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Oxygen Index of Fire-Retardant-Treated Plywood in Burning Test[1]

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酸素指數法에 의한 耐火處理合板의 燃燒試驗[1]

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ABSTRACT

To obtain relative effectiveness in fire resistance among fire retardant chemicals, oxygen indices were determined for 3.5 mm thick, three-ply, meranti plywoods, treated with 5 commercial chemicals and water and then press-dried, through Up and Down method following oxygen index test of ASTM D 2863-77.

The oxygen indices obtained were 28.4 for ammonium sulfate, 26.7 for monoammonium phosphate, 43.4 for diammonium phosphate, 30.1 for borax-boric acid, 32.4 for minalith, and 25.5 for water. Therefore, diammonium phosphate was found to rank first in fire-retardant effectiveness, followed by minalith, borax-boric acid, ammonium sulfate, and monoammonium phosphate in turn, judging from the fact that highly flammable materials are likely to have a low oxygen index.

Key words : fire retardant chemicals, plywood, oxygen index

要 約

耐火劑間의 相對的인 耐火效果를 比較, 檢討하여 보기 위하여 메란티(meranti)合板에 5種의 耐火劑와 水處理를 하고 熱板乾燥시킨 다음, ASTM D 2863-77의 酸素指數法에 의거하여 Up and Down法을 통하여 각각의 酸素指數를 구하였다.

試驗結果에 의하면 黃酸암모늄이 28.4, 第1燐酸암모늄이 26.7, 第2燐酸암모늄이 43.4, 硼砂-硼酸이 30.1, 미날리스가 32.4, 그리고 水處理가 25.5의 酸素指數를 나타냈는데, 燃燒性이 큰 物質일수록 낮은 酸素指數를 지닌다는 事實로 미루어 볼 때 耐火劑間의 耐火效果는 第2燐酸암모늄, 미날리스, 硼砂-硼酸, 黃酸암모늄, 第1燐酸암모늄의 順으로 크다는 것을 구명할 수 있었다.

INTRODUCTION

Plywoods have been favorably used for interior contents due to good workabilities, aesthetic values, etc. but their weakness against disastrous fire remains a critical problem to be solved. Where a greater level of fire resistance is needed, wood products such as plywood can be treated with fire retardants.

The effect of fire retardants on fire resistance can be easily estimated through a convenient procedure of oxygen index test. This test measures the minimum concentration of oxygen,

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i.e. oxygen index, in a flowing mixture of oxygen and nitrogen required to maintain flaming combustion of a specimen. The oxygen index is expressed as volume percent of oxygen in the flowing mixture and highly flammable materials are likely to have a low oxygen index.

Originally, this oxygen index test was developed for obtaining a numerical indication of relative flammability of polymeric materials by Fenimore and Martin(1966a, b). Later, the applicability of using this oxygen index test to obtain an indication of the relative flammability of fire-retardant-treated wood and wood products was proved by White(1979), Yoshimura and Umemura (1980), Yoshimura and Miwa(1980), and Yoshimura and Horii(1980).

White(1979) reported that oxygen indices were higher in Douglas-fir plywoods treated with 8 fire retardant chemicals than in untreated one, and increased with an increase in the treatment level of chemicals. Yoshimura and Horii(1980) indicated that sapwoods of *Cryptomeria japonica* and *Fagus crenata* treated with diammonium phosphate showed higher oxygen indices than with ammonium sulfamate.

On the other hand, Lee and Kim(1982) suggested that meranti plywoods treated with fire retardant chemicals showed greater fire resistance than untreated one and the most effective chemical was diammonium phosphate on the bases of weight loss rate, burning time, flame-exhausted time, and carbonized area. Lee(1984) noted that the rate of weight loss decreased as the chemical retention increased, especially in monoammonium phosphate treatment, in a study on solid wood of *Populus alba-grandulosa*.

This study was conducted to obtain and compare oxygen indices of meranti plywoods treated with commercial fire retardant chemicals including water.

MATERIALS AND METHODS

Plywood Specimens

Meranti(*Parashorea* spp.) plywoods, 3.5mm thick, three-ply, for upper interior grade were purchased and cut into 15cm x 15cm sections for fire retardant treatments. The sections with visible defects such as starved joint, knot, etc. were excluded and clear sections were conditioned in laboratory of 65% RH and 15℃ for 14 days.

Fire Retardant Treatments and Press-drying

Fire retardant chemicals used in this experiment were listed in the Table 1.

The clear plywood sections controlled to moisture content of 12.69-12.74% were treated with chemicals in specially devised soaking baths, 5 liter beakers to which thermometer attached, in a thermostatic chamber for 9 hours. Hot, at 60℃ for 6 hours, and subsequent cold, at 11℃ for 3 hours, soaking method was applied to water, ammonium sulfate, monoammonium phosphate, and diammonium phosphate. Only hot soaking

Table 1. Fire retardant chemical types, compositions, and chemical retentions.

Chemical type	Composition	Ratio(%)	Concentraion(%)	Chemical retention[Kg/(30cm)^3]
Ammonium sulfate	$(NH_4)_2SO_4$	100	20	1.263
Monoammonium phosphate	$NH_4H_2PO_4$	100	20	1.353
Diammonium phosphate	$(NH_4)_2HPO_4$	100	20	1.331
Borax-boric acid	$Na_2B_4O_7$	60	20	1.226
	H_3BO_3	40		
Minalith	$(NH_4)_2HPO_4$	10	20	0.906
	$(NH_4)_2SO_4$	60		
	$Na_2B_4O_7$	10		
	H_3BO_3	20		
Water(control)	Plain tap water	100	–	–

Table 2. Press-drying rates and specific gravities of treated plywoods

Chemical type	Drying rate (% M.C./min.)	Sp.Gr.
Ammonium sulfate	1.287	0.60
Monoammonium phosphate	1.712	0.63
Diammonium phosphate	0.980	0.53
Borax-boric acid	0.594	0.57
Minalith	0.743	0.62
Water(control)	2.040	0.53

treatment at 60℃ for 9 hours, however, was applied to borax boric acid and minalith due to their immiscibility and low solubility in cold solution. These soaking methods followed treating processes by Kim *et al.*, (1984).

After fire retardant treatments, the samples were redried between aluminium cauls in a hot press at a platen temperature of 90℃ and a pressure of 3.52kg/cm². Press-drying was progressed following Chen's cyclic step drying method (1978) and terminated at the moisture content of ca. 10%, the target point calculated backward from the measured weight during drying process. The press-drying rates of samples treated with chemicals and specific gravities after press-drying were tabulated in Table 2.

Oxygen Index Test

The oxygen index was determined, through large N technique of Up and Down method by Dixon and Massey(1969), for each fire retardant treatment with the help of an Oxygen Index Test Apparatus, Model FTA Serial No. 945, Stanton Redcroft Co., following the oxygen index test of ASTM D 2863-77(1977).

The specimens for oxygen index test were cut into 6.5mm wide and 70-120mm long with the grains of faces and backs parallel to the length from the plywood sections of 15cm x 15cm after chemical treatments and press-drying. The number of specimens for each treatment was 20 and tested at the gas flow rate of 3.05cm/sec. in the test column.

The oxygen concentration interval of 1.0 % was used in up-and-down sequences of trials. In an oxygen concentration, the specimen burned for at least 3 minutes after removal of igniter or burned down 50mm was marked with X sign and the specimen extinguished before the satisfaction with the criteria of 3 minutes or 50mm was marked with O sign. If a test at an oxygen concentration was marked with O or X sign, the next test was made at the higher or lower oxygen concentration by its interval of 1.0%, respectively.

The oxygen index($\bar{x}$) and standard deviation(s) were calculated, based on only O's or X's depending on which has the smaller total, by using below equations :

$$\bar{x} = \bar{y}_i \pm d/2, \quad \bar{y}_i = \Sigma y_i n_i / \Sigma n_i$$

$$s = 1.620(s_y^2/d^2 + 0.029), \quad s_y^2 = \frac{\Sigma y_i^2 n_i - (\Sigma y_i n_i)^2/n}{n-1}$$

where, y_i : oxygen concentration value

n_i : frequency of O's or X's at y_i level

d : interval of oxygen concentration

+ or − : used when the analysis is based on the O's or X's, respectively

$n = \Sigma n_i$

RESULTS AND DISCUSSION

Results of 20 tests for each chemical treatment in 3.5mm thick, three-ply, meranti plywoods by up and-down sequences of trials are depicted in Fig. 1 and the oxygen indices calculated from the test results using large N technique are tabulated in Table 3.

Table 3 indicates that oxygen indices range from 25.5 for water treatment (control) to 43.4 for diammonium phosphate treatment and the chemical treatments appear to be effective in fire resistance in different degrees with chemical types. The diammonium phosphate ranks first in fire-retardant effectiveness, followed by minality, borax-boric acid, ammonium sulfate, and monoammonium phosphate, judging from the fact that highly flammable materials are likely to have a low oxygen index.

Goldstein(1973) described that diammonium phosphate ranked first in fire-retardant effectiveness, followed closely by monoammonium phos-

Chemical type	Oxygen concentration (%)	Replication 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Frequency O's	X's
Ammonium sulfate	26	O				O		O														3	
	27		O		X		X		O		O											3	2
	28			X						X		O										1	2
	29												O				O		O		X	3	1
	30													O		X		X		X		1	3
	31														X								1
Monoammonium phosphate	25												O				O					2	
	26	O				O						X		O		X		O				4	2
	27		O		X		O		O		X				X				O		X	4	4
	28			X				X		X										X			4
Diammonium phosphate	40	O																				1	
	41		O		O																	2	
	42			X		O		O										O		O		4	1
	43						X		O								X		X		O	2	3
	44									O		O				X						2	1
	45										X		O		X							1	2
	46													X									1
Borax-boric acid	28							O		O		O										3	
	29				O		X		X		X		O									2	3
	30	O		X		X								O								2	2
	31		X												O		O				O	3	1
	32															X		O		X		1	2
	33																		X				1
Minalith	30	O																				1	
	31		O		O		O		O		O											5	
	32			X		X		X		X		O				O						2	4
	33												O		X		O		O		X	3	2
	34													X				X		X			3
Water(control)	24	O										O		O								3	
	25		O		O				O		X		X		O		O				O	6	2
	26			X		O		X		X						X		O		X		2	5
	27						X												X				2

Figure 1. Results of 20 tests for each chemical treatment by up-and-down sequences of trials. X and O represent the specimen burned for at least 3 minutes after removal of igniter or burned down 50 mm and the specimen extinguished before satisfaction with the criteria of 3 minutes or 50 mm, respectively.

Table 3. The oxygen indices and standard deviations for each chemical treatment.

Chemical type	Flow rate (cm/sec.)	No. of specimen	Frequency* O's	Frequency* X's	Average oxygen index	Standard deviation
Ammonium sulfate	3.05	20	11	9	28.4	3.5
Monoammonium phosphate	3.05	20	10	10	26.7	1.1
Diammonium phosphate	3.05	20	12	8	43.4	3.0
Borax-boric acid	3.05	20	11	9	30.1	3.7
Minalith	3.05	20	11	9	32.4	1.4
Water(control)	3.05	20	11	9	25.5	0.9

* Frequency from 20 tests in up-and-down sequences of trials.

phate, and these mono- and diammonium phosphates were highly effective in reducing both flaming and glowing but borax-boric acid only suppressed flaming. White(1979) showed that the oxygen index increased with the increase in the treatment level of fire retardant chemicals and

ranged from 21.7 for the untreated to 78.6 for treated, 1/4 inch thick Douglas-fir plywoods with sodium tetraborate decahydrate and disodium octaborate tetrahydrate at treatment level of ca. 7lb/ft^3. Yoshimura and Horii(1980) noted that oxygen indices of *Cryptomeria japonica* and *Fagus crenata* sapwoods treated with diammonium phosphate were higher than with ammonium sulfamate.

From the results of weight loss rate, burning time, flame-exhausted time, and carbonized area, Lee and Kim(1982) reported that fire resistance of meranti plywoods treated with chemicals were higher than the untreated and diammonium phosphate ranked the greatest in fire-retardant effectiveness, followed in turn by monoammonium phosphate and ammonium sulfate, and borax-boric acid and minalith. Borax-boric acid and minalith, however, appear to be more effective in fire resistance than monoammonium phosphate and ammonium sulfate in evaluation of oxygen index values in this experiment, differently from the result of Lee and Kim(1982). Recently, Lee and Chung(1989) noted that oxygen indices of untreated, 9mm thick plywood, medium density fiberboard, and particleboard were 26.9, 26.9, and 26.2, respectively.

CONCLUSION

The oxygen indices of 3.5mm thick, three-ply, meranti plywoods treated with fire retardant chemicals and water were 28.4 for ammonium sulfate, 26.7 for monoammonium phosphate, 43.4 for diammonium phosphate, 30.1 for borax-boric acid, 32.4 for minalith, and 25.5 for water treatment. Because highly flammable materials are likely to have a low oxygen index, the diammonium phosphate appears to rank first in fire-retardant effectiveness, followed in turn by minalith, borax-boric acid, ammonium sulfate, and monoammonium phosphate.

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