

Note

THE CHARACTERIZATION OF SELECTED NYLON POLYMERS BY OXYLUMINESCENCE

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Ashby [1] was the first to note that various polymers, such as polypropylene, polyethylene, and others, emitted light when heated in an air or oxygen atmosphere. He noted that this light emission was dependent on the partial pressure of oxygen and its intensity was proportional to the concentration of oxygen in contact with the polymer sample. Since this was a new phenomenon for polymers in the solid state, and since the emission was proportional to the oxygen concentration, he named it oxyluminescence. Schard and Russell [2,3] also investigated this phenomenon and use the method to study the oxidative degradation of various polymers. They measured the oxygen consumption during oxyluminescence and also investigated the kinetics of the process. Wynne and Wendlandt [4,5] investigated the oxyluminescence of Alathon 1 (polyethylene) as well as other polymers in N_2 , O_2 and air. They found a first-order dependence on oxygen concentration for the oxyluminescence process.

Since the oxyluminescence (OL) curve of selected polymers appears to be unique, it is reasonable to assume that the OL curves might be used as a means of identification of the polymers under standard conditions of furnace heating rate and atmosphere. The results for the various nylon polymers reported here appear to confirm these assumptions.

EXPERIMENTAL

The light emission apparatus has been described previously [6]. The DTA function was not employed for these studies. Sample sizes ranged in mass from 10 to 20 mg (usually in pellet form), and a furnace heating rate of $12^\circ C \text{ min}^{-1}$ and an oxygen flow rate through the furnace of 40 ml min^{-1} were employed. A modification was made in the recording system in that a Bascom-Turner Model 8110-4 data center recorder [7] was used to record the data rather than a X - Y plotter.

The nylon polymer samples were obtained from Scientific Polymer Products, Inc., Webster, NY.

RESULTS AND DISCUSSION

The light emission of the oxyluminescence is very weak with intensities estimated at 10^{-10} to 10^{-8} lumens [1]. With nylon, the strongest emitter, the light emission could be seen by the dark-adapted human eye. The spectrum of this oxyluminescence has not been recorded, but it has been estimated that 50% of the emission has wavelengths between 420 and 515 nm and the other 50% at 300–420 nm [1]. Thus, it is in the blue-violet spectral region.

The OL curves for nylon 6 are given in Fig. 1. Three curves are presented. The first is the I (OL) vs. T (temperature) curve. The second is the I_c (corrected OL) vs. T curve; this curve was obtained by subtraction of the background radiation (the third curve in the figure) from the I - T curve, using the data center recorder mathematical routines. The corrected OL curve shows that the light emission consists of a single broad peak with shoulder peaks on the leading and trailing edges and with a peak maximum at about 260°C. The temperature dependence of the peak maximum with furnace heating rate was not evaluated but, as with other thermal analysis techniques, it is probably heating rate dependent.

The OL curves for the other nylon polymers are given in Figs. 2 and 3. All of the curves are corrected for background as previously discussed.

The OL curves for nylon 6, 6/12, 6/T, 11 and 12 are quite similar in appearance in that they all consist of a broad prominent peak with peak maxima between 250 and 300°C. The shoulder peaks on the leading and trailing edges of the main peaks are different. The OL curves for nylon 6/6, 6/9 and 6/10 are unique in that they consist of two or more curve maxima. For example, the curve peaks in nylon 6/9 have maxima at 225 and 335°C,

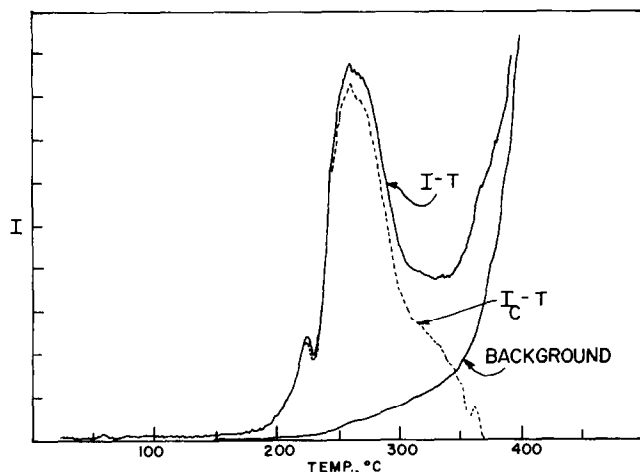


Fig. 1. Oxyluminescence curves of nylon 6. I - T is the experimentally determined curve and I_c - T the curve after subtracting the background emission.

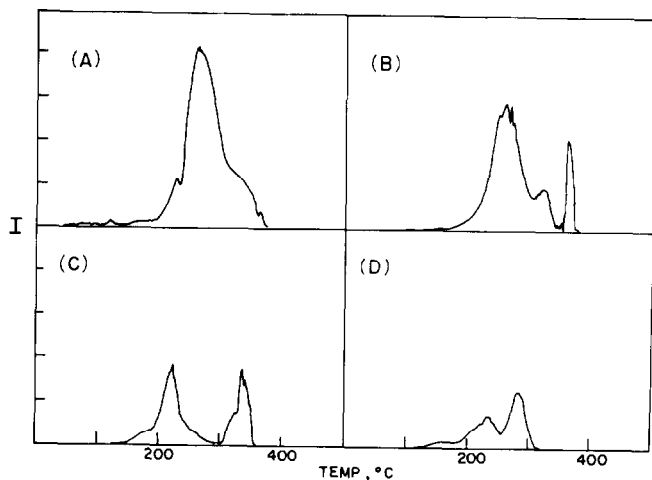


Fig. 2. Oxyluminescence of various nylon polymers. (A) Nylon 6 \times I/3; (B) nylon 6/6; (C) nylon 6/9; (D) nylon 6/10.

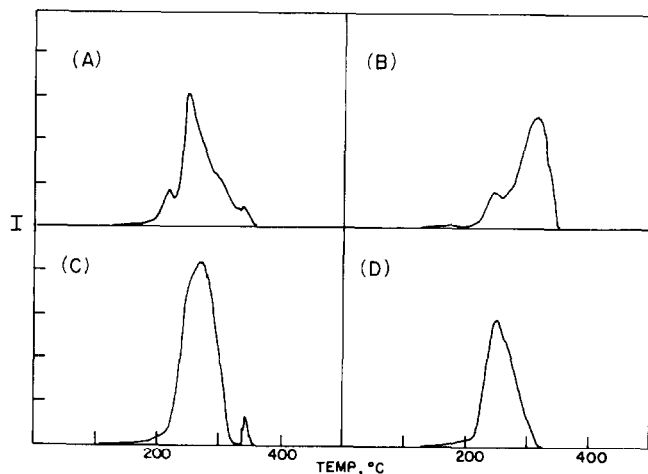


Fig. 3. Oxyluminescence of various nylon polymers. (A) Nylon 6/12; (B) nylon 6/T; (C) nylon 11; (D) nylon 12.

respectively, while nylon 6/6 has a peak with a maximum at 375°C, the highest temperature recorded in any curve peak.

In conclusion, the corrected OL curves recorded under these conditions may be useful for the characterization of the various nylon polymers, supplementing other thermal analysis data.

ACKNOWLEDGEMENT

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