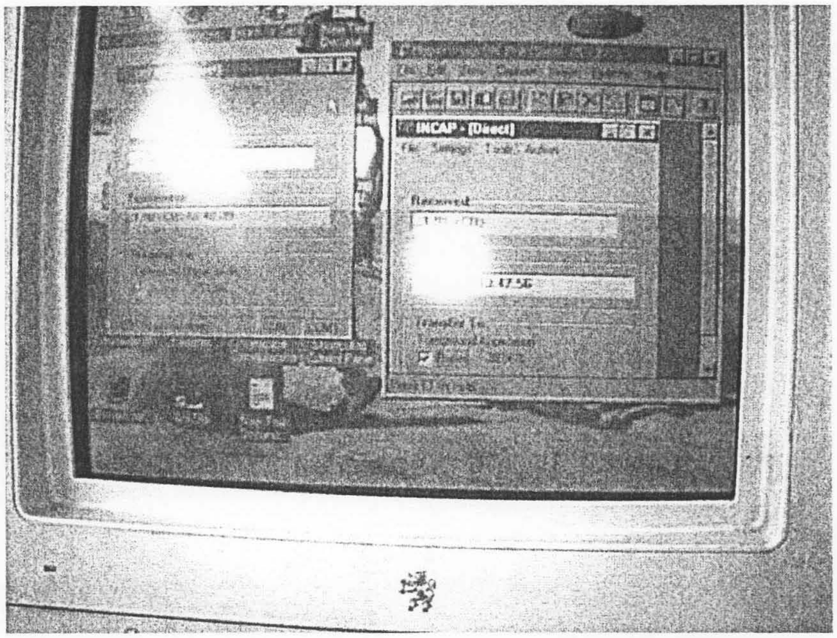

Figure 5: Computer screen showing the software in action.

Dripping was an erratic process. The rate of dripping was determined by fitting a straight line through the mass dripped versus time data. The average rate of dripping, for each treatment was obtained by averaging the data from 6 different samples.

### *7.3 Differential Scanning Calorimetry and Differential Thermal Analysis*

When a substance undergoes a physical or chemical change, a corresponding change in enthalpy is usually observed. This forms the basis of the technique known as differential thermal analysis (DTA) in which the change is detected by measuring the enthalpy difference between the material under study and an inert reference standard. The sample is placed in a heating block and the temperature increased at a uniform rate, usually 5 to 20 °C.min$^{-1}$. The sample temperature is monitored by means of a thermocouple and compared with the temperature of the inert reference. An empty sample pan is usually used as reference. If the change is exothermic, the sample temperature will exceed the reference temperature, for a short period, but if the change is endothermic, the sample temperature will lag behind the reference temperature. This temperature difference are observed as peaks, e.g. for the melting endotherm associated with crystallisation. Another type of change can also be detected. Since the heat capacities of sample and reference are different, $\Delta T$ is never actually zero, and a change in heat capacity, such as that associated with glass transition, will cause a shift in the base line position. Other changes such as decomposition, crosslinking, and the existence of polymorphic forms can also be detected. DTA are usually of a qualitative nature, so the results have limited usefulness.

To overcome these drawbacks a modified method known as Differential Scanning Calorimetry (DCS) was developed. In this technique, the sample temperature is controlled, by varying the heat input, to be the same as that of the reference. The thermograms obtained look similar to those from a DTA, but actually represents the amount of energy supplied to the system. The area under the peaks is therefore proportional to the change in enthalpy that has occurred, e.g. heats of crystallisation, melting and reaction. Normally an empty sample pan is used as reference. Calibration of the instrument will allow the heat capacity of a sample to be calculated in a quantitative manner.

A Perkin-Elmer DSC9 and a Netzsch STA 409 simultaneous TG/DTA instrument were used to collect calorimetric data. Sample masses of approximately 10 to 15 mg were weighed into either aluminium or platinum pans. With the Perkin-Elmer instrument the atmosphere was oxygen supplied at a flow rate of approximately 20 ml/min. With the Netzsch instrument the atmosphere was nitrogen supplied at a flow rate of approximately 20 ml/min. In both cases the scan rate was set at 10°C per minute.

### *7.4 Thermogravimetric Analysis*

In thermogravimetric analysis (TGA) the mass loss of a sample as a function of temperature is measured. The temperature is usually ramped at a constant scan rate. A Netzsch STA 409 simultaneous TG/DTA instrument was used to collect gravimetric data using the experimental conditions described above.

## *7.5 Rheometry*

A Rheometric International parallel plate rheometer was used to determine melt viscosities. Sufficient fabric was placed on the hot lower plate of the instrument, and the temperature then cooled down from ca. 300°C at a rate of 10°C/min. The atmosphere used was nitrogen.