Identification of Two Pass Regimes for Nylon-66 Based Rods in the Critical Oxygen Index Test

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Synopsis

Critical Oxygen Index (COI) values for rods of a nylon-66 based material of various diameters have been determined at 40°C. The existence of two discrete COI values has been identified for many sizes above a critical diameter: the two indexes correspond to clean burning and to burning with melting and dripping. The difference between the two indexes is dependent on rod diameter and can be greater than five units.

INTRODUCTION

The Critical Oxygen Index (COI) was introduced in 1966 by Fenimore and Martin[1] as a means of describing the flammability of small samples of polymeric materials, and was defined as "the minimal volume fraction of oxygen in a slowly rising gaseous atmosphere that will sustain the candle-like burning of a stick of polymer." The method has formed the basis of many national and international testing standards and has been applied to a wide range of substances including fabrics, foams, and even liquids.

Various sample and test parameters are known to affect the COI[2] and it has recently been shown that for thermoplastics the COI depends on the mode of burning in the test.[3] Materials that exhibit pronounced melting and dripping during combustion yield COI values that are dependent on the size of the test sample, whereas materials that burn smoothly display COI values that are independent of sample size. The surface area of sample involved in combustion can also be important in determining the dependence of the COI on sample dimensions. For instance, white Acetal in rod form yielded COI values independent of rod diameter, whereas the same material tested in sheet form, when a significantly greater surface area was involved in the combustion process, displayed COI values dependent on sheet thickness.[3]

Nylatron GS, which is a nylon-66 with molybdenum disulfide and other fillers added in controlled quantities, burned with severe melting when tested in sheet form to yield COI values dependent on sheet thickness.[3] The present study was undertaken in order to determine whether, as a result of the extent of melting that accompanied combustion, the COI values for rods of this material would exhibit a dependence on sample diameter.

EXPERIMENTAL

Rods of Nylatron GS (Polypenco Ltd.) of various diameters and in lengths of 170–180 mm were left in an oven at 40°C for at least seven days before being tested to ensure constant water content.

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COI values were determined using a Stanton Redcroft Hot Flammability Test Apparatus (HFTA)[4] modified with a 40 mm diam restriction annulus at the column outlet to ensure that a representative atmosphere was present during testing.[5] The rod samples, mounted vertically on the central axis of the column, were ignited at their upper surfaces by a 30 s application of a butane flame. Timing started when the butane flame was removed and the sample was deemed to burn successfully at a particular oxygen concentration if burning lasted for longer than 180 s.[6] A statistical testing method[7] was used to calculate the COI values.

All experiments were done with an environmental temperature of 40°C and a flow rate for the mixture of oxygen and nitrogen equivalent[8,9] to a column gas velocity of 40 mm/s.

RESULTS AND DISCUSSION

Rods of Nylatron GS exhibited two individual modes of burning behavior.

Ideal candle-like burning occurred at around 21% oxygen over the range of rod diameters investigated. This was characterized by a stable yellow flame burning above a small pool of melt located entirely on the top of the rod. There was no melting or dripping down the sides of the test pieces. The individual COI values for this mode of burning were highly reproducible and found to be independent of rod diameter over the range 3.2–15.1 mm investigated (Table I). These results compare favorably with those obtained for Perspex rod[3] which burned in a similar ideal fashion to yield COI values that were independent of rod diameter over the range 2–15 mm.

Tests conducted in atmospheres containing 23–28% oxygen produced a different mode of burning. On removal of the igniting flame the strong yellow flame remaining on the top of the sample began to produce molten material at a rate faster than that at which it was consumed. The top of the sample became slightly mushroom-shaped for a short while, and then melt began to flow from the upper

TABLE I
COI Values for Nylatron GS Rod Equilibrated to 40°C Prior to Testing

Rod diam (mm)	COI for candle-like burning	Mean	COI for burning with melt production	Mean
3.2	20.7, 21.2, 21.4	21.1	a	—
3.9	a	—	24.1, 24.4, 24.6	24.4
4.8	a	—	24.6, 24.7, 24.8, 25.6	24.9
6.1	a	—	a	—
6.4	a	—	a	—
8.0	21.5, 21.6, 22.0	21.7	27.3, 27.4, 27.5	27.4
9.6	a	—	26.5, 26.9, 27.7	27.0
10.1	21.7	21.7	23.0, 23.6	23.3
11.2	22.2	22.2	25.5, 25.8, 25.9	25.7
12.1	21.6	21.6	24.9, 25.3, 25.4	25.2
12.8	21.7, 21.7, 21.7	21.7	26.5, 26.6, 27.6	26.9
14.4	20.7, 20.9	20.8	b	—
15.1	21.4, 21.6	21.5	25.9	25.9

[a] Not detected.
[b] Not investigated.

surface down the sides of the test sample. Flame was often carried from the surface by the melt rivulets and occasionally combustion at the upper surface ceased completely. The duration of this type of burning was found to depend on oxygen concentration and it was apparent that by altering this the burning could be made to last for less than or longer than 180 s. Since this is the duration of burning required for a pass in the COI test, it was evident that a second COI value existed for these test samples.

Values obtained for this second COI are listed in Table I. COI values for rods burning in the dripping mode were easily obtained using the statistical testing method,[7] and were quite reproducible for a given rod diameter, especially in view of the erratic nature of the combustion process.

The COI obtained for the dripping mode of combustion was found to vary with the diameter of the test piece. The maximum COI value was found with 8.0 mm diam rod, which tended to exhibit greater melting and flame loss from the upper surface than the other sizes investigated. With the smaller diameter rods it was possible to detect only one of the COI values, probably because the reduced surface area of the melt zone made a distinction difficult between the two forms of burning. With 6.1 and 6.4 mm diam rods no useful results could be obtained since high oxygen levels were required to ensure burning of the rods on igniter removal and the samples were engulfed in flame once dripping occurred. The transition from sample diameters with only one index to diameters for which two indexes could be determined occurs at around 6 mm diam for Nylatron GS. For sample diameters of 8 mm or more, two distinct COI values could be detected in nearly all the cases investigated, probably because the increased cross-sectional area of the test samples enabled two stable burning conditions to exist.

The above observations are highly significant when it is considered that the oxygen concentration at which the sample passes the COI test may be approached from above or below. Thus, in experiments with Nylatron GS rods 8 mm or more in diameter, if the region of critical burning behavior is approached by gradually reducing the oxygen concentration in each test, starting from 28%, the COI corresponding to burning with melt production will be obtained. Conversely, by starting with an initial oxygen concentration of about 20% and doing experiments at progressively increasing oxygen levels, ideal candle-like burning behavior will be encountered. This latter mode of burning will yield a COI value that can, depending on the diameter of the test piece, be more than five units less than that obtained when burning is accompanied by copious melt production.

The appearance of samples of 12.8 mm diam rods (nominal 1/2 in.) after 180 s of burning at the two COI values (21.7 and 26.5) is shown in Figure 1. The amount of sample consumed in 180 s of candle-like burning was negligible and the rod length at the end of the experiment was still 180 mm. More than 10 mm of the length of the rod burning in 26.5% oxygen was consumed during the burning period, indicating the severity of combustion at this COI value.

Fenimore and Martin[10] have previously reported that polymer samples that flow or drip during combustion can give unexpectedly high COI values since heat is carried away from the combustion zone by the molten polymer flow. When the test material was formed about a ceramic wick and burned like a candle, a reduced and more representative COI value was obtained. Similar effects of sample flow and dripping have been discussed by Murch[11] and Gilleo.[12] Both

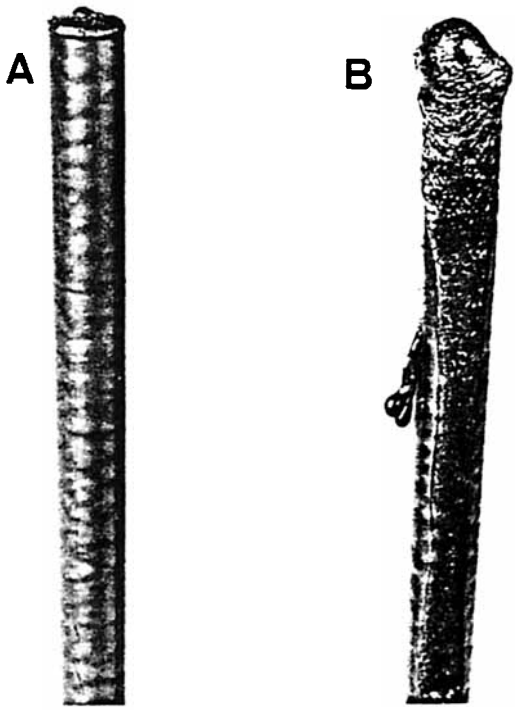

Fig. 1. The appearance of 12.8-mm-diam Nylatron GS rods after 180 s of burning in the COI test with an environmental temperature of 40°C. Sample A was burned at the lower COI value (21.7) and Sample B at the higher COI value (26.5).

authors have obtained reduced COI values by preventing the dripping that occurs on combustion of certain polymers.

Stuetz et al.[13] have recently discussed the oxygen demand for various top burning modes of polypropylene rods, and in each case reported that the oxygen concentration required for steady burning depended on the diameter of the test piece. For polypropylene, the mode of burning involving extensive dripping from the melt zone required a significantly higher oxygen concentration for combustion than that for burning on a glass thread wick.

The present study with Nylatron GS represents the first instance in which two reproducible COI values have been shown to exist for a material tested without the use of stabilizing wicks. Attempts to identify the existence of two COI values for pure nylon 66 rods were not successful. Nylon 66 rods are noticeably more flexible than the doped Nylatron GS form and all burning was accompanied by copious melting. Experiments with oxygen concentrations in the 17–30% range indicated that not until the oxygen level exceeded about 30% was the flame likely to last for longer than 180 s.

CONCLUSIONS

The existence of two discrete COI values has been identified for many rods of a doped nylon-66 above a critical diameter. This observation is important when it is considered that the oxygen concentration at which the sample passes the COI test can be approached from above or below: thus, depending on the

starting oxygen concentration, different COI values could be obtained for a given test sample. The difference between these COI values depends on the diameter of the rod under test and can be greater than five units.

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References

1. C. P. Fenimore and F. J. Martin, *Mod. Plast.*, **43**, 141 (1966).
2. R. K. Wharton, *Fire Mater.*, **3**, 39 (1979).
3. R. K. Wharton, *Fire Mater.*, **5**, 73 (1981).
4. A. C. F. Kamp, R. S. Lewis, and J. P. Redfern, in *Proceedings of the 1st European Symposium on Thermal Analysis*, Heyden, London, 1976, pp. 440–443.
5. R. K. Wharton, *Fire Mater.*, **5**, 6 (1981).
6. Standard Method for Measuring the Minimum Oxygen Concentration to Support Candle-like Combustion of Plastics (Oxygen Index), Test Method D2863-77, American Society for Testing and Materials, Philadelphia, 1977.
7. See, for example, Determination of Minimum Oxygen Concentration for Flame Propagation Following Top Surface Ignition of Vertically Oriented Specimens, Australian Standard 2122.2—1978, Standards Association of Australia, North Sydney, 1978.
8. R. K. Wharton, *Fire Mater.*, **4**, 59 (1980).
9. R. K. Wharton, *Fire Mater.*, **5**, 85 (1981).
10. C. P. Fenimore and F. J. Martin, *Combust. Flame*, **10**, 135 (1966).
11. R. M. Murch, Abstracts of the International Symposium on Fire Safety of Combustible Materials, University of Edinburgh, October 1975, p. 333.
12. K. B. Gilleo, Polymer Conference Series, University of Detroit, May 1973.
13. D. E. Stuetz, A. H. DiEdwardo, F. Zitomer, and B. P. Barnes, *J. Polym. Sci., Polym. Chem. Ed.*, **18**, 987 (1980).

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