Manufacture of Semi Non-combustible Wood-fiber Insulation Boards by Inorganic Fire-retardant Treatment

Se-Hwi Park, Min Lee,* Eun-Chang Kang, Sang-Min Lee, and Kugbo Shim

Fire-retardant performance was imparted to the existing wood-fiber insulation boards (WIB) *via* internal and external treatment with silica- and phosphorus-based fire-retardants. The combustion and smoke characteristics were investigated using a cone calorimeter. Based on combustion for 600 s, the weight loss and shrinkage of WIBs decreased due to fire-retardant treatment. The time to ignition was delayed to more than 400 s on the WIBs treated internal and external fire- retardant (WIB-IEs), whereas that of WIB with only internal treatment (WIB-I) was 5 s. The overall heat release rate (HRR), HRR_{peak}, and total heat release (THR) of WIB-IE specimens decreased, and the fire resistance standard Class II was satisfied. The WIB-IE2 showed higher fire resistance performance, with a HRR_{mean} level of 6.7 kW/m^2 and a THR of 1.3 MJ/m^2. The WIB-I showed extremely low total smoke release (TSR) compared to the external fire-retardant treated specimen. However, the externally treated WIB-IEs had an increased TSR of 165 to 256 m^2/m^2 due to the increase in incomplete combustion caused by the fire-retardant. After fire-retardant treatment, CO_2 generation decreased because the rate of complete combustion decreased, but CO emission increased slightly. Therefore, silica- and phosphorus-based fire-retardants by internal and external treatments were suitable for WIBs.

Keywords: Wood-fiber insulation; Fire resistance; Two-step treatment; Urea-formaldehyde resin; Cone calorimeter

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INTRODUCTION

In recent years, the public interest in environmentally friendly and energy-efficient buildings has been increasing. Additionally, the energy policies for buildings are becoming transformed toward increasing the energy efficiency. Currently, the applicability of various technical elements is progressing to realize such environmentally friendly and energy-efficient buildings. The technologies for reducing the energy demand have been increasingly emphasizing the development of efficient external and internal insulation, as well as high-performance windows and doors.

Since the industrial revolution, cheap petrochemical products are being produced superfluously and have become an indispensable part of life. The insulation materials utilized in the construction activities (apartment, building, plants, *etc.*) in South Korea are primarily polystyrene (PS) foam, polyurethane (PU) foam, and fiberglass. The PS foam and PU foam insulation possess the advantages of light weight and excellent thermal conductivity; however, they are vulnerable to fire and produce toxic gases during combustion. Particularly, because commercial insulation products are treated with halogen and phosphorus-based fire retardants, the harmful gases generated during combustion can cause many deaths and damage to properties. In contrast, fiberglass insulation exhibits good tolerance to fire and biodegradation, but its thermal conductivity is higher than that of PS and PU insulation. Moreover, fiberglass particulates have proven to be harmful to the human body.

In order to find better substitutes for petroleum-based derivatives, researchers and developers have been focusing on environmentally compatible materials derived from renewable sources (Pimentel 2003; Chen *et al.* 2015a). Wood products, which are both environmentally friendly and natural, have been receiving significant attention as substitutes for petroleum-based polymers. Ever since the development of dry process fiberboards in the 1960s, boards have been made to the desired density and thickness by applying heat and pressure with a variety of adhesives (Youngquist 1999). Furthermore, ultra-low-density fiberboards have been developed using the liquid frothing method, and they are typically treated with various chemicals to impart fire-retarding properties (Xie *et al.* 2011; Özdemir and Tutuş 2013; Niu *et al.* 2014; Chen *et al.* 2015a,b,c).

Wood fiber has been used as a raw material for insulation in low-density fiberboards, and can replace petrochemical insulation; further, it has an additional advantage of being recyclable. Low-density fiberboard is a high-quality construction material with excellent thermal conductivity and dimensional properties (Kawasaki *et al.* 1998). Many studies have revealed that the thermal conductivity of a wood-fiber insulation board (WIB) depends on its density and other factors, such as the type of resin, resin content, and length of the fiber (Kawasaki *et al.* 1998; Lee *et al.* 2019).

Most existing insulation materials are combustible and are composed of substances that can release toxic gases that are harmful to the human body, thus limiting their application (Thomsen *et al.* 2001; Debenest *et al.* 2010; Uddin *et al.* 2020). To overcome this problem, alternative materials are being studied and developed. However, noncombustible and flame-retardant thermal insulating materials have not been developed yet (Uddin *et al.* 2020). In a previous study, WIB was prepared with a melamine-urea-formaldehyde resin adhesive (35% resin content), but the developed boards were still susceptible to fire damage (Lee *et al.* 2019b). On the other hand, in the previous research of WIB flame retardant, a particular method was used due to concerns about the weak strength of WIB and the decrease of thermal properties. Although some of the fire retardant performance was demonstrated through this method, it might be difficult to apply to ordinary fire retardant products. Therefore, there is need for research focusing on simplifying the flame retardant treatment process and expanding its generality. In conclusion, in this study, a semi-noncombustible WIB was prepared using urea-formaldehyde (UF) resin adhesive with a two-step fire-retardant treatment.

EXPERIMENTAL

Materials

Wood fibers (*Pinus densiflora*, 7.2% moisture content, M.C.) and a 60% wax emulsion were obtained from Dongwha Enterprise (Incheon, Korea). The mean dimensions of the wood fibers were 1.65 mm (length) × 37.2 µm (width). The distribution of the lengths of the wood fibers is displayed in Table 1. Formaldehyde solution (37 wt%) was provided by the Sunchang Corporation (Incheon, Korea), and urea (99%) was purchased from Hu-chems (Seoul, Korea). All the chemical reagents used in this study were of American Chemical Society (ACS) grade, and were purchased from DaeJung Chemicals & Metals Co., Ltd. (Siheung-si, Korea). The curing agent consisted of ammonium chloride, which is used widely. The prepared ammonium chloride was diluted with water and adjusted to a concentration of 20%.

Table 1. Distribution of the Lengths of the Wood Fibers Used in This Study

Fiber Length (mm)	< 0.3	0.3 to 0.5	0.5 to 1.0	1.0 to 2.0	2.0 to 3.0	> 3.0
Distribution (%)	2	35	30	13	17	3

In this study, wood fiber, which is an organic material, was treated with internal/external (IE) fire retardants to realize fire-resistance performance. Internal fire retardants were intended to impart fire resistance performance to an insulation board, whereas external fire retardants were applied on the surfaces of the board (both front and rear) to enhance the fire-resistance performance. Examples of internal and external fire retardants are listed in Table 2. Internal fire retardants are generally utilized in the resin application process, which can affect the performance of the adhesive. Therefore, the internal fire retardant used in this study was selected by considering its miscibility with the adhesive, appropriate pH that does not affect the curing speed of the adhesive, and appropriate solid content to minimize the amount of fire retardant used. Three types of aqueous fire retardants, all containing phosphate, phosphorus, and silica in different proportions, were used as the external fire retardant that was applied on the surfaces after hot pressing. The external fire retardant 1 (E1) was diluted with water to achieve appropriate viscosity and solids content.

Table 2. Main Constituents and Properties of Internal and External Fire Retardants

Name	Type	Base Component	pH	Specific gravity (mg/mL)	Provider†
I	Internal	Phosphoric acid	4.9	1.107	Jinyoung PAC
E1	External	Sodium silicate	11.0	1.878	Self-developed
E2		Phosphate, Silica	6.0	1.166	GG Chemical
E3		Phosphoric acid	7.9	1.129	Jeonbuk National University

†Jinyoung PAC, Pusan, Korea; GG Chemical, Pyuengtaek, Korea; Jeonbuk National University, Jeonju, Korea

Methods

Synthesis of adhesive

The UF resin was synthesized by an alkaline–acid reaction at a molar ratio of 0.85 F/U. The characteristics of the synthesized resin were determined according to the Korean Standard KS M 3705 (2015). The final pH, solid content of the UF resin, specific gravity, final viscosity, and gel time at 100 °C of the synthesized UF resin were 7.8, 57.4%, 1.22 mg/mL, 102 mPa·s, and 163 s, respectively.

Manufacturing of the wood insulation board

The manufacturing process of WIB is illustrated in Fig. 1. The size of the WIB was 350 mm × 300 mm, and its thickness was 20 mm. Further, the target density was 150 kg/m^3, and the resin content was 35% of the weight of the dried wood fiber. A 60% wax emulsion was added with 1% by weight of the dried wood fibers, and 3% of hardener by weight of the adhesive's solid content was added in the adhesive.

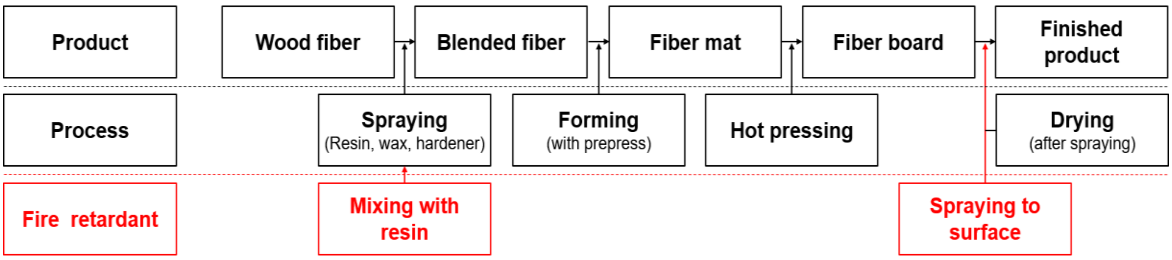

Fig. 1. Manufacturing process of wood fiber insulation boards in this study

The amount of internal fire retardant used was based on 5% of the weight of dried wood fibers, considering the performance of the fire retardant. The adhesive, wax emulsion, hardener, and internal fire retardant were individually weighed and mixed, and then sprayed onto a wood fibers in a drum mixer. The treated wood fibers were weighed based on the target density in a molding frame to make a mat; the measured moisture content of the mat was between 16% and 17%. The as-prepared mat was hot-pressed (AT-S200T; Anjeon Hydraulic Machinery Co. LTD., Gimpo-si, Korea) at 150 °C for 5 min at a target pressure of 5 kgf/cm^2 in a thermocompressor.

After hot pressing, the prepared WIBs were cooled and treated with three different external fire retardants on both the face and back surfaces; 90 g/m^2 of each fire retardant was sprayed on the WIBs on each side. Thus, the WIBs treated with fire retardants were dried in a conventional oven for 3 h at 80 °C and then stored at a temperature of 20± 1 °C at a relative humidity of 65 ± 5%.

Table 3. General Information of the Internal and External Fire Retardants

Sample Name†	Internal Retardant	External Retardant			
		Type	Solid Content (%)	Viscosity (mPa·s)	Spread Volume (g/m^2)
WIB-I	I	-	28.0	30	-
WIB-IE1		E1	50.0	150	90
WIB-IE2		E2	28.7	28	90
WIB-IE3		E3	26.3	63	90

†WIB-I: Wood insulation board treated by internal fire retardant; WIB-IE: wood insulation board treated by internal and external fire retardants

Evaluation of fire performance

The combustion and smoke release properties, such as heat release rate (HRR), total heat release (THR), total smoke release (TSR), and CO/CO_2 release, were measured for each fire retardant-treated WIB according to the KS F ISO 5660-1 (2018) standard by the cone calorimeter (Fire Testing Technology Ltd, East Grinstead, UK). Further, the rate of change in length, width, thickness, and weight loss after burning for 600 s was investigated. According to the Building Standard Law of Korea, three classes of fire resistance for interior finishing materials are specified (Table 4). Class I (non-combustible materials) requires testing for 20 min for a combustion of 50 kW/m^2. For Class II (semi-noncombustible materials), the testing condition is 10 min for a combustion of 50 kW/m^2. Class III (fire-retardant materials) requires 5 min for a combustion of 50 kW/m^2. Each class requires less than 8 MJ/m^2 of THR, and the HRR should not exceed 200 kW/m^2 for 10 s.

Table 4. Performance Standards of Fire Retardancy Test Conditions (KS F ISO 5660-1 (2018))

Class	Test Condition		Performance Standard
	Heating Condition	Heating Time	
Non-combustible Material	50 kW/㎡	20 min	- THR is less than 8 MJ/m^2 - HRR_{peak} is under 200 kW/m^2 for 10 consecutives - Tunnels, cracks, and holes are not found after test
Semi non-combustible Material	50 kW/㎡	10 min	
Fire-resistant Material	50 kW/㎡	5 min	

Thermal conductivity

The density and thermal conductivity of WIBs were evaluated according to KS F 3200 (2016) and KS L 9016 (2017) standards. A thermal conductivity analyzer (λ-Meter EP500e; Messtechnik GmbH, Dresden, Germany) was used to analyze the thermal conductivity in this study. The tested samples were cut to the dimensions of 20 cm × 20 cm.

RESULTS AND DISCUSSION

Insulation Performance

The insulation properties of the insulation materials are generally evaluated based on the thermal conductivity, thermal permeability, and thermal resistance, which are typically closely related to the density and microstructure of the insulation materials (Uysal *et al.* 2009; Zahedsheijani *et al.* 2012). Table 5 shows the insulation properties of the fire retardant-treated WIBs. The densities of the prepared WIB-Is and WIB-IEs were 0.149 to 0.155 g/cm^3. The WIBs were prepared such that their density was close to the target density of 0.150 g/cm^3, but the WIB-IEs showed slightly higher values due to the external fire-retardant treatment. The thermal conductivities of the specimens were 0.0359 to 0.0387 W/m·K, which were generally similar to those of a previous study (Lee *et al.* 2019b). However, the thermal conductivities of WIB-IE1 and WIB-IE2 decreased slightly to 0.0370 W/m·K and 0.0359 W/m·K, respectively; thus, silica could hinder the objective of improving the insulation performance.

This phenomenon is caused by the anisotropic mechanism of heat conduction on the WIBs due to the separation of the structure between the surface and the interiors (Sulistyo *et al.* 2009; Pradha *et al.* 2012). Due to the fire-retardant treatment on the front and rear sides, the WIBs were represented by three layers of different densities. The surface (front and rear) layer, which became relatively complicated and hardened by the application of silicate, hindered the heat transfer to the inner layer. As the heat migration in the horizontal direction along the surface layer increases, heat loss may occur, making heat migration into the inner layers of the WIB difficult (Sulistyo *et al.* 2009).

Table 5. Density and Insulation Properties of the Wood-fiber Insulation Boards Samples Manufactured by Different Treatments

Samples	WIB-I	WIB-IE1	WIB-IE2	WIB-IE3
Density (g/cm^3)	0.149	0.155	0.154	0.155
Thermal Conductivity (W/m·K)	0.0380	0.0370	0.0359	0.0387
Thermal Transmittance (W/m^2·K)	2.023	1.986	1.892	2.016
Heat Resistance (m^2·K/W)	0.494	0.504	0.529	0.496

In contrast, WIB-I and WIB-IE3, which were commonly treated with a phosphoric acid-containing fire retardant, exhibited a small improvement in thermal conductivity. Phosphoric acid, which contains three hydroxyl groups, forms a typical polyol complex with hydrogen ions in wood fibers (Chen *et al.* 2015a). This resulted in improved bonding properties and directional simplification of wood fibers, thereby improving their mechanical properties and thermal conductivity (Pradha *et al.* 2012). In addition, because the inner and outer fire retardants comprised similar components, the layer separation phenomenon was not notable compared between WIB-IE1 and WIB-IE2.

Combustion Performance

Figure 2 shows the specimens and Table 6 lists the changes in dimensions of the specimens, both after burning for 600 s. The changes in length, width, thickness, and weight of WIB-I after combustion were 19.5%, 19.3%, 31%, and 83%, respectively. The WIB-IEs with external fire-retardant treatment improved the overall reduction rate. A carbonization layer was formed on the external fire retardant-treated WIBs, but not on the internal fire retardant-treated WIBs. Phosphorus and silica were the major constituents of the three fire retardants. Phosphorus reacts with water and oxygen during combustion to produce condensed phosphate. Soot and condensed phosphate combine to form a thin layer on the surface that blocks the contact of oxygen during combustion (Pan *et al.* 2012).

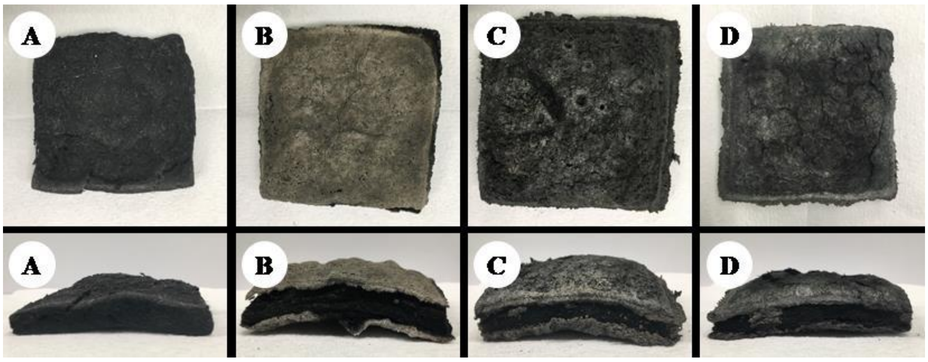

Fig. 2. Images of WIB-I and WIB-IEs after burning for 600 s: (A) WIB-I, (B) WIB-IE1, (C) WIB-IE2, and (D) WIB-IE3

The charcoal layer, known as char, serves to protect the combustion source inside the material. Sodium silicate or silicic acid contained in the silica-based fire retardant forms a physically strong char layer upon combustion (Kashiwagi *et al.* 2000). The changes in the dimensions of WIB-IE2 were smaller than those of the other specimens because the char layer, which prevented the action of the fire retardant containing phosphorus and silicon, functioned effectively. In contrast, WIB-IE3 exhibited slightly larger changes in dimensions than the other two specimens treated by external fire retardants. Therefore, it can be concluded that the silica-based fire retardant showed better fire-retardant performance than the phosphorus-based fire retardant.

Table 6. Dimensional Change Rates of the Wood-fiber Insulation Boards After Burning for 600 s and Analyzed by Cone Calorimeter

Samples	WIB-I	WIB-IE1	WIB-IE2	WIB-IE3
Length (%)	19.5	9.7	4.9	15.0
Width (%)	19.3	9.8	6.7	13.3
Thickness (%)	31.1	11.6	3.3	19.5
Weight (%)	83.0	51.1	51.3	62.5

Combustion Properties

The cone calorimeter is the most widely used device in the field of fire safety engineering for studying the fire behavior of various materials in the thermal degradation stage. The condensation process that occurs by the removal of moisture from an object is based on the principle that the heat produced in the combustion of all organic materials is directly related to the amount of oxygen required for combustion (Huggett 1980). Table 7 lists the results of combustion properties for WIB-I and WIB-IEs.

Time to ignition (TTI) is defined as the time at which a specimen burns for at least 10 s; it is an important factor evaluated as a measure of ignitability and commencement of combustion (Niu *et al.* 2014). The WIB-I, treated only with an internal fire retardant, reached the TTI in 5 s, and showed similar performance as the WIBs without fire-retardant treatment (Lee *et al.* 2019). In contrast, the TTI of WIB-IEs was delayed by 400 s; thus, it was considered to be able to resist fire exposure for at least 400 s. It has been reported that low-density fiberboards do not withstand high radiant heat (50 kW/m^2), and that their TTI is short, *i.e.*, less than 20 s (Tsantaridis 2003; Niu *et al.* 2014; Chen *et al.* 2015). External fire retardants have been shown to have a remarkable effect on delaying the ignition time.

The effective heat of combustion (EHC) is usually defined as the heat released on a complete combustion of a material under standard conditions and provides additional information regarding the fire-retarding behavior of the material. The EHC is derived from the mass loss rate (MLR) calculated by the ignition time and a fixed time interval. The MLRs of the specimens were 0.041 g/s (WIB-I), 0.024 g/s (WIB-IE1), 0.024 g/s (WIB-IE2), and 0.028 g/s (WIB-IE3). The MLR results showed the same trend as the weight loss rate. The EHC of WIB-IEs was 1.75 to 12.9 MJ/kg, which was less than that of WIB-I (19.9 MJ/kg). Wood-based materials with more than one decomposition form (C, CO, CO_2, NO_x, H_2O, SO_x, *etc.*) have inconsistent EHC values (Luche *et al.* 2011). Furthermore, the decomposition behavior is made more complicated by each fire-retardant treatment with different components; thus, the EHC range is wider. Particularly, WIB-IE2 was measured to have an EHC of 1.75 MJ/kg, which is extremely low compared with other specimens. In general, EHC is proportional to the amount of carbon and carbon-intensive char, which combust easily (Babrauskas 2006). Therefore, the lower EHC of WIB-IE2 was due to the lack of combustible carbon or the char formation caused by the fire retardant during the thermal decomposition.

Table 7. Combustion Properties of Wood-fiber Insulation Boards Manufactured with Different Treatments

Samples	TTI (s)	MLR_{mean} (g/s)	EHC (MJ/kg)	SMLR (g/s·m^2)	HRR_{mean} (kW/m^2)	HRR_{peak} (kW/m^2)	THR (MJ/m^2)
WIB-I	5	0.041	19.9	4.45	51.0	99.78	30.9
WIB-IE1	488	0.024	8.70	3.08	25.6	31.87	3.1
WIB-IE2	418	0.024	1.75	3.34	6.7	21.91	1.3
WIB-IE3	409	0.028	12.9	2.58	22.7	36.28	4.6

TTI: time to ignition, MLR: mass loss rate, EHC: effective heat of combustion, SMLR: specific mass loss rate, HRR_{mean}: mean heat release rate, HRR_{peak}: peak heat release rate, THR: total heat release

The specific mass loss rate (SMLR) is the ratio of the MLR and the surface area of the specimen exposed to heat flux. It can be used to calculate the HRR by multiplying with EHC. The external fire retardant-treated WIBs showed lower SMLR, and it was possible to confirm whether the surface of the specimen was resistant to fire. The SMLR of WIB-IE2 (3.34 g/s·m^2) was the highest among the other WIB-IEs, but the HRR was lower because of the lower EHC.

The HRR is the most important factor reflecting the risk of fire; higher HRR indicates that heat could more easily affect the surface of the specimen (Babrauskas and Richard 1992). The HRR and THR curves for the combustion behavior of the WIB-I and WIB-IEs are shown in Figs. 3 and 4, respectively.

For WIB-I, the typical combustion characteristics were 51.0 kW/m^2 (HRR_{mean}), 99.7 kW/m^2 (HRR_{peak}), and 30.9 MJ/m^2 (THR). In addition, the internal fire-retardant treatment did not affect the combustion suppression, which was similar to the results of a previous study (Lee *et al.* 2019). Two general peaks occurring in the medium-density fiberboard were observed in the HRR curve of the WIB (Lee *et al.* 2011).

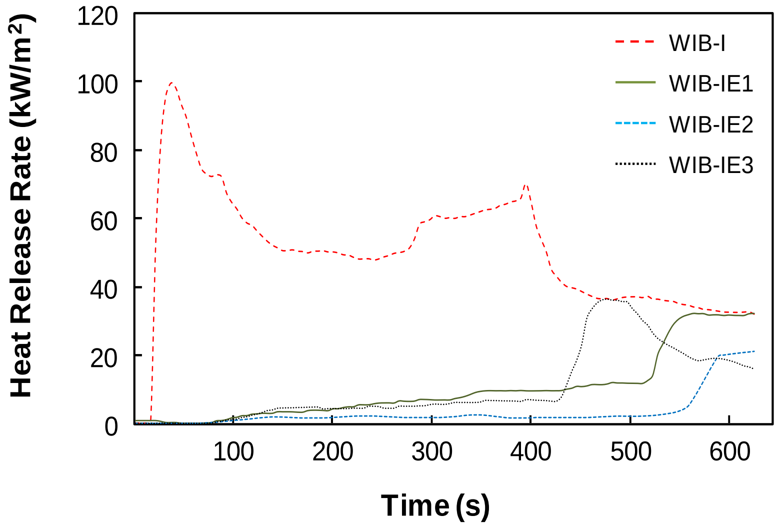

Fig. 3. Heat release rate curve of the wood-fiber insulation boards manufactured with different treatments

The first sharp peak appeared after ignition and was the most important factor in evaluating the fire intensity. The second peak was observed after 350 s, and the transmitted heat wave was reflected from the rear of the test specimen, which was an effect of increasing the combustion rate (MSHA 2015). In contrast, the HRR peaks of WIB-IEs in the earlier combustion stage were not observed due to sufficient thickness of the carbonization layer formed by the fire retardant (Tsantaridis 2003; Lee *et al.* 2011). The WIB-IEs showed a remarkably lower HRR after the commencement of combustion; HRR_{peak} was reached at 530 s (WIB-IE1), 600 s (WIB-IE2), and 480 s (WIB-IE3). Then, the HRR decreased, and the carbide on the surface of the WIBs tended to enter an afterglow effect stage, in which it turned into gray ash.

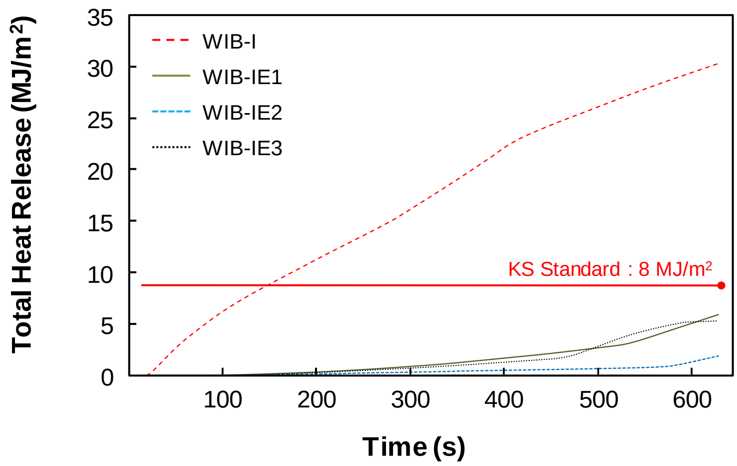

Fig. 4. Total heat release curve of the wood-fiber insulation boards manufactured with different treatments

The observed HRR_{peak} and HRR_{mean} values generated during this combustion series were 21.9 to 36.9 kW/m^2 and 6.7 to 25.6 kW/m^2, respectively. The results showed a notable difference from the existing fiberboards without external fire-retardant treatment. The HRR_{peak} did not exceed 200 kW/m^2 for 10 s; thus, it met the standard. Finally, this trend was reflected in THR; all WIB-IEs achieved 8.0 MJ/m^2, which corresponded to Class II fire retardant standards in THR (KS F ISO 5660-1 (2018)). Although there were differences in the combustion characteristics by the fire retardant, it was determined that the external treatment method was more effective than the internal treatment. Table 8 shows the manufacture method and combustion properties of other studies related to WIB fire retardant treatment. In the manufacture process, the fire retardant was mixed with fibers. The combustion performances of the specimens produced by internal and external flame retardant treatment (two-step treatment) were 6.7 to 25.6 kW/m^2 (HRR_{peak}), 1.3 to 3.1 MJ/m^2 (THR), and 51.1 to 62.5% (weight loss), respectively. The overall results were improved and it was confirmed that the two-step treatment of this study was effective.

Table 8. Combustion Properties of WIBs According to Different Manufacture Methods

Name	Fire retardant	Manufacture method	HRR_{peak} (kW/m^2)	THR (MJ/m^2)	WL (%)	References
KPa	Si-Al	Blended to fiber	84.52	10.05	-	Niu *et al*. 2014
SF	Si-Al	Blended to fiber	141.29	15.63	-	Chen *et al*. 2015a
FF	CP		153.74	21.36	-	
MF	Si-Al, CP		100.76	14.77	-	
Si10	10% Si sol	Blended to fiber	-	-	56.2	Chen *et al*. 2015b
MT1	Inorganic film	Coated to board	-	-	-	Chen *et al*. 2015c
WL: Weight loss, Si-Al: Si-Al compounds, CP: chlorinated paraffins						

Smoke Properties

Smoke suffocation is the most dangerous factor in a fire damage; the smoke in the combustion phase is analyzed through many ways. The measured parameters related to smoke generation are listed in Table 9.

Table 9. Smoke Properties of the Wood-fiber Insulation Boards Manufactured with Different Treatments

Samples	TOC (g)	TSR (m^2/m^2)	SEA (m^2/kg)	COY (kg/kg)	CO_2Y (kg/kg)	CO/CO_2
WIB-I	19.8	1.5	0.08	0.0403	1.46	0.0276
WIB-IE1	2.0	165.0	32.80	0.0287	0.57	0.0504
WIB-IE2	1.0	256.0	86.63	0.0256	0.21	0.1219
WIB-IE3	2.9	166.8	177.92	0.0533	0.75	0.0711
TOC: total oxygen consumed, TSR: total smoke release, SEA: specific extinction area, COY: carbon monoxide yield, CO_2Y: carbon dioxide yield, CO/CO_2 ratio: COY divided by CO_2Y						

The total oxygen consumed (TOC) represents the consumption of oxygen, which is the basis of cone calorimeter measurement and is proportional to the heat of combustion generated. It showed a high correlation with the HRR and THR results. The smoke production rate (SPR) is measured by the extinction coefficient, where the light or radiation is absorbed by a smoke-measuring device; it is used to analyze the smoke generated in the combustion process. The TSR level of WIB-I was very low, *i.e.*, 1.5 m^2/m^2, due to the lower content of combustibles and complete combustion of wood fibers. The smoke produced was proportional to the amount of combustibles and the area that could be burned as per ISO 5660-1 (2018).

In a previous experiment (Lee *et al.* 2019b), the TSR of the WIB was 1.3 to 3.4 m^2/m^2; there was no notable difference from WIB-I, and the effect of the internal fire retardant on TSR during the combustion was not remarkable. In contrast, the smoke generation of WIB-IEs increased rapidly, which was determined to be due to the combustion suppression effect caused by the external fire retardant. It was determined that the incomplete combustion with a large amount of smoke increased due to the chemical or physical reactions of phosphoric acid and silicic acid. Chen *et al.* (2014) reported the TSR of a low-density fiberboard treated with a Si–Al fire retardant to be 130 to 140 m^2/m^2, which is similar to that in this study. In general, commercially available petrochemical-based insulations (PS and PU foam) exhibit a TSR of larger than 900 m^2/m^2. On comparing the TSRs of petrochemical-based insulations and WIB-IEs, it was established that low TSR has the advantage of reducing the death rate caused by smoke in the event of a fire. In addition, the smoke release pattern of each material revealed that the WIB-IEs released smoke slowly for longer periods, whereas the petrochemical-based insulation material released smoke in a relatively short period (less than 1 min) with a high SPR.

The specific extinction area (SEA) is obtained by dividing the SPR by the MLR, which tends to decrease with the increase incomplete combustion (Bei *et al.* 2014). The WIB-I with low smoke generation had a very low SEA of 0.08, whereas a relatively higher SEA was determined for WIB-IEs. Carbon monoxide and CO_2 generated during the combustion of combustible materials have been managed intensively over a long period of time because even a small amount of these gases is dangerous to the human body (King 1949; White and Dietenberger 2010). The yield of CO and CO_2 and their concentration from the WIB specimens are shown in Figs. 5 and 6, respectively. Both WIB-IE1 and WIB-IE2 exhibited the reduced CO generation. The carbonized layer produced in the condensed phase of silicic acid or phosphate contained in both specimens interfered with the burning of volatile materials and hampered oxygen contact, which are associated with incomplete combustion. In contrast, WIB-IE3 had a higher CO production amount of 0.0533 kg/kg, which was related to the combustion of phosphoric acid in the vaporization stage (Kimmerle 1974).

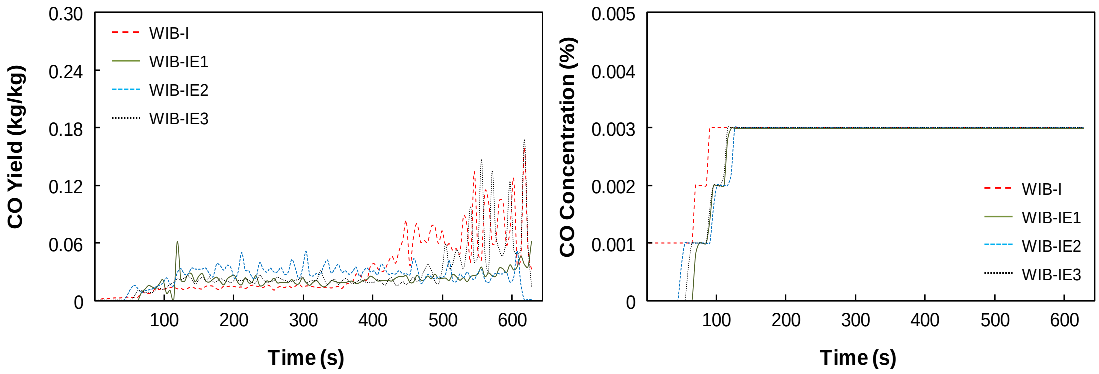

Fig. 5. CO yield and CO concentration curves for the wood-fiber insulation boards manufactured with different treatments

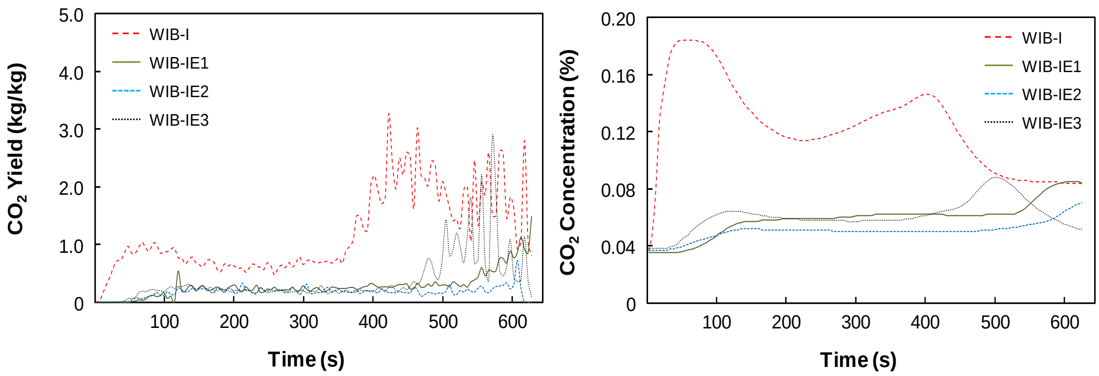

Fig. 6. CO_2 yield and CO_2 concentration curves of the wood-fiber insulation boards manufactured with different treatments

Trimethyl phosphate produced during the thermal decomposition of phosphoric acid reacts with oxygen first to interfere with methane-oxygen reaction, thereby slowing the progress of a fire (Weil and Levchik 2000). In addition, the proportion of incomplete combustion increased because it reduced the concentration of hydrogen atoms (Weil and Levchik 2000). Further, the carbon monoxide yield (COY) curve of WIB-IE3 showed a similar trend to that of WIB-I and increased after 500 s. Li (2003) and Niu *et al.* (2014) reported that large amounts of CO are released during the process of burning combustion, in which carbide is turned into ash.

The Occupational Safety and Health Administration (OSHA) recommend that occupational groups should not be exposed to higher than 200 ppm continuously for 8 h (OSHA 2009). Furthermore, CO_2 is associated with the combustion mechanism as a complete combustion by-product when the three elements of combustion (flammables, ignition, and oxygen) with continuous action are met (Chen *et al.* 2015). For WIB-I, COY soared after 350 s when the decomposition of carbides began, because it was produced by the oxidation of carbon from the organic material. For WIB-IEs, it was confirmed that a similar trend was delayed to 500 to 600 s.

CONCLUSIONS

1. Commercially available (silica- or/and phosphorus-based) and self-developed (silica-based) fire retardants were applied on wood-fiber insulation boards (WIBs) as internal treatment (mixing in resins) and external treatment (spraying on surfaces). The manufacturing process attempted in this experiment did not affect the basic properties and the thermal conductivity of the WIBs.
2. A careful observation of the specimens burned for 600 s revealed that the weight loss rate and the length reduction rate tended to decrease due to the external fire-retardant treatment. Particularly, WIB-IE2 recorded the smallest rate of dimensional change.
3. The analysis of combustion characteristics using a cone calorimeter revealed that the time to ignition (TTI) was delayed by 400 to 480 s due to the external fire-retardant treatment. The overall heat release rate (HRR), the peak value (HRR_{peak}), and total heat release (THR) of WIB-IEs were reduced substantially, and the fire retardancy Class II was satisfied. However, WIB-I showed similar combustion characteristics as a typical WIB.
4. The HRR_{mean} value was reduced to 6.7 kW/m^2 due to the low effective heat of combustion (EHC) value of 1.75 MJ/kg of WIB-IE2. The WIB-IE2 showed an overall fire-retardant performance equivalent to the fire retardant standard Class II.
5. In the total smoke release (TSR) results, WIB-I exhibited the least smoke generation (1.5 m^2/m^2). However, the TSR of the external fire retardant-treated WIBs was higher (165 to 256 m^2/m^2) due to the increase in incomplete combustion caused by the fire retardant. The TSR level of WIB-IEs was lower than that of commercial PU foams; thus, WIB-IEs have the advantage of reducing the death rate caused by smoke by providing sufficient evacuation time to escape smoke damage during a fire.
6. The two-step treatment for fire retardant showed better performance than the processing method employed in previous studies. This method is expected to be used as a technique that can be applied universally to commercial products.

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