Oxygen Index Evaluation Of Fire-Retardant-Treated Wood

Robert H. White

ABSTRACT. The applicability of using the oxygen index test (ASTM D 2863-76) to obtain an indication of the relative flammability of fire-retardant-treated wood products was investigated. The oxygen index is the minimum percentage oxygen that is required to maintain flaming combustion of a specimen under specified laboratory conditions. Within the plastics industry, the test is used in research and development and in quality control. Since the oxygen index test offers a single numerical value and requires relatively small specimens, it was desirable to determine its applicability to wood products.

Specimens from nine boards of southern pine were tested to obtain oxygen index values for untreated wood and wood at two treatment levels of diammonium phosphate. Next, untreated southern pine specimens were tested for the effects of grain direction, MC, and thickness of specimen. Finally, specimens from a single sheet of Douglas-fir plywood were treated with eight chemicals at four treatment levels and tested for oxygen index. The plywood oxygen index results were compared with available data for the fire tube, modified Schlyter, and 8-foot tunnel tests.

The results show that the oxygen index test can be used as an indication of the flammability of a fire-retardant-treated wood sample relative to other fire-retardant-treated and untreated wood products, the results for the untreated and treated samples showed a range of 22 to 78 and an average coefficient of variation of 3 percent.

THE OXYGEN INDEX TEST measures the minimum concentration of oxygen in a flowing mixture of oxygen and nitrogen that will just support flaming combustion. This test **(1)** has the advantage of producing a single numerical value which is an indication of relative flammability. The oxygen index, or limiting oxygen index, is the minimum percentage oxygen that is required to maintain flaming combustion of a specimen under specified laboratory conditions. Highly flammable materials are likely to have a low oxygen index. For plastics, the test has been shown to have good precision and reproducibility within and between laboratories. Within the plastics industry, the test is used in research and development and in quality control. Since the oxygen index test offers a single numerical value and requires relatively small specimens, it was desirable to determine whether the test would be a suitable and precise indicator of the relative flammability of wood products; this was the primary objective. Auxiliary objectives were to determine the distribution and variability of the oxygen indexes; the effect of density, general direction of anisotropy, moisture content (MC), and thickness of specimen on results; and the correlation of oxygen index with other flammability or flame spread tests.

Background

The oxygen index test developed by Fenimore and Martin (9-11) is a convenient procedure for obtaining a numerical indication of relative flammability of polymeric materials. The oxygen index has been determined for a wide range of materials (14, 15). Published values for wood products are listed in Table 1.

The oxygen index is generally recognized as an indication of general flammability or extinguishability. Results, however, are affected by modifications of the test procedures. In the standard candlelike determination, the uppermost surface of the specimen is ignited. A large reduction in the high oxygen index of some materials can result if the test is run by igniting the bottommost surface of the specimen (4). An example is a drop from 49.6 to

The author is Forest Products Technologist, USDA Forest Service, Forest Prod. Lab., Madison, Wis, The author thanks J. Haskell for help in the statistical design of the study, and R. Edlund for conducting the oxygen index tests. This paper was received for publication in December 1978.

TABLE 1. – *Oxygen indexes for wood products.*

Materials	Oxygen index	Reference
Cabinet birch	20.5	15
Red oak	22.7	15
White pine	20.9	23
Sugar maple	21.2	23
Pine, France	22.4	14
Poplar, France	22.5	14
Oak, France	24.6	14
Fiberboard, France	22.1	14
Particleboard, France	24.5	14
Plywood	19.7	23
Plywood, France	25.4	14
Plywood, FR, France	73.6	14
Cardboard, France	24.7	14
Paper towel, treated with 16% add-on diammonium phosphatesingle thickness	40.4	4
double thickness	49.6	4

29 for a double thickness of paper towel treated with 16 percent add-on diammonium phosphate (4). The oxygen index may also depend on the thickness or cross-sectional area of the specimen (9, 16). In tests involving arrangements of wood dowels, Rasbash and Langford (22) found that extinction was obtained when the oxygen concentration was reduced to between 13.2 and 19 percent depending on the direction of propagation and the arrangement of the dowels. Another modification of the standard test is to use an opposed flow diffusion flame instead of the conventional coaxial flow diffusion flame (21).

A major factor in the determination of the oxygen index is the temperature of the specimen and the gas mixture (6, 9, 13). Using oxygen indexes for polymeric materials, Johnson (17) concluded that the oxygen index will be 92, 78, and 55 percent of the oxygen index at 25°C when the gas mixture temperature is 100°, 200°, and 300°C, respectively. Heat losses due to specimen configuration or external heat input can affect the relative ranking of materials as well as the numerical values (5).

Comments on possible models of the oxygen index test are given by Fenimore and Martin (9, 20). By considering the energy balance in small laminar pool fires at extinction, Kanury (18) developed an equation for the dependency of the oxygen index on various physicochemical properties of the test system. Among the properties included in the analysis were combustion activation energy, ambient temperature, pool diameter, heat of combustion, feedback heat transfer coefficient and latent heat of pyrolysis.

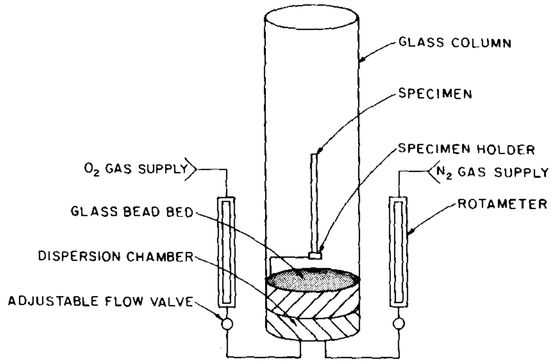

Figure 1. – Diagram of the oxygen index flammability test apparatus.

Experimental Procedure

Apparatus and Test Procedure

The oxygen index apparatus (Fig. 1) is designed to allow a candlelike burning of the specimen in a slowly rising mixture of oxygen and nitrogen. In the test, a specimen is placed in the holder at the center of the base of the test column. The flow valves are adjusted to obtain the desired initial oxygen concentration and total flow rate. The oxygen and nitrogen flow into the dispersion chamber and through the glass bead bed the gases thus are mixed and dispersed evenly over the cross section of the test column. The specimen is ignited so the entire top tip of the specimen is burning like a candle. A gas flame at the end of a tube with a small orifice is used to ignite the specimen.

For physically self-supporting plastic specimens (6.5- by 3.0- by 70- to 150-mm dimensions), the oxygen concentration is above the oxygen index if the specimen burns for at least 3 minutes after the igniter is removed, or if the specimen burns down 50 mm. The concentration is below the oxygen index if the specimen stops flaming before the criteria (3 min. or 50 mm) are satisfied. The procedure is repeated with a new specimen and a higher or lower oxygen concentration until the lowest concentration of oxygen that will satisfy the criteria is determined. The concentration in percentage is reported as the oxygen index. ASTM Standard D 2863-76 (1) specifies that the difference between oxygen concentrations that will and will not pass the criteria should be reduced to 0.2 percent or less. The entire procedure is repeated for three total gas flow rates between 3 and 5 cm per second. The average of the three results is usually reported.

For a material that exhibits erratic burning characteristics, it may not be possible to reduce the difference between oxygen concentrations, that will and will not satisfy the criteria, to 0.2 percent or less. For such materials, the critical concentration may be obtained by using a statistical method. One method is to test a fixed sequence of specimens (trials). If in a trial the oxygen index criteria are satisfied (success), the oxygen concentration is reduced by a fixed amount for the next trial (or increased if the criteria are not met (failure)). The result is an up-and-down (higher-and-lower concentrations) sequence of success and failure. Statistical techniques can be used to evaluate the sequence and obtain an estimate of the mean critical concentration for the material.

Variability Study

Nine sets of southern pine specimens were tested as an evaluation of the variability in the oxygen index testing of wood. Samples were cut from nine untreated 127- by 19-mm (5- by 3/4-in.) planks of southern pine sapwood. A randomized complete block design was used to divide the samples into one untreated and two treated groups. Those treated had either calculated 2.7 pounds per cubic foot, or calculated 7.9 pounds per cubic foot diammonium phosphate. After pressure treatment of the 457- by 127- by 19-mm (18- by 5- by 3/4-in.) samples, in which complete penetration was assumed, the samples were air-dried and conditioned at 80°F and 30 percent relative humidity (RH). Then the 3- by 6.5- by 150-mm test specimens were cut from the samples, so the grain direction was parallel to the length of the specimens .

For each plank and treatment, 3 oxygen indexes were determined by testing 3 groups of 20 specimens each in up-and-down sequences of trials. The difference between consecutive concentrations of oxygen in the sequence was kept constant at 1 percent.

Parameter Study

In the parameter study, the effects of grain direction, MC, and specimen thickness were investigated. Eight types of specimens (Table 2) were cut from each of three planks of untreated southern pine. For each plank and specimen type, three oxygen indexes (different total flow rates) were determined by testing sets of six to nine specimens in up-and-down sequences of trials with a concentration interval of 1.0 percent. For specimen types 1, 5, 6, and 7, extra specimens from the planks were ovendried to obtain the MC.

TABLE 2. – *Parameter study—types of specimens.*

Type	Variation from standard[1]
1	None
2	6.4 mm thick
3	9.5 mm thick
4	12.7 mm thick
5	80°F, 65% RH conditioning (11% MC)
6	80°F, 80% RH conditioning (13% MC)
7	80°F, 90% RH conditioning (17% MC)
8	Grain direction perpendicular to length

[1] The standard specimens were 3.2 mm thick, 6.4 mm wide, and 70 to 150 mm long. The conditioning was 80°F and 30 percent RH (6% MC), and the grain direction was parallel to the length of the specimen.

Treated Plywood

The oxygen index was determined for plywood specimens treated with eight chemicals at four treatment levels. The specimens were 3.2 mm wide and 70 to 150 mm long. They were cut from a single sheet of 1/4-inch-thick, three-ply, Douglas-fir plywood of A-C exterior grade. The grain of the plywood faces was parallel to the length of the specimen. The specimens were cut before treatment.

The eight chemicals used for treatment were sodium tetraborate decahydrate, disodium octaborate tetrahydrate, boric acid, monoammonium phosphate, ammonium sulfate, zinc chloride, ammonium polyphosphate 11-37-0 (liquid fertilizer), and sodium dichromate. The specimens were pressure-treated with solutions of the chemicals to obtain treatment levels of approximately 2, 3, 4, and 6 pounds of anhydrous chemical per cubic foot. The treatment levels were computed from the solution concentrations and the weight of the specimens immediately before and after treatment. Specimens were conditioned at 80°F and 30 percent RH before testing.

The oxygen index was determined for each chemical and treatment level by testing three sets (different total flow rates) of six to nine specimens in up-and-down sequences of trials with a concentration interval of 1.0 percent.

Results and Discussion

Variability Study

Because both successes and failures in satisfying the criteria were obtained at the same oxygen concentration, statistical techniques were used to evaluate the sequences of trials. The sequences of trials were first evaluated using the “large N” technique of Dixon and Massey (7), in which the formulas to estimate the sequence mean and sequence

TABLE 3. – *Variability study–"large N" oxygen indexes.*

Plank No.	Treatment level (lb./ft.^{3})	Sequence one		Sequence two		Sequence three	
		Mean	Std. dev.	Mean	Std. dev.	Mean	Std. dev.
				UNTREATED			
1	–	23.3	0.8	22.8	0.4	22.7	1.1
2	–	23.7	1.6	23.5	2.1	23.6	0.9
3	–	22.5	0.9	22.2	0.9	22.0	1.2
4	–	23.6	1.3	24.5	0.8	24.1	0.5
5	–	25.3	0.9	25.8	0.8	25.1	0.5
6	–	23.5	0.8	24.2	1.5	23.5	0.9
7	–	23.9	1.6	23.7	0.8	23.7	0.7
8	–	23.4	0.2	22.6	0.2	23.1	0.5
9	–	23.6	1.0	23.8	0.9	23.5	0.9
				LOW TREATMENT			
1	2.43	31.0	1.0	30.5	1.7	30.0	1.6
2	2.83	35.6	1.4	35.5	6.5	37.3	2.4
3	2.78	35.5	4.1	35.9	1.6	31.2	24.5
4	2.70	45.8	4.6	42.2	2.1	40.6	2.6
5	2.77	50.7	4.2	46.8	9.2	43.0	3.4
6	2.62	43.5	22.1	38.1	5.8	37.3	2.7
7	2.90	37.9	3.3	36.9	3.3	38.3	5.7
8	2.92	47.9	2.2	40.5	2.5	39.9	2.1
9	2.70	36.1	2.7	34.1	4.2	35.9	7.5
				HIGH TREATMENT			
1	6.88	63.9	0.9	63.0	2.4	60.2	2.7
2	8.14	78.7	2.0	76.9	0.8	77.0	0.5
3	8.18	67.2	2.2	67.9	1.4	65.7	7.2
4	8.10	76.4	3.5	76.9	15.6	77.9	7.0
5	8.06	72.3	24.9	75.7	1.4	76.3	6.1
6	7.61	65.5	6.5	65.3	2.1	65.5	5.6
7	8.63	74.6	3.5	76.8	13.9	80.6	4.0
8	8.34	75.7	1.2	78.1	2.3	79.6	1.4
9	7.53	60.0	1.9	60.3	1.8	60.6	2.7

standard deviation are based on the number of successes or failures at each test level (Table 3). Then the sequences were reevaluated using the "small N" technique of Dixon and Massey (7), which uses the order of the successes and failures of six to nine specimens to obtain an estimate of the sequence mean. These two procedures were compared to determine the sensitivity of the oxygen index to the test procedure.

Sequence variability. – One difficulty with the techniques is the need to select a constant interval of oxygen concentration between consecutive trials. The interval must be less than twice the standard deviation and should be greater than one-half the standard deviation of the individual specimens.

The standard deviation estimates ("large N") for the individual sequences (concentration intervals of 1.0) ranged from 0.2 to 2.1 for the untreated specimens, 1.0 to 24.5 for the low treatment level specimens, and 0.5 to 24.9 for the high treatment level specimens. The average sequence standard deviation was 0.9 for untreated, 5.0 for low treatment level, and 4.7 for high treatment level. These estimates suggest that the interval of 1.0 is satisfactory for untreated wood but an interval of 2.0 or 3.0 may be better for treated solid wood.

The estimates of sequence standard deviation based on 20 trials (Table 3) can be used to compute the standard error of the mean. The averages of the standard errors of the sequence mean from the equation of Dixon and Massey (7) were 0.3 for the untreated specimens and 1.3 for the treated specimens. The averages of the sequence coefficient of variation were 4 percent for the untreated, 13 percent for the low treatment level, and 6 percent for the high treatment level specimens.

Plank variability. – A different total flow rate was used for each of the three sequences (Table 3). An analysis of variance was performed to determine whether there was a significant difference between the three flow rates. The analysis indicated no differences due to total flow rates, so the three tests at different total flow rates were used to compute the standard errors of the mean for each plank. The standard error of the plank means were averaged and this resulted in standard errors of 0.2 for untreated, 1.3 for low treatment, and 0.8 for high treatment. The averages of the plank coefficients of variation of the three tests were 1 percent for the untreated, 6 percent for the low treatment, and 2 percent for the high treatment specimens.

An analysis of variance indicated no significant differences between the oxygen indexes computed using the "small N" technique of evaluating 6 to 9 trials and those computed using the "large N" technique of evaluating 20 trials. For the "small N" data, the averages of the standard error of the plank means were 0.3 for untreated, 1.1 for low treatment, and 1.1 for the high treatment specimens. The averages of the plank coefficients of variation of the three tests were 2 percent for the untreated, 4 percent for the low treatment, and 3 percent for the high treatment. If greater standard error of the mean is acceptable, the "small N" technique (7) can be used to evaluate two to five trials (instead of six to nine trials) which involve at least one reversal of response. For the parameter study and the treated plywood, the "small N" method was used to obtain the oxygen index.

Population variability. — The oxygen indexes normally reported as the oxygen index of the material is the average of the results obtained using three different total flow rates. The variation between the nine planks reflect the variability in a sampling of a population of treated and untreated southern pine. The average oxygen indexes ("large N" data) for the nine planks were 23.6 for untreated, 38.4 for the low treatment, and 71.1 for the high treatment of diammonium phosphate. The corresponding coefficients of variation were 3.8, 13.2, and 10.2 percent.

An analysis of variance indicated that there were significant differences between the oxygen indexes for the three treatment levels. The untreated density ranged from 28.2 pounds per cubic foot to 40.3 pounds per cubic foot, and the data did not indicate any correlation between density and oxygen index.

Variability factors. – Variability in the behavior of the wood specimens reduces the precision of the oxygen index test. Some of the variability was due to the inhomogeneity of specimens composed of springwood and summerwood. In some cases, the specimens would burn down the edges along the springwood instead of burning in a candlelike fashion. The springwood probably had a lower retention of the fire-retardant chemical due to its relatively thin cell walls. Also, gradients in the retention of the diammonium phosphate in the samples treated probably caused variations in the treatment level of the individual specimens. Since the determination of the oxygen index involves the testing of a sequence of individual specimens, the precision of the results depends upon the variability of the oxygen index of the individual specimens and the statistical technique used to evaluate the total set of results.

Parameter Study

While the number of tests was too small to quantify the relationships, the results of the parameter study do indicate that MC, thickness of specimen, and general direction of anisotropy can affect the oxygen index. The oxygen index of the untreated southern pine increased when the thickness of the specimen was increased from 3.2 mm to 6.4 mm, but did not increase appreciably with further increases in thickness (Fig. 2). This may be due to the minimum dimension remaining constant at 6.4 mm (width of the type 1 specimen) as the thickness was increased from 6.4 mm to 12.7 mm.

As expected, the oxygen index increased with increases in the **MC** of the untreated specimens (Fig. 3). The data points in Figures 2 and 3 are the averages for the three planks.

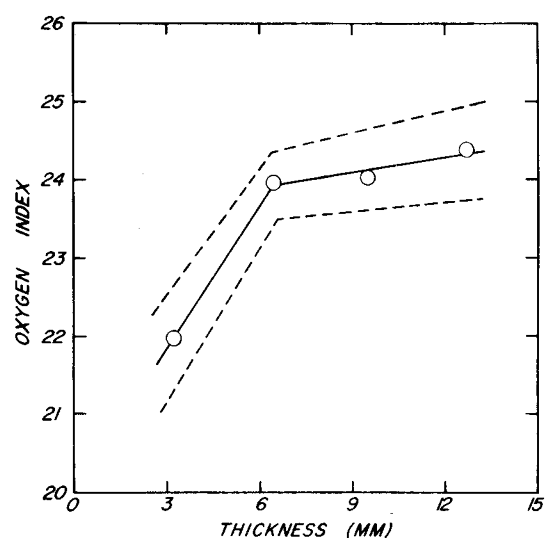

Figure 2. – Change in oxygen index with changing specimen thicknesses for 6.4-mm-wide southern pine specimens. (Solid line is average oxygen index; dashed lines are the average oxygen index plus or minus average plank standard deviation.)

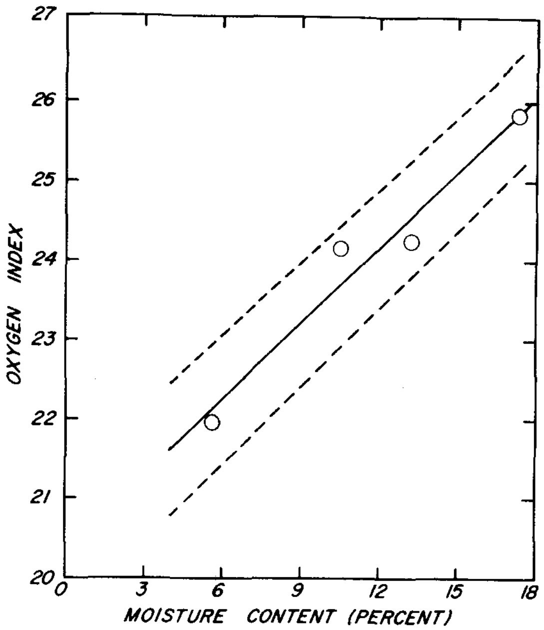

Figure 3. – Oxygen index increased linearly with increasing MC in southern pines specimens. (Solid line is average oxygen index; dashed lines are the average oxygen index plus or minus average plank standard deviation.)

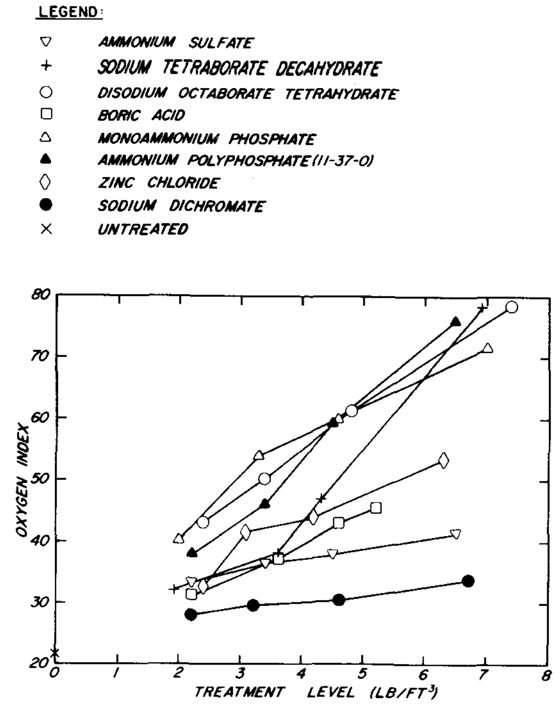

Figure 4. – Oxygen index of plywood treated with eight fire retardants and at various treatment levels. The oxygen index for the untreated plywood was 21.7.

The dashed lines in the figures represent the average oxygen index plus or minus one average standard deviation of the three oxygen indexes (different total flow rates) that were obtained for each plank and specimen type.

Comparison of results for specimen type 1 with specimen type 8 (Table 2) indicated that specimens with the grain of the wood parallel to the length (70- to 150-mm dimension) had an average oxygen index 2.0 higher than specimens with the grain perpendicular to the length of the specimen. We consider an increase of 2.0 significant, since the average of the standard deviations of the three oxygen indexes obtained for each plank and specimen type was 0.66 for the parallel grain and 0.32 for the perpendicular grain. Manley and Sidebotham (19), in tests on glass fiber-reinforced polyesters, also reported higher oxygen indexes for samples cut parallel to the fiber than for samples cut perpendicular.

Treated Plywood

The oxygen index results for the treated, 1/4-inch-thick plywood (Fig. 4) indicate that the oxygen index increased with an increase in the treatment level of the fire-retardant chemicals. The oxygen index results ranged from 21.7 for the untreated plywood to 78.6 for treated specimens.

The oxygen indexes were compared with published 8-foot tunnel furnace (ASTM E 286-69) (3), fire-tube (ASTM E 69-50) (2), and modified Schlyter panel (12) tests results for similarly treated 3/8-inch-thick Douglas-fir plywood (8). The treatment levels obtained in the treatment of the oxygen index specimens were not exactly identical to the treatment levels reported for the plywood used in the other tests. Linear interpolation was used to correct data on the flame spread index (8-ft. tunnel), average 3 minutes flame height (Schlyter panel), maximum 3 minutes weight loss (fire tube), and final weight loss (fire tube) for the differences. Comparison of the data (Figs. 5 to 8) indicates some correlation between the oxygen index and the other tests. The curves in the graphs were obtained by linear regression analysis of the data and have correlation coefficients of 0.81 (Schlyter panel flame height) to 0.89 (fire tube final weight loss). The curves seem to indicate that the oxygen index is different from the other tests in that it is

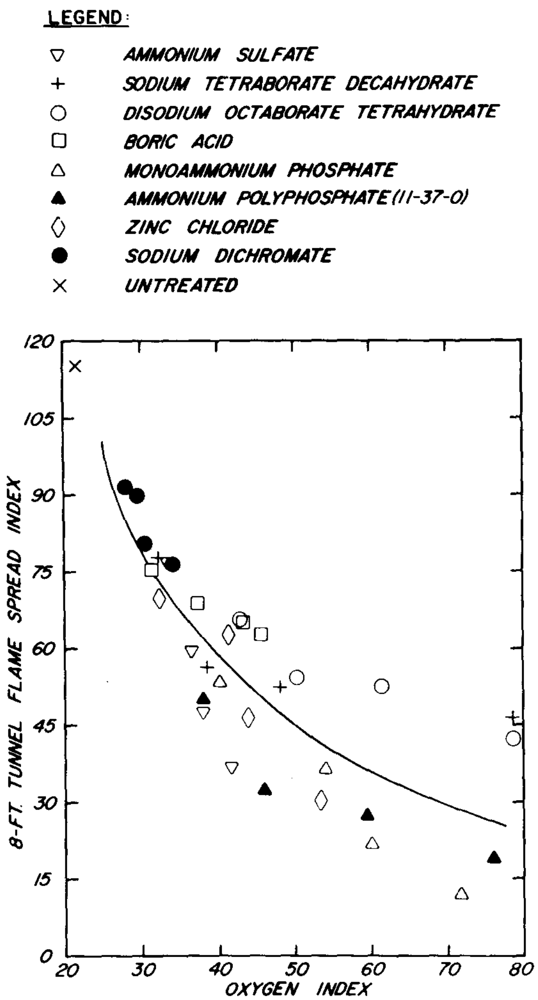

Figure 5. – Oxygen index versus 8-foot tunnel flame spread index. The equation of the curve is:

$$\text{flame spread} = -9.055 + \frac{2687}{\text{oxygen index}}.$$

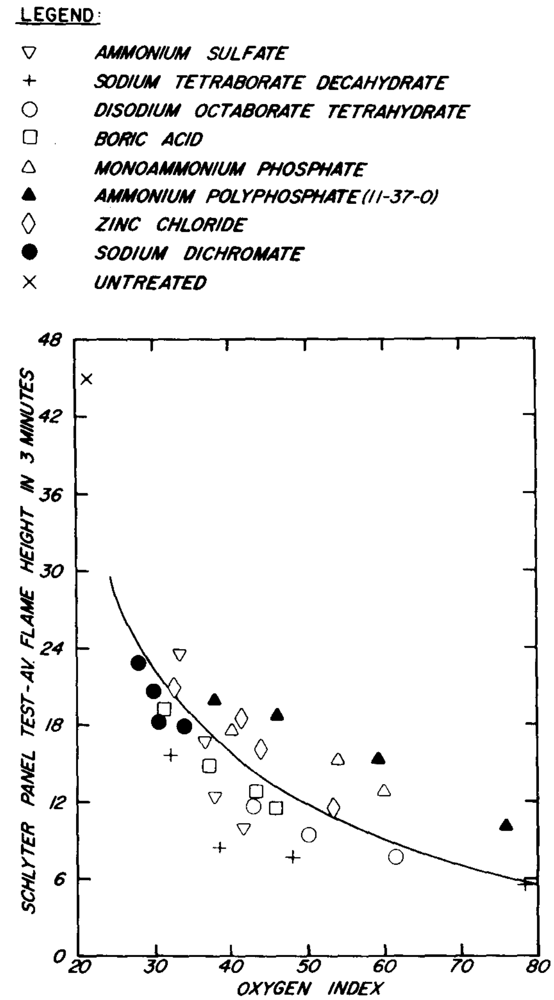

Figure 6. – Oxygen index versus modified Schlyter panel test average flame height in 3 minutes. The equation of the curve is:

$$\text{flame height} = 4.86 + \frac{824.6}{\text{oxygen index}}.$$

sensitive to changes in treatment level at high treatment levels.

The correlation is limited to the fire-retardant-treated plywood tested. Correlations between the oxygen index and other flammability tests have been obtained when limited to similar materials and generally do not apply when different types of materials are considered.

In the testing of the highly treated specimens (particularly sodium tetraborate and zinc chloride), only a small flame was visible. Failure was recorded if flaming ceased even though there was continued glowing or smoldering. In general, the behavior of the plywood specimens was less erratic than that of the solid wood specimens of the initial evaluation. This is indicated by an average coefficient of variation of 2 percent for the three tests of the plywood specimens at different total flow rates compared with 4 percent for the treated southern yellow pine specimens of the initial evaluation. The plywood specimens tended to burn in a more candlelike fashion and fewer burned down the edges as was a problem with the solid wood specimens. This is probably due to the lack of any distinct earlywood or latewood in the plywood specimens.

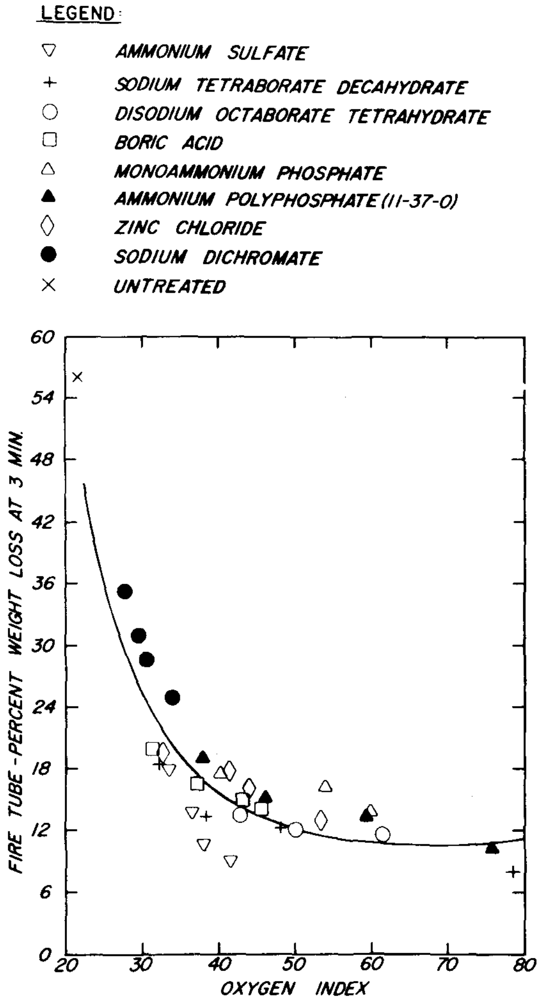

Figure 7. – Oxygen index versus fire tube maximum percent weight loss at 3 minutes. The equation of the curve is: ℓn(3-min. weight loss)=13.04–3.32ℓn(oxygen index) + 0.049 (oxygen index).

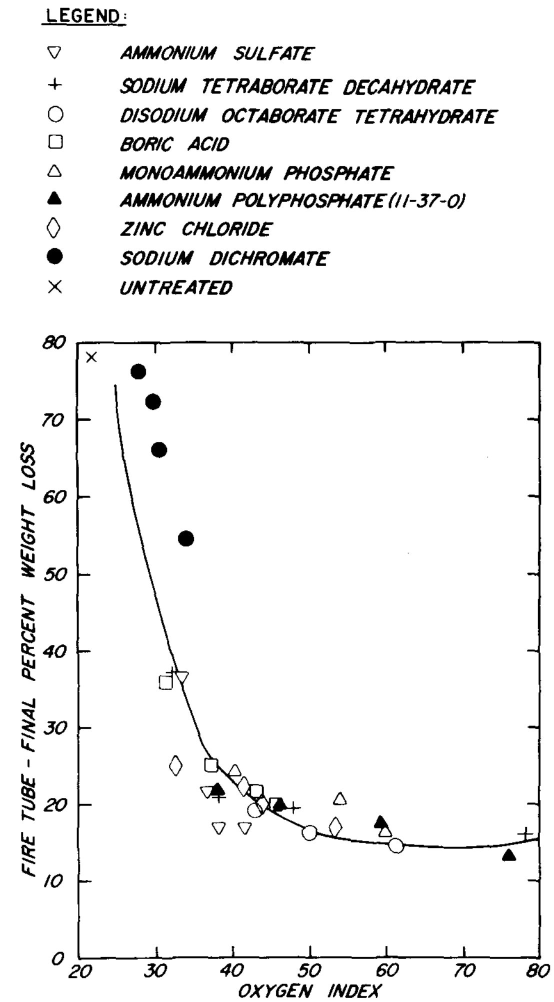

Figure 8. – Oxygen index versus fire tube final percent weight loss. The equation of the curve is: ℓn (final weight loss) = 17.25 - 4.57 ℓn (oxygen index) + 0.069 (oxygen index).

Limitations on Results

It should be noted that while high oxygen indexes may indicate a high treatment level and/or an effective fire-retardant chemical, it would be erroneous to infer from oxygen indexes the oxygen requirements, or the burning characteristics, of the material in real fire situations. Real fires involve substantially different exposure conditions and related factors than those in the oxygen index test. As noted previously, the test procedure and environment can affect the oxygen index obtained in the test.

Conclusions

The results show that the oxygen index test can be used as an indication of the flammability of a fire-retardant-treated wood sample relative to other fire-retardant-treated and untreated wood products. The relatively small size of the specimens required for the test makes the oxygen index test a useful test in the research and development or quality control of fire-retardant-treated wood products. A quality control procedure could involve testing a small number of specimens at one or two oxygen concentration levels related to the requirements of the product. The "small N" technique appears adequate to indicate the oxygen index of a treated wood product. The MC, grain direction, and dimensions of the specimens did affect the oxygen index. Because the test involves the flaming combustion of a material initially at room temperature and under mild exposure conditions, the data can only be used as a measure of behavior under the conditions of the test method and not under actual fire conditions.

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