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Note

THE CHARACTERIZATION OF CELLULOSE DERIVATIVES BY OXYLUMINESCENCE

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Although Ashby [1] found that various polymers exhibited a weak light emission, which he termed oxyluminescence (OL), when heated in air or oxygen, we have found that this is a general phenomenon for many organic compounds. Organic compounds such as cellulose derivatives (which are also polymers) exhibit a weak light emission when heated in an oxygen atmosphere. This is a true oxyluminescence reaction and not ignition such as was observed with coordination compounds containing vigorous oxidizing and reducing ligands [2,3].

Since the OL vs. temperature curve is unique for each cellulose derivative, it is proposed that they be used as a characterization technique to supplement the data of other thermal analysis methods.

EXPERIMENTAL

The oxyluminescence apparatus has been previously described [3,4] except that the DTA function was not employed. The outputs from the photometer and temperature sensor were recorded on a Bascom-Turner Model 8110-4 data center recorder [5]. This recording system permitted disk storage of the raw data and mathematical manipulation of the corrected OL data, which is presented here. The sample sizes ranged in mass from 10 to 20 mg, and a furnace heating rate of 12°C min^{-1} and an oxygen flow rate of about 40 ml min^{-1} were employed.

The cellulose derivatives, in powder form, were commercially available samples obtained from Scientific Polymer Products, Inc., Webster, NY.

RESULTS AND DISCUSSION

As found in the oxyluminescence of nylon polymers [1,6], the light emission was at a very low level, estimated previously [1] at 10^{-10} to 10^{-8} lumens. Also, as previously reported, the spectral distribution of the OL was

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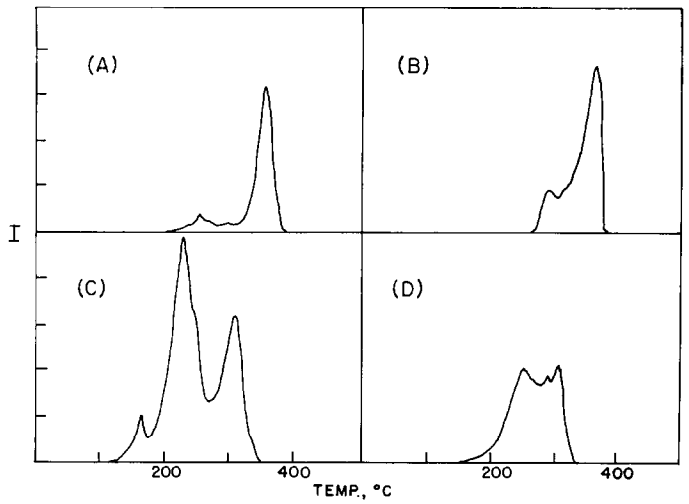

Fig. 1. Oxyluminescence of various cellulose derivatives. (A) Cellulose acetate; (B) cellulose propionate; (C) ethyl cellulose; (D) methyl cellulose.

not determined, so it is not known if the wavelength range is in the blue-violet range of the visible spectrum. Perhaps further studies will reveal this information. Since the OL is related to the thermal decomposition of the cellulose derivatives, perhaps this technique can be used to investigate the degradation kinetics and mechanisms, as was previously described for other polymers [7,8].

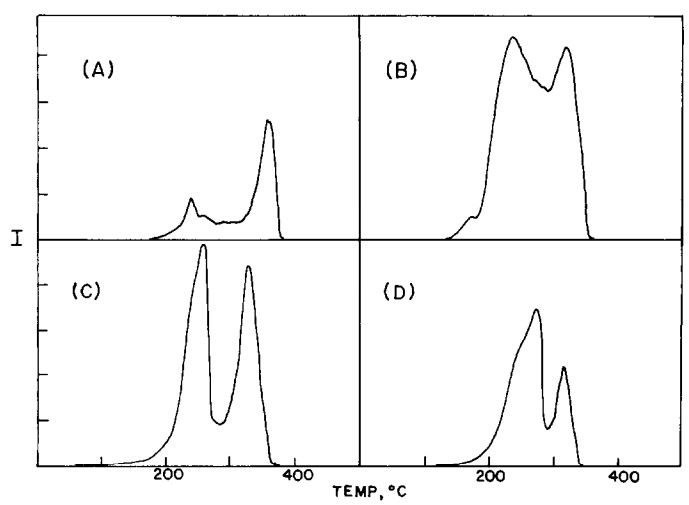

Fig. 2. Oxyluminescence of cellulose derivatives. (A) Cellulose acetate butyrate; (B) hydroxypropyl cellulose$\times 2I$; (C) hydroxypropyl cellulose, 10% hydroxypropyl, 30% methoxy; (D) hydroxybutylmethyl cellulose, 8% hydroxybutyl, 20% methoxy.

The OL curves of the cellulose derivatives, corrected for background as previously described [6], are given in Figs. 1–3. All of the OL curves are quite qualitative in nature but appear to be fairly reproducible under similar

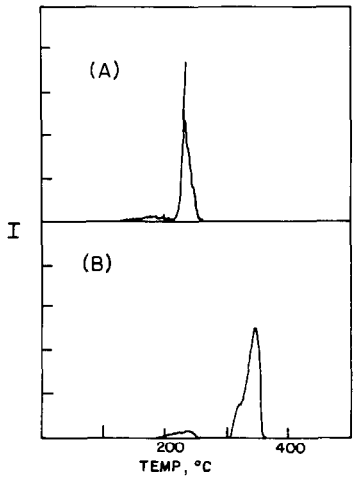

Fig. 3. Oxyluminescence of several cellulose derivatives. (A) Cellulose sulfate, sodium salt $\times I/5$; (B) cellulose triacetate.

conditions of heating rate, furnace atmosphere, and sample size. Changing any of these parameters, as with other thermal analysis techniques, would no doubt change the intensities (I) and maximum peak temperatures. OL curve peaks were found in the temperature range from 200 to 400°C, with most of the prominent peaks in the 200–300°C range. In most cases, two peak maxima were observed in the curve but in others, shoulder peaks added to the number of peak maxima.

The OL curves for ethyl cellulose, hydroxypropyl cellulose (10% OHPr) and hydroxybutyl cellulose all have similar features, as do methyl cellulose and hydroxypropyl cellulose. Also, cellulose acetate and cellulose acetate butyrate exhibit similar features.

Since all of the curves are unique for the cellulose derivative, they appear to be capable of a rapid identification of the compound, perhaps supplementing other thermal analysis techniques. The OL technique is simple in principle and practice and the characterization time is of the order of 20–30 min.

ACKNOWLEDGEMENT

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