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Fire Performance of the Wood Treated with Inorganic Fire Retardants*1

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ABSTRACT

To prepare the eco-friendly fire retardant wood, Japanese red pine (*Pinus densiflora*), Hemlock (*Tsuga heterophylla*), and Radiata pine (*Pinus radiata*) were treated with inorganic chemicals, such as sodium silicate, boric acid, ammonium phosphate, and ammonium borate. Different combination and concentration of those chemicals were impregnated by vacuum/pressure treatment methods. The electron-beam treatment was used to increase the chemical penetration into the wood. The fire performance of the fire retardant treated wood was investigated. The penetration of chemicals into the wood was enhanced after electron beam treatment. Ignition time of the treated wood was the most effectively retarded by sodium silicate, ammonium phosphate, and ammonium borate. The most effective chemical combination was found at 50% sodium silicate and 3% ammonium borate, which satisfied flammability criteria for a fire retardant material in the KS F ISO 5660-1 standards.

Keywords : eco-friendly fire retardant wood, sodium silicate, ammonium borate, electron beam

1. INTRODUCTION

Nowadays the wooden house has been increased in Korea because of the interest in healthy life and recreation. But due to weakness of fire the wood has still evaded in the housing market. It is a time that more positive methods to spread of fire safety wood. There has been many research work to improvement of the wood's combustibility, mainly focus the flame retardant agent treatment to make retardant wood. The retardants such as phosphate, nitrogen, boric acid compound, mainly used for retardant treated wood. Retardant treated wood showed delay the ignition time and decreased the velocity of heat release and flame spread. The performance of the treated wood did not effected by species and the toxic emission was less than untreated wood (Kozlowski, 1955; Mikkola, 1991; Toshiro *et al*., 2003; Lee, 2008; Choi, 2011).

Recently, the concern of the retardant agent focused on low-harmfulness, low-smoking, low-implantation. And these reason, brominated flame retardants under going regulation (Hardy, 1999). The ways of lowering the flammability of wood and wood products by chemical treatments are by chemical impregnation of wood (Ondrej *et*

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Photo. 1. Pressure treatment of fire retardants.

Photo. 2. Cone calorimeter test of fire retardant treated wood.

Table 1. Weight increase rate (%) by electron beam treatment (dry condition)

Treatment	Electron beam Treatment (kGy)	Japanese red pine (%)	Hemlock (%)	Radiata pine (%)
Sodium silicate (50%) + Boric acid (3%)	Control	19.79	16.58	50.76
	100	58.8	52.32	65.15
	200	47.34	63.51	71.07
	300	16.56	55.3	65.15
Sodium silicate (50%) + Diammonium phosphate (3%)	Control	29.45	25.55	26.03
	100	37.62	61.66	73.63
	200	52.32	65.31	64.6
	300	48.77	55.47	65.46
Sodium silicate (50%) + Ammonium borate (3%)	Control	8.1	8.85	37.74
	100	38.53	50.52	72.75
	200	36.68	65.98	64.23
	300	64.08	67.81	68.85

al., 1999). We has been tried to develop eco-friendly retardant chemicals and their treating methods. We used to inorganic chemicals to make environmental friendly fire retardant wood (Son *et al*., 2012). In order to increase the penetration of chemicals into the wood, we introduced the electron beam irradiation. Fire retardant performance of the retardant treated wood was investigated.

2. MATERIALS and METHODS

Sodium silicate, boric acid, di-ammonium phosphate, ammonium borate were used for fire retardants. Japanese red pine, Hemlock, Radiata pine specimens (12 × 100 × 600 mm) were dried to 12% (MC) before retardants treatment. The electron beam treatment was accepted for pre-treatment to increase chemical penetration. The range 100～300 kGy electron beam were

Table 2. Ignition time of different fire retardant treated wood

Species	Main Chemicals	Sub Chemicals	Ignition time
Japanese red pine	Sodium silicate 30%	boric acid 3%	40
		di-ammonium phosphate 3%	29
		ammonium borate 3%	39
	Sodium silicate 50%	boric acid 3%	57
		di-ammonium phosphate 3%	50
		ammonium borate 3%	48
Hemlock	Sodium silicate 30%	boric acid 3%	57
		di-ammonium phosphate 3%	64
		ammonium borate 3%	48
	Sodium silicate 50%	boric acid 3%	139
		di-ammonium phosphate 3%	282
		ammonium borate 3%	207
Radiata pine	Sodium silicate 30%	boric acid 3%	45
		di-ammonium phosphate 3%	45
		ammonium borate 3%	120
	Sodium silicate 50%	boric acid 3%	249
		di-ammonium phosphate 3%	-
		ammonium borate 3%	-

adjusted to the wood specimens.

Chemical treatment schedule was two steps. First, the Sodium silicate (30, 50%) was treated for main retardants. Secondly, boric acid (3%), di-ammonium phosphate (3%), ammonium borate (3%) was treated by vacuum (78 kPa, 30 minute), pressure (18 kg/cm^2, 2 hour) respectively.

Specimens with dimensions 100 × 100 × 12 mm were prepared for Cone calorimeter testing according to KS F ISO 5660-1. Prepared specimens of plywood were conditioned at 50% R.H. and 25°C prior to the Cone calorimeter tests. Tests were carried out at an external irradiance of 50 kW/m^2 in a horizontal orientation.

The capacity of the treated wood was estimated by KS F ISO 5660-1 (Voltage: 50 kW, Heating time: 300 seconds).

3. RESULTS and DISCUSSION

The electron beam treatment was accepted for the increase retention value. Table 1 show the weight increase rate of the spacemen treated with different retardants after electron beam treatment. The radiate pine shows the most retention value among the three control specimens. But radiate pine did not increase the retention value remarkably after the electron beam treatment compared to other 2 specimens. Japanese pine tree increased 30% compared to the control. Hemlock also increased by electron beam pretreatment to 49%. The relation between the intensity of the electron beam and the retention of chemical did not show the coincidence. The

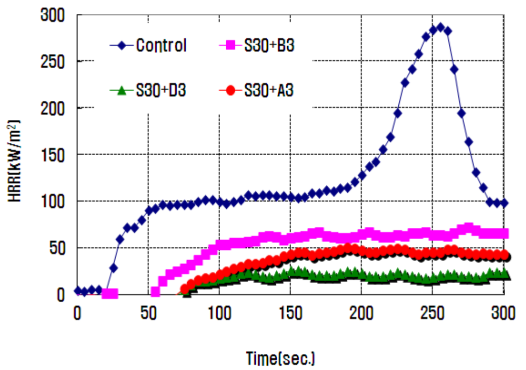

Fig. 1. HRR of Japanese red pine treated wood.
*S30 + B3 ; Sodium silicate (30%) + Boric acid (3%),
S30 + D3 ; Sodium silicate (30%) + Diammonium phosphate(3%),
S30 + A3 ; Sodium silicate (30%) + Ammonium borate (3%)

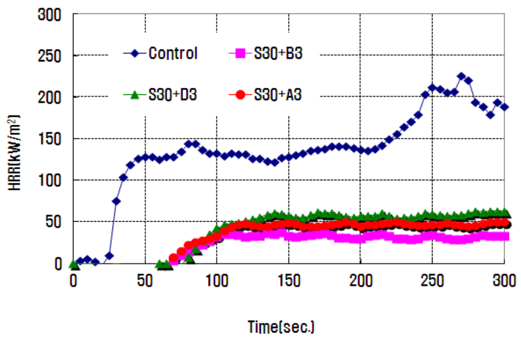

Fig. 3. HRR of Hemlock treated wood.
*S30 + B3 ; Sodium silicate (30%) + Boric acid (3%),
S30 + D3 ; Sodium silicate (30%) + Diammonium phosphate (3%),
S30 + A3 ; Sodium silicate (30%) + Ammonium borate (3%)

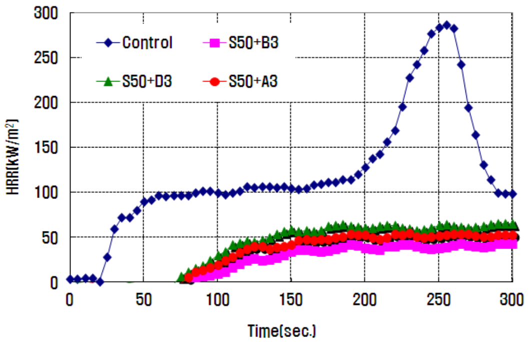

Fig. 2. HRR of Japanese red pine treated wood.
*S50 + B3 ; Sodium silicate (50%) + Boric acid (3%),
S50 + D3 ; Sodium silicate (50%) + Diammonium phosphate (3%),
S50 + A3 ; Sodium silicate (50%) + Ammonium borate (3%)

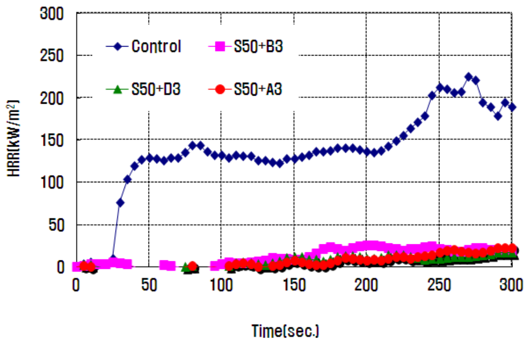

Fig. 4. HRR of Hemlock treated wood.
*S50 + B3 ; Sodium silicate (50%) + Boric acid (3%),
S50 + D3 ; Sodium silicate (50%) + Diammonium phosphate (3%),
S50 + A3 ; Sodium silicate (50%) + Ammonium borate (3%)

more specific research work that analysis for effect of electron beam to wood are needed.

The fire retardant performance of the retardant treated wood was estimated by a cone calorimeter. The ignition time, heat release rate and oxygen consumed rate were estimated. The ignition time of the specimen treated with fire retardant agent is shown in Table 2. The ignition time of the retardant wood had big differences among the wood species and the chemical combination used. The ignition time of treated wood was largely influenced by weight increase rate. The test specimens treated with the 50 percent sodium silicate and 3 percent diammonium phosphate, 50 percent sodium silicate and 3 percent ammonium borate were most delay ignition

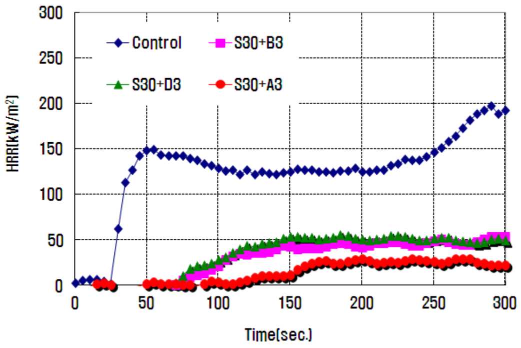

Fig. 5. HRR of Radiata pine treated wood.
*S30 + B3 ; Sodium silicate (30%) + Boric acid (3%),
S30 + D3 ; Sodium silicate (30%) + Diammonium phosphate (3%),
S30 + A3 ; Sodium silicate (30%) + Ammonium borate (3%)

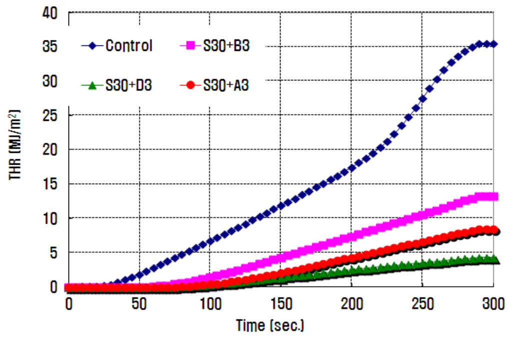

Fig. 7. THR of Japanese red pine treated wood.
*S30 + B3 ; Sodium silicate (30%) + Boric acid (3%),
S30 + D3 ; Sodium silicate (30%) + Diammonium phosphate (3%),
S30+A3 ; Sodium silicate (30%) + Ammonium borate (3%)

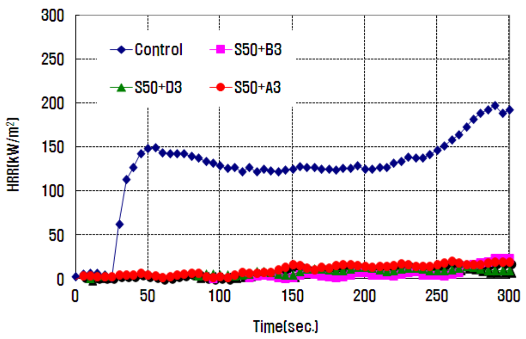

Fig. 6. HRR of Radiata pine treated wood.
*S50 + B3 ; Sodium silicate (50%) + Boric acid (3%),
S50 + D3 ; Sodium silicate (50%) + Diammonium phosphate (3%),
S50 + A3 ; Sodium silicate (50%) + Ammonium borate (3%)

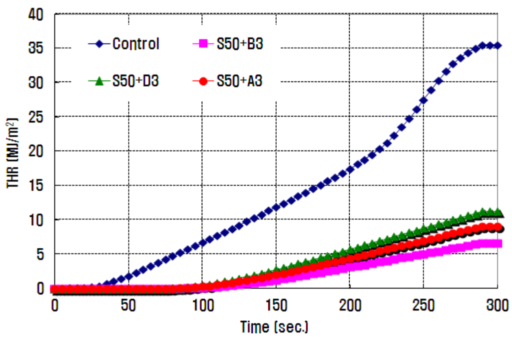

Fig. 8. THR of Japanese red pine treated wood.
*S50 + B3 ; Sodium silicate (50%) + Boric acid (3%),
S50 + D3 ; Sodium silicate (50%) + Diammonium phosphate (3%),
S50+A3 ; Sodium silicate (50%) + Ammonium borate (3%)

time due to high retention value.

Heat release test results of specimens are shown in Figs. 1～6. The standards (3-grade) of the KS F ISO 5660-1 present that the peak heat release rate should under 200 kW/m^2 during 10 seconds. The peak heat release rate of an untreated specimen exceed 200 kW/m^2. All of the specimens were satisfied this guideline. The most effective value shows on the sodium silicate (50%) hemlock treated wood.

The untreated wood shape of the curve shows typical for wood. The first part of the curve shows conditions at ignition. When the sustained flame burning begins the heat release rate is high since the char is not present on the surface and there is no barrier for heat transfer into the material. However in the process of pyrolysis of wood the char layer is formed. This

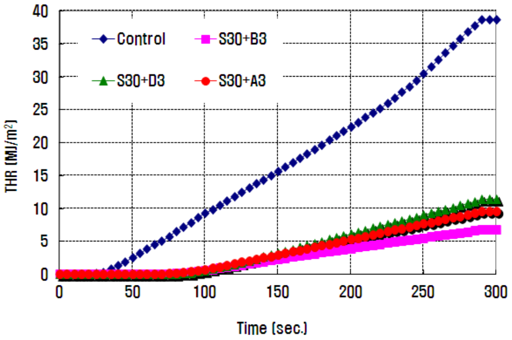

Fig. 9. THR of Hemlock treated wood.
*S30 + B3 ; Sodium silicate (30%) + Boric acid (3%),
S30 + D3 ; Sodium silicate (30%) + Diammonium phosphate (3%),
S30 + A3 ; Sodium silicate (30%) + Ammonium borate (3%)

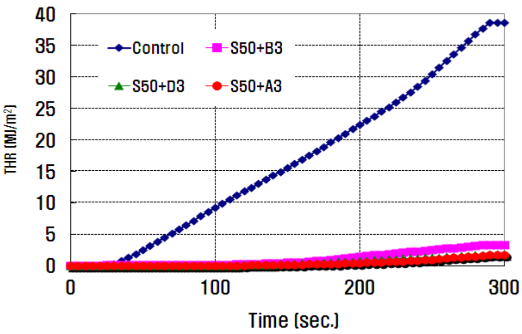

Fig. 10. THR of Hemlock treated wood.
*S50 + B3 ; Sodium silicate (50%) + Boric acid (3%),
S50 + D3 ; Sodium silicate (50%) + Diammonium phosphate (3%),
S50 + A3 ; Sodium silicate (50%) + Ammonium borate (3%)

char layer causes a barrier for heat transfer into the material and the process of pyrolysis is slowed down. But in the treated wood ignition was slowed down and the second peak did now showed, which means the burning did not happened.

The Total heat release rate (THR) of each specimens is shown in Figure 7～12. According to the KS F ISO 5660-1, THR (Total Heat re-

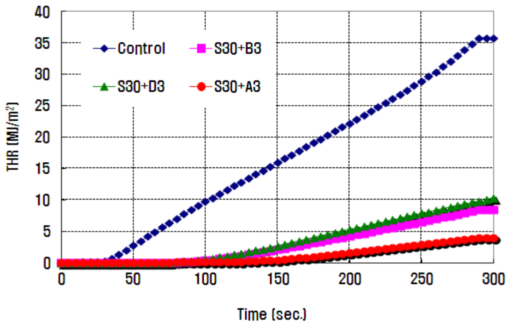

Fig. 11. THR of Radiata pine treated wood.
*S30 + B3 ; Sodium silicate (30%) + Boric acid (3%),
S30 + D3 ; Sodium silicate (30%) + Diammonium phosphate (3%),
S30 + A3 ; Sodium silicate (30%) + Ammonium borate (3%)

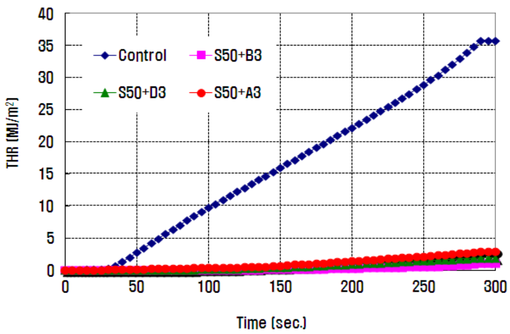

Fig. 12. THR of Radiata pine treated wood.
*S50 + B3 ; Sodium silicate (50%) + Boric acid (3%),
S50 + D3 ; Sodium silicate (50%) + Diammonium phosphate (3%),
S50 + A3 ; Sodium silicate (50%) + Ammonium borate (3%)

lease rate) value on 3rd-grade should under 8 MJ/m^2. The Japanese red pine was over 8 MJ/m^2 on the 200 seconds. The content was 30% in the hemlock treatment and sodium silicate, over the 8 MJ/m^2 on the 230 seconds.

As increasing the sodium silicate content, the Hemlock treatments satisfied the THR value of the every chemical combinations. There was no crack in the back surface of the every treated

Table 2. Gas emission of the fire retardant treated wood

Species	Treatment	CO emission (kg/kg)	CO_2 emission (kg/kg)	CO/CO_2	Total smoke emission (m^2/m^2)
Japanese red pine	Control	0.016	1.042	0.015	112.565
	S50 + B3	0.017	0.682	0.025	13.741
	S50 + D3	0.016	0.738	0.022	26.958
	S50 + A3	0.014	0.719	0.019	16.592
Hemlock	Control	0.023	1.143	0.020	380.22
	S50 + B3	0.047	0.573	0.082	2.334
	S50 + D3	0.037	0.452	0.082	0.109
	S50 + A3	0.041	0.554	0.074	1.291
Radiata pine	Control	0.023	1.180	0.020	351.42
	S50 + B3	0.014	0.699	0.020	8.443
	S50 + D3	0.014	0.733	0.019	0.654
	S50 + A3	0.032	0.602	0.053	1.807

* S50 + B3 : Sodium silicate (50%) + Boric acid (3%)
S50 + D3 : Sodium silicate (50%) + Diammonium phosphate (3%)
S50 + A3 : Sodium silicate (50%) + Ammonium borate (3%)

wood specimen after test.

The CO and CO_2 gas emission of the each spceimen treated with fire retardants is shown Table 2. The smoke emission of the retardant treated wood was remarkably reduced compared to control. The CO gas emission shows the same value for all specimens including control. The CO_2 gas mass of the all treated wood were lower than that of control wood.

4. CONCLUSIONS

The fire retardant performance of inorganic fire retardant treated wood was investigated. The electron beam treatment showed possibility of the pre-treatment methods for increasing penetration of retardants. The sodium silicate and ammonium phosphate, ammonium borate was effective to fire retardant of the woods. The effective chemical combination, 50% sodium silicate and 3% ammonium borate was accounts for grade 3 of the KS F ISO 5660-1 standards. The retardant treated wood could reduced the CO_2 gas emission.

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