

## Fire Properties of *Pinus densiflora* Utilizing Fire-retardant Chemicals Based on Borate and Phosphorus (II) – Thermal and Gas Emission Characteristics

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The pyrolysis characteristics of untreated pine and fire retardant-treated pine (*Pinus densiflora*) were measured by using thermogravimetric analysis according to the ASTM E1131-08 (2012) regulation. Fourier transform infrared spectroscopy was used to monitor changes in chemical groups of fire-retardant treated specimens before and after the combustion test. In addition, the microstructures of the untreated specimen and the fire-retardant treated specimen after cone calorimeter testing were determined by scanning electron microscopy. Combustion gas toxicity was evaluated according to the test method described in Naval Engineering Standard 713 (1990). The emitted combustion gases of all specimens were carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), nitrogen oxide (NO<sub>x</sub>), and acrylonitrile. The thermal decomposition rate was reduced by about one-third that of the fire-retardant treated specimen compared to the reduction rate of the untreated specimen. These results are useful for guiding the safe utilization of fire retardant-treated wood and wood-based materials for building applications.

*Keywords:* Fire retardant-treated wood; Thermal stability; Fire safety; Hazardous gases; Korean *Pinus densiflora*

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### INTRODUCTION

The requirements of fire retardants for building materials are divided into different fire resistance, including reaction to flammability and gas toxicity. Fire-retardant chemicals, which are used in various polymer materials, are added to prevent fires and delay the spread of flame. Such treatments can be very effective for materials that require fire retardancy and fire safety (Fahy and Proulxm 2001; Kawahara and Yanagisawa 2003; Kobes *et al.* 2010; Stec and Hull 2011; Veen and Boer 2012). In addition, when a fire occurs, the materials should not generate even a small amount of toxic gas. Because most fire casualties and fire-related personal injuries result from the generation of toxic gases, fire retardant chemical composites should be added to the building materials. When fires occur, this is important because it is difficult for people inside a building that is on fire to escape due to the toxic substances in the combustion gases (Fahy and Proulxm 2001; Kobes *et al.* 2010; Stec and Hull 2011).

When fire-retardant chemicals were first used, they were classified into various types, including halogen-based, brominate-based, phosphorous-based, and other inorganic chemical species (Mfiso *et al.* 2014). Among these fire retardant chemicals, the use of brominated fire retardants is forbidden in Europe. Brominated fire retardants generate a large amount of toxic gases, such as hydrogen bromide (HBr), dioxins, *etc.*, at the time of

combustion (Dassari *et al.* 2013; Liang *et al.* 2007; Mfiso *et al.* 2014). Although the use of halogen-based fire retardants is not yet regulated in Korea, studies of halogen-free fire retardants have been conducted (Jang and Choi 2009; Chung and Jin 2010; Lim *et al.* 2011). In this study, fire-retardant chemical composites (FRCs), including boron- and phosphorus-based chemicals, were developed by mixing using a water-based solvent (Seo *et al.* 2017).

In Korea, for a fire to be considered severe, it must affect more than 60% of the area in a building. In addition, according to the statistical survey of the last 10 years since 2000 in Korea, wood and wood-based materials made up 95% of the interior building materials in newly constructed apartments and houses in Korea (Park *et al.* 2011; Seo *et al.* 2013). The fire risk of materials can be estimated based on certain factors, such as speed of flame spread, heat release rate, total heat release, and generation of combustion gas (Qian *et al.* 2014; Wei *et al.* 2015; Yew *et al.* 2015). According to previous studies and statistical data on personal injuries in fires, injuries caused by toxic gas inhalation were more common than burns caused by direct flames (Liang and Ho 2007; Cho *et al.* 2012; Stec *et al.* 2008; Kobes *et al.* 2010; Molyneux *et al.* 2014). Specifically, 6.78% of fire-related deaths resulted from flames in Korea (Lee 2006). Clearly, the toxic gases emitted during combustion are of greater concern than the flames themselves. However, sufficient research has not yet been conducted to determine and evaluate the toxicity characteristics of the combustion of building materials in Korea. Therefore, it is necessary to research the improvement of fire retardant performance in wood and reduce hazardous combustion gases.

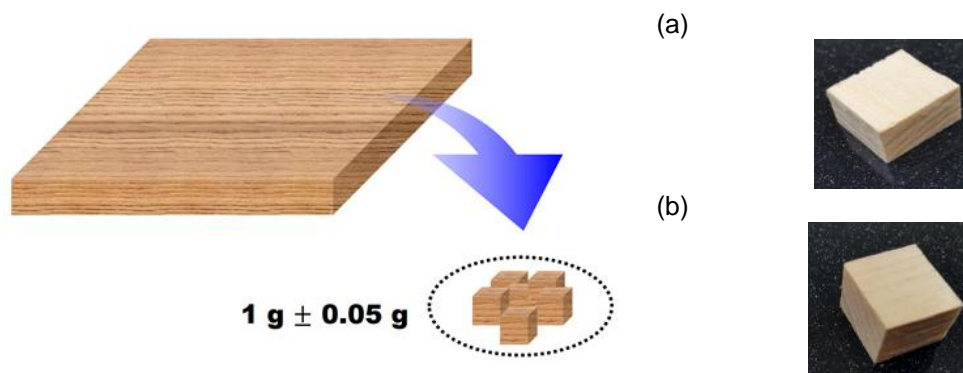
In this study, the FRCs was developed by mixing using a water-based solvent as done in prior research (Seo *et al.* 2017). The FRCs is composed of boric acid, borax, diammonium phosphate, and carbonite-based chemicals. Their composition ratios are presented in the materials part of this paper. The composites were prepared *via* the pressure-impregnation method inside wooden specimens, and the process with pressure-impregnation proceeded in the cylinder. In addition, the microstructures of the FRC-treated wood specimens were characterized after combustion tests *via* scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FT-IR). Thermogravimetric (TG) analysis was used to identify the expression of thermal stability. Furthermore, in accordance with the Naval Engineering Standard (NES) 713, it was determined that smoke was generated during the combustion of the FRC-treated wood and untreated wood. The objective of this study was to measure the toxic gas content and the toxicity index of the FRC-treated wood and untreated wood, and thereby to provide an initial database of the fire resistant wood-based building materials.

## EXPERIMENTAL

### Materials

In this experiment, Korean red pine (*Pinus densiflora* Siebold & Zucc.) from Pyeongchang in Gangwon, South Korea was used as a test specimen. It was prepared in the form of a 100 mm × 100 mm × 10 mm plate for FRC treatment and then redone to a size of 10 mm × 10 mm × 10 mm and a mass of 1 g ± 0.16 g to 0.25g (Fig. 1) according to the NES 713 standard (1990). The specimens were pretreated at 23.1 °C ± 1.8 °C and 49% ± 5% relative humidity and then stabilized for 3 h in a desiccator for FRC treatment. Figure 1 shows two types of untreated and FRC-treated specimens (Fire retardant treated wood by pressure-treatment; FRTW-pressure specimens). In the previous study (Seo *et al.* 2017), it

was concluded that pressurizing FRCs was more effective in improving the fire retardant performance of wood. Therefore, in this paper, the experiment was conducted to compare FRTW-pressure and untreated test specimens. FRTW-pressure test specimens were produced with the fire retardation process by applying pressure to FRCs.



**Fig. 1.** Prepared specimens according to NES 713; (a) Untreated wood, (b) FRTW-pressure

The specimens for thermogravimetric analysis were ball milled to sizes of less than 120-mesh, and they were dried for 48 h at 60 °C. The test specimens were reduced to 2 mm to 4 mm in size after the cone calorimeter test. The chemical compositions of the FRCs were the same as the previously conducted study (Seo *et al.* 2017). Table 1 shows the composition of the fire retardant chemicals (FRCs) used in this study and the formula of the ingredient. Among the chemical compositions of FRCs, di-ammonium phosphate and potassium carbonate were used as the main fire-retardants. The ratios given in the table are the percentages of the chemical species dissolved in water. Therefore, FRCs could be applied to wood test specimens through pressure treatment methods. Because these were not high concentrations, all of the mixtures were aqueous solutions having suitably low viscosity.

**Table 1.** Composition and Formula of Fire-retardant Chemicals (Seo *et al.* 2017)

Distribution	Formula	CAS No.	Dissolution Characteristics	Composition Rate (wt.%)
Fire retardant chemicals				
Di-ammonium phosphate	$(\text{NH}_4)_2\text{HPO}_4$	7783-28-0	Water-soluble	20
Di-sodium tetraborate decahydrate	$\text{Na}_2\text{B}_4\text{O}_7$	1303-96-4	Water-soluble	3
Potassium carbonate	$\text{K}_2\text{CO}_3$	584-08-7	Water-soluble	20
Ammonium carbonate	$(\text{NH}_4)_2\text{CO}_3$	506-87-6	Water-soluble	8
Phosphoric acid	$\text{H}_3\text{PO}_4$	7664-38-2	Water-soluble	20
Water	$\text{H}_2\text{O}$	7732-18-5	Used as a solvent	

## Methods

### *Microstructure characterization of FRCs/P. densiflora*

The microstructures of the FRTW-pressure specimen and untreated specimen after combustion testing were determined by SEM (S-3200-N, Hitachi, Tokyo, Japan) at room temperature. The specimens were observed under the acceleration voltage condition of 20 kV to collect the SEM images. In addition, FT-IR (Thermo Scientific, Nicolet 380, Madison, WI, USA) was used to monitor changes in the chemical groups inside the wood

pores caused by the applied impregnation of the FRCs. The specimens were analyzed over the range of  $650\text{ cm}^{-1}$  to  $4000\text{ cm}^{-1}$  with a spectrum resolution of  $4\text{ cm}^{-1}$ . The analyses were conducted to measure the char layer that was formed by foaming on the surface of the material when the specimen was burned. The test specimens were ground to a size of 100 mesh. And then those specimens were mixed with KBr powder according to tablet method.

#### *Thermal characteristics of FRCs/P. densiflora*

To confirm the improved thermal stability of the FRC-treated specimen compared to the untreated specimen, TG analysis (TA Instruments Inc., New Castle, DE, USA) was performed to determine the thermal characteristics of the wooden specimens. The analysis was carried out from room temperature to  $800\text{ }^{\circ}\text{C}$ , a heating rate of  $10\text{ }^{\circ}\text{C}/\text{min}$ , and under a nitrogen atmosphere at the flow rate of  $100\text{ mL}/\text{min}$ .

#### *Combustion gas toxicity assessment*

To evaluate the toxic components of the combustion gases, the flue gas was evaluated for toxicity index according to NES 713 (1990). The NES 713 provided the concentrations of 13 different toxic gases, which are expressed as a factor of the density that is fatal to humans after 30 min of exposure time (NES 713 1990; Liang and Ho 2007). In addition, this can be quantitatively analyzed for the toxic components of the combustion gas using the test method specified in the ISO technical report (ISO/TR 16312-2). The Toxicity Test Apparatus (FESTEC Co., Seoul, Korea) was used to test the toxicity of the gases in the FRC-treated wood and the untreated wood. Figure 2 shows the test equipment and the test process.

The ignition fuel gas in the chamber was adjusted to a methane flow rate of  $2\text{ L}/\text{min}$  and an air flow rate of  $15\text{ L}/\text{min}$  with a burning duration that allowed a flame to be sustained. The specimens were exposed to a flame that was continuously burning. After the specimens were completely burned, a mixing fan was used to mix the gases in the chamber. Then, the mixed gases were detected using a gas detector tube (Ham *et al.* 2002; Kannan *et al.* 2005).

This enabled the identification of the amount of combustion gases produced by burning the specimens. The amounts of toxic gases ( $C_{\theta}$ ) in the combustion products were detected and estimated based on the changes in the various colors of the tube caused by the reacting gases. The formula given below was used to mathematically determine the concentration of the gas produced when  $100\text{ g}$  of the material was burned, and the combustion products were in a chamber with a volume of  $1\text{ m}^3$  (NES 713 1990).

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$$C_{\theta} = (C_i \times 100 \times V) / m \quad (1)$$

where  $C_{\theta}$  is the final concentration of gas (ppm) in a  $1 \text{ m}^3$  volume chamber,  $C_i$  is the correct concentration of gas in the test chamber (ppm),  $m$  is the mass of the specimen (g), and  $V$  is the volume of test chamber ( $\text{m}^3$ ).

(a) Test equipment



(b) Experimental process

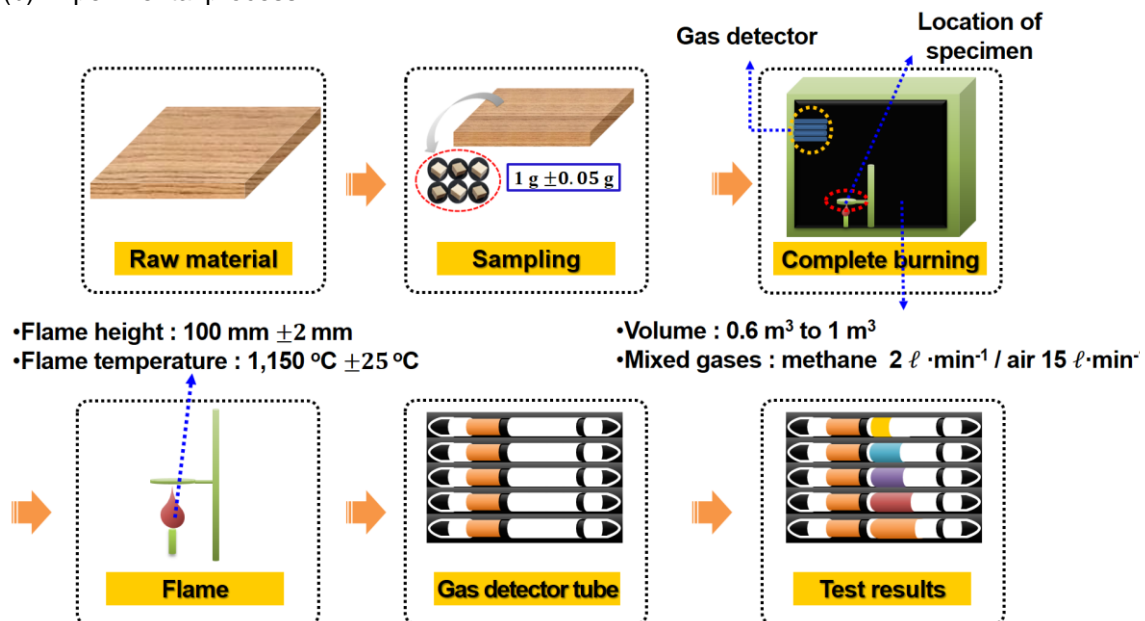


Fig. 2. Experimental equipment and procedure according to NES 713 (1990)

Equation 1 was used to calculate  $C_{\theta}$  for each of the gases to determine the precision of replication; it is then possible to determine the averages values of the respective gases,

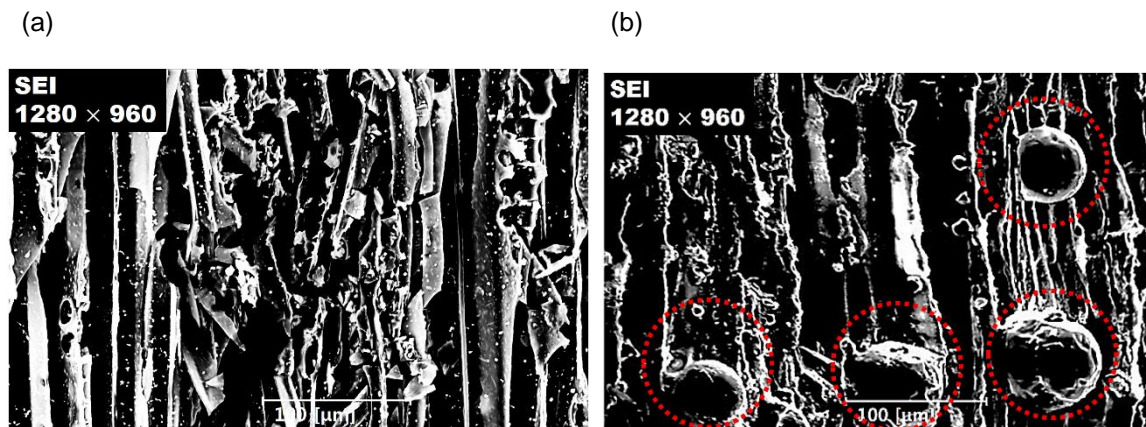
$$\text{Toxicity Index} = C_{\theta 1} / (C_{f 1}) + C_{\theta 2} / (C_{f 2}) + \dots + C_{\theta n} / (C_{f n}) \quad (2)$$

where 1, 2, ...,  $n$  represents each of the gases detected and  $C_f$  is the concentration of the detected gasses that is considered fatal to humans after 30 min of exposure (ppm).

## RESULTS AND DISCUSSION

### Characteristics of Microstructure of the FRCs-treated *Pinus densiflora* S.

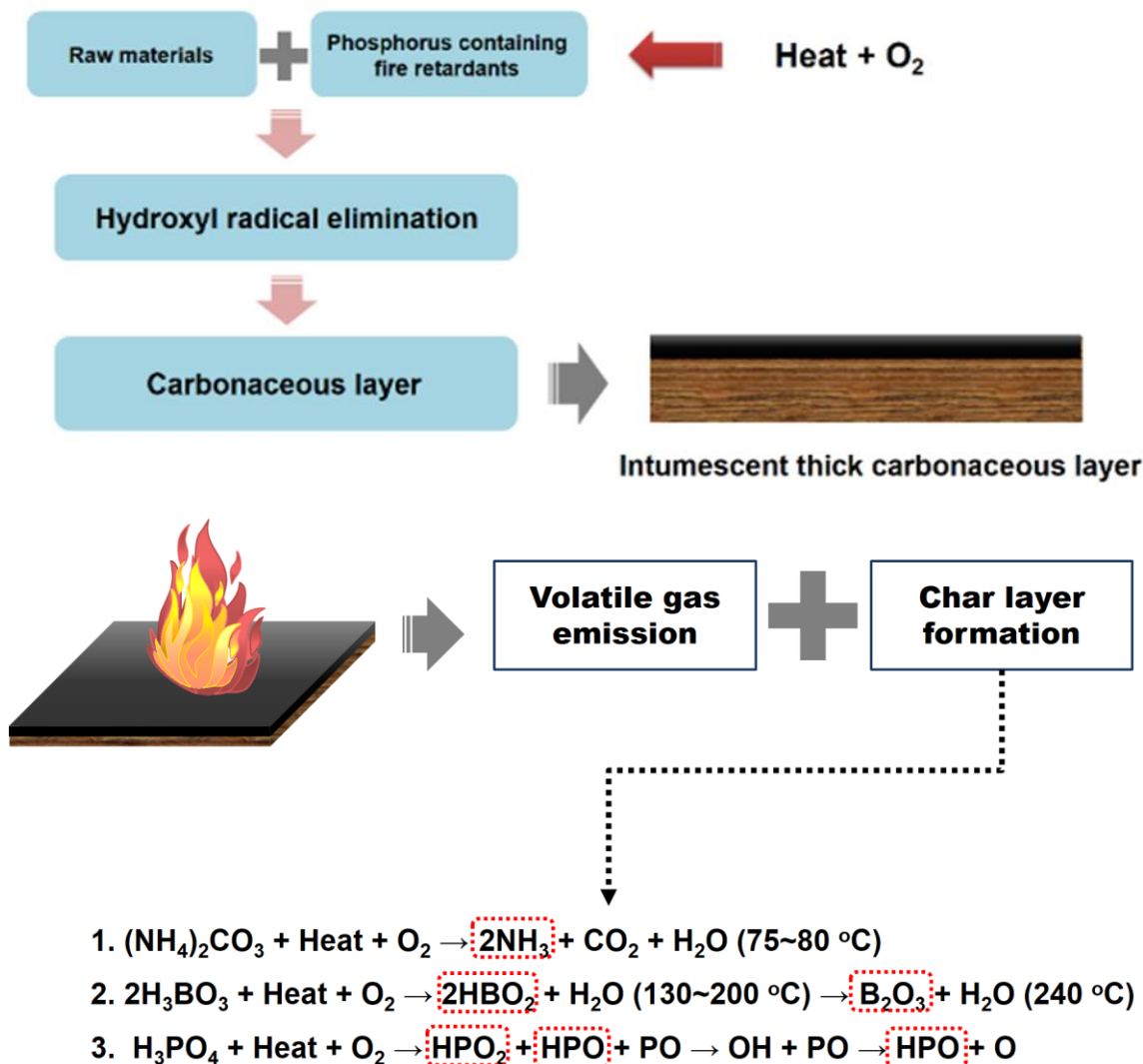
The SEM was used to evaluate the FRC-treated *Pinus densiflora* after combustion testing. Figure 3 shows the SEM images of the FRTW-pressure specimens after the combustion test. As shown, the internal structure of the carbonized wood collapsed. However, foam formed in the internal structure of the FRTW-specimen. Formation of the foam due to the FRCs was marked with a red circle in the SEM image. It swelled up as it burned inside the FRTW-pressure specimen.



**Fig. 3.** SEM image of radical section in wooden specimens; (a) Untreated wood, (b) FRTW-pressure

The formation of foam was attributed to the combustion of phosphorus chemicals. As heat is applied to phosphorus chemicals, water vapor, metaphosphoric acid ( $\text{HPO}_3$ ), monobasic phosphate ( $\text{HPO}$ ), oxidized phosphate ( $\text{PO}$ ), and other gases are released, and then char layers are formed on the surface of the material. The foam is caused by the gas that is released as the char layer forms. According to Chung and Jin (2010), when a fire-retardant such as FRCs is impregnated into the pit openings of wood fibers, the fire-retardant swells when burned, thereby forming a char in the interior and on the surface of the wood (Chung and Jin 2010). The SEM images show that the FRCs achieved their morphology during combustion when they became impregnated inside the wood. This result also indicated that FRCs were fixed inside the wood, which positively affected the formation of the char layer (Wu *et al.* 2012). Figure 4 shows the reaction mechanism of the phosphorus chemicals.

The FT-IR absorption spectra of the untreated specimen and the FRTW-pressure specimen are shown in Fig. 5. The pine had the molecular formulae  $\text{C}_6\text{H}_{10}\text{O}_5$  (cellulose) and  $\text{CH}=\text{CHCH}_2\text{O}$  (lignin; Korean pine) (Kim *et al.* 2012). In a comparison of the spectra of the two specimens, the absorbance of the FRC-treated specimen was confirmed to be lower overall. These changes resulted from the degree of infrared absorption weakening the materials (Doyle 1992). It has been reported that the type of fire retardant-treated materials had a high correlation with the elements of internal bonding force (Anderson *et al.* 1991a,b; Moore and Owen 2001).



**Fig. 4.** Reaction mechanism (Chung and Jin 2010) and char formation of phosphorus chemicals

Anderson *et al.* (1991a), Choi *et al.* (1987), and Wu *et al.* (2012) reported that the cellulose was in the range of 1250  $\text{cm}^{-1}$  to 1000  $\text{cm}^{-1}$ , lignin was in the range of 1350  $\text{cm}^{-1}$  to 1200  $\text{cm}^{-1}$ , and 1600  $\text{cm}^{-1}$  to 1400  $\text{cm}^{-1}$ , respectively, and they had characteristic spectra. In addition, it was reported by Wu *et al.* (2012) that the spectrum showed strongly near 3400  $\text{cm}^{-1}$ . Based on the results of previous studies, the spectrums in this study were analyzed. The specimen surfaces are mainly composed of cellulose, hemicellulose, and lignin by made up of hydroxyl group (OH), carbon-hydrogen bond (C-H), and Carbonyl group (C=O and C-O) functional groups. The spectrum peak in the range of 3500  $\text{cm}^{-1}$  to 3000  $\text{cm}^{-1}$  indicates the presence of hydroxyl groups inside the materials (Colthup *et al.* 1964; Choi *et al.* 1987; Pandey and Pitman 2003). Also, it is attributed to the presence of flame-retardant agents, according to reports by Pabeliña *et al.* (2012). The region of 3500  $\text{cm}^{-1}$  to 3300  $\text{cm}^{-1}$  was assigned to the hydroxyl group. In particular, the spectrum in this range is the hydroxyl group originating from the moisture inside the material. Also, the spectrum in the region of 3400  $\text{cm}^{-1}$  to 3000  $\text{cm}^{-1}$  range indicates  $\text{NH}^+$  absorption in di-

ammonium phosphate. In comparing the peaks of natural wood with FRTW-pressure specimen, no differences were found. According to Wen, such findings can be expected when the amount of added FRCs is small or the fixation is not good inside (Wen 2015).

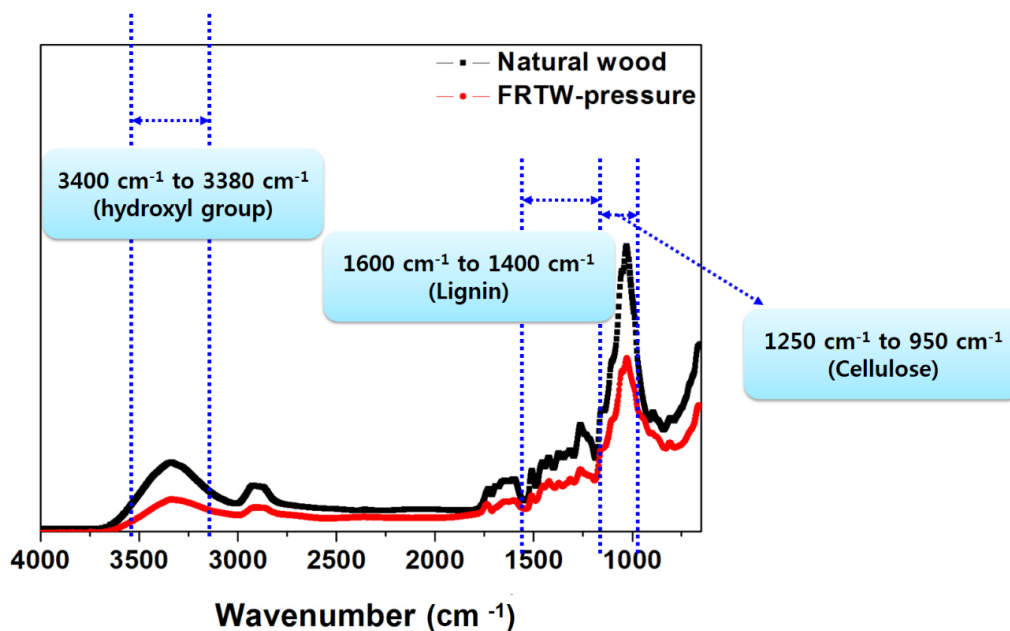


Fig. 5. FT-IR spectra of natural wood and FRCs treated wood

The spectra of the FRC-treated specimen before and after the combustion test are shown in Fig. 6. The reason for carrying out this comparison was to find evidence as to whether impregnation of the wood had been achieved, leading to proper functioning of the fire retardant. Thermal decomposition of the wood was in progress as the temperature increased. It occurred in hemicellulose from 180 °C to 350 °C, in cellulose from 275 °C to 350 °C, and in lignin from 250 °C to 500 °C (Kim and Jeune 2009; Lowden and Hull 2013). Among the components of wood, the lignin of char is produced as a thermal decomposition product. The biggest difference in the spectrum of the specimen after combustion was the decreased spectral absorbance in the range before and after 3400 cm<sup>-1</sup>; the range of this spectrum meant that moisture was reduced. The change in absorbance means that the OH group was initially present and it disappeared. In a previous study it was reported that the absorbance disappeared at a temperature of 600 °C or more (Inoue *et al.* 2002). Furthermore, the spectrum around 1590 cm<sup>-1</sup> was clearly formed, and it was determined that the aromatic ring of lignin was destroyed to form an aromatic C-H bond in agreement with the results of Lee and Lee (2008). The specificity in the spectrum of the FRTW-pressure specimen after combustion shows that the peak at 1000 cm<sup>-1</sup> to 500 cm<sup>-1</sup> increased very strongly. These results were related to the formation of a C-X bond (1400 cm<sup>-1</sup> to 600 cm<sup>-1</sup>) due to the formation of char because of the carbonization of the test specimen. In addition, the peaks at 1500 cm<sup>-1</sup> to 1100 cm<sup>-1</sup> and 1100 cm<sup>-1</sup> to 850 cm<sup>-1</sup> were allocated to stretching vibration of P-O-P, and especially, peaks at 1350 cm<sup>-1</sup> to 1100 cm<sup>-1</sup> were allocated to stretching vibration of P=O (Wen 2015). According to the sentence described above, it was concluded that the FTIR spectra were not observed because the amount of added FRCs was small or not fixed inside the wood. However, when the results were confirmed after combustion test, it was judged that it was able to prevent fire.



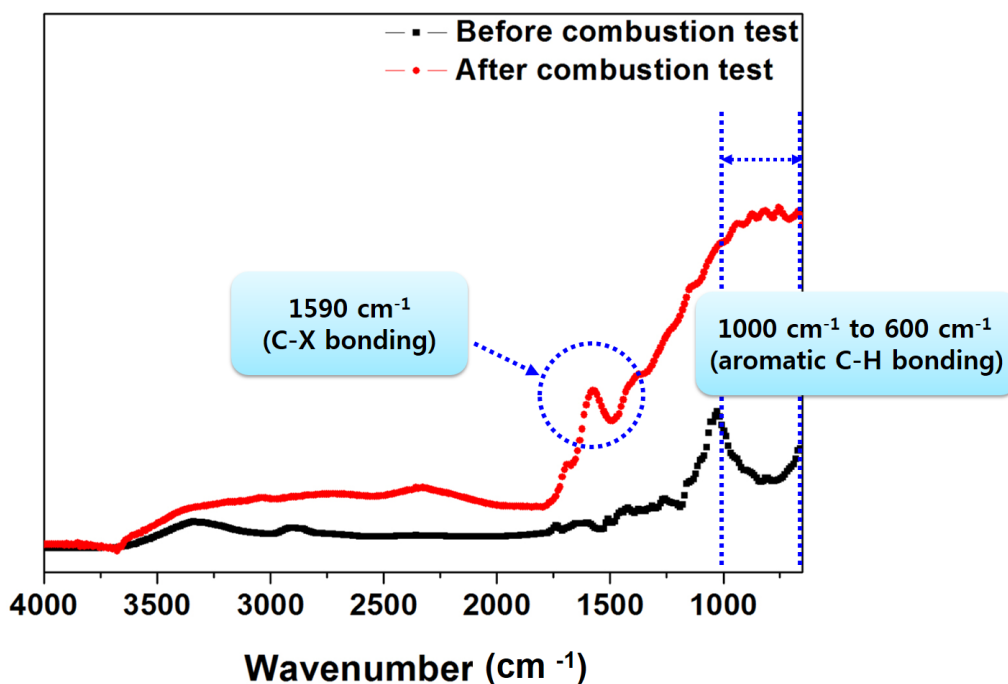


Fig. 6. FT-IR spectra of FRTW-pressure specimen before and after combustion

### Thermal Properties

The TG analysis was used to measure the thermal properties of the specimens, as well as to determine mass changes in the materials based on the time-to-temperature changes. The specimens used in the TG analysis are generally ground to a size of less than 120-mesh, or cut to a size of 2 mm to 4 mm. The specimens used in this experiment were cut to a size of 2 mm or less and again ground to a size of 120-mesh or less. Figure 7 shows the TG analysis results for the thermal degradation behaviors of all the wood specimens. The mass loss rate and the values obtained by differentiating the values are shown in the graph (Fig. 5). As temperature increased, pyrolysis occurred in all of the wood specimens. First, surface burning began when the temperatures ranged between 30 °C and 100 °C. According to the results of the TG analysis, the temperature ranges at which surface combustion of the material began when the temperatures ranged between 30 °C and 100 °C. For the untreated specimen, the peak differential value occurred because ignition and carbonization took place simultaneously on the surface of the wood.

When temperatures ranged from 250 °C to 400 °C, decomposition combustion progressed and pyrolysis progressed after the char layer cracked and desorption occurred. The untreated specimen showed slight pyrolysis even at temperatures below 200 °C, but pyrolysis proceeded rapidly between 350 °C and 380 °C. TG analysis is a method of thermal stability analysis in which the weight of a specimen is measured over time as the temperature changes. It provides information about chemical phenomena including thermal decomposition and solid-gas reactions (*e.g.*, oxidation or reduction). In this study, the thermal decomposition characteristics of the material were measured. As a result of analysis, the weight residual amount of the FRTW-pressure specimen was three times higher than that of the untreated specimen.

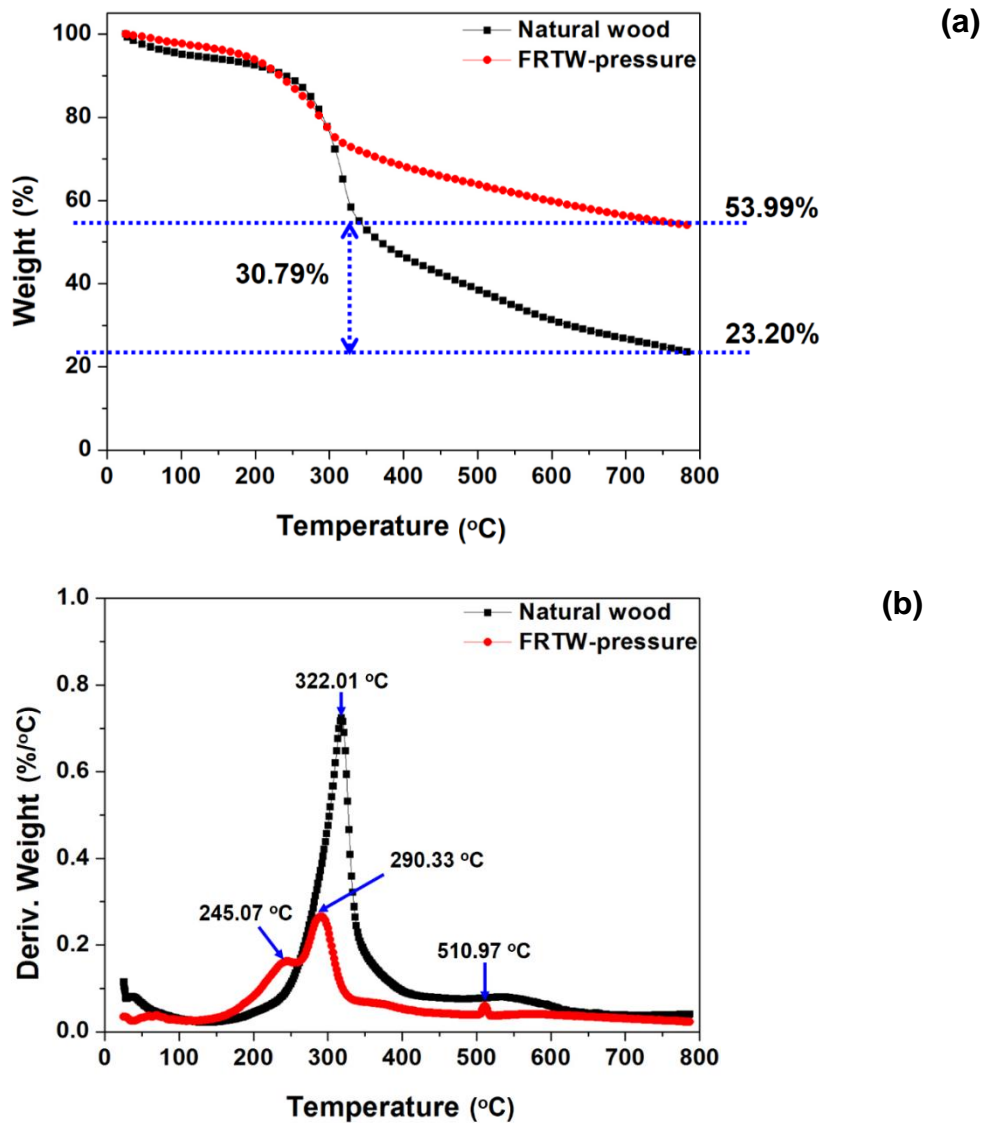


Fig. 7. TG-DTG curves as a function of time for all wood specimens; (a) TG curves, and (b) DTG curves

### Toxicity Index of the Combustion Gases

Table 2 shows the lethal concentrations of the harmful components of the combustion gases and the test results for all the wood specimens based on the NES 713 (1990) standard. The NES 713 (1990) standard specifies that among the combustion gases generated by a fire, carbon dioxide (CO<sub>2</sub>), carbon monoxide (CO), hydrogen sulfide (H<sub>2</sub>S), ammonia (NH<sub>3</sub>), formaldehyde (CH<sub>2</sub>O), hydrogen chloride (HCl), acrylonitrile (CH<sub>3</sub>CN), sulfur dioxide (SO<sub>2</sub>), nitrogen oxide (NO<sub>x</sub>), phenol (C<sub>6</sub>H<sub>6</sub>O), hydrogen cyanide (HCN), hydrogen bromide (HBr), and 13 different gas components and emission amounts, such as hydrogen (H), can be confirmed (Ham *et al.* 2002; Kannan *et al.* 2005; Liang *et al.* 2007; Kim 2016). The differences in the types of gases emitted from all three of wood specimens were not statistically significant. The types of gases released from the untreated specimens were: CO<sub>2</sub>, CO, CH<sub>2</sub>CHCN, and NO<sub>x</sub>.

**Table 2.** The Toxicity Concentration Fatal to Humans Based on the NES 713 (1990) Standard

Gases	Chemical Formula	Cr (Critical Limit, ppm)	Untreated Specimen	FRC-pressure-treated Specimen
Carbon dioxide	CO <sub>2</sub>	100,000	87,919	15,038
Carbon monoxide	CO	4,000	488	251
Hydrogen sulfide	H <sub>2</sub> S	750	0	0
Ammonia	NH <sub>3</sub>	750	0	0
Formaldehyde	HCHO	500	0	0
Hydrogen chloride	HCl	500	0	0
Sulphur dioxide	SO <sub>2</sub>	400	0	0
Acrylonitrile	CH <sub>2</sub> CHCN	400	15	25
Nitrogen oxide	NO <sub>x</sub>	250	49	301
Phenol	C <sub>6</sub> H <sub>5</sub> OH	250	0	0
Hydrogen cyanide	HCN	150	0	0
Hydrogen bromide	HBr	150	0	0
Hydrogen fluoride	HF	100	0	0

However, their emissions were somewhat different. First, the amount of CO<sub>2</sub> emitted from the untreated specimen was 88,000 ppm, which was higher than the amount emitted by the other specimens. This meant that there was a high probability of complete burning of the natural untreated wood, but it did not surpass the standard. In contrast, the CO emissions from all three of the specimens were lower than the CO<sub>2</sub> emissions. However, compared to the total amount of released gases, the CO emissions were higher in the FRTW-pressure specimens than in the untreated specimen. The results for the FRTW-pressure specimens were determined to be due to the combustion inhibiting effect.

In the case of conventional fire retardants, bromine and fluorine are detected in the post-combustion gases. However, these were not detected in the fire-retardant chemicals used in this study. It was also confirmed that no harmful components, such as NH<sub>3</sub> and CH<sub>2</sub>O, were present. In particular, in the case of NH<sub>3</sub>, it was the theoretical gaseous substance released during the combustion of ammonium diphosphate, but this was not detected in this experiment. This is because it is not captured in the gas detection tube when the emission of gas is less than 50 ppm. However, NO<sub>x</sub> was released from both of the FRC-treated specimens. The NO<sub>x</sub> was mainly emitted from the combustion process, and NO and NO<sub>2</sub> have harmful effects on the human body. To suppress the generation of NO<sub>x</sub>, some methods can be used to reduce the amount of excess air or to lower the temperature at the time of combustion (Terrill *et al.* 1987). However, these methods are difficult to use as a practical alternative when a fire actually occurs. The best way to reduce the concentration of harmful gases emitted in a fire is to ensure that the emission of harmful gases is low when the materials are burning. Pyrolysis of di-ammonium phosphate in FRCs is thought to be the cause of the release of NO<sub>x</sub>. A small amount of NO<sub>x</sub> was also released from the untreated specimen. These results were similar to the results reported by Wilkins and Murray (1980), who found that nitrogen compounds, such as gaseous nitric acid, are released by pyrolysis of the extracts in wood when the wood is burned.

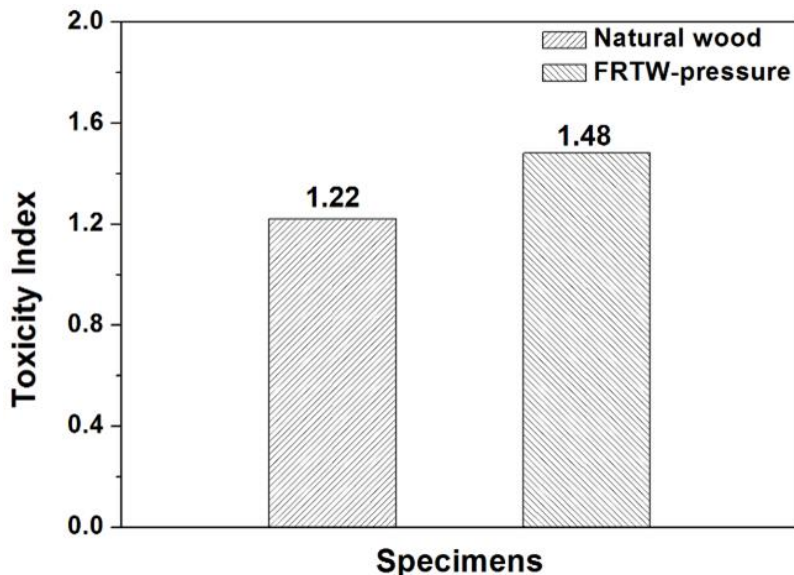


Fig. 8. Average value of the toxicity index for all the wood specimens

The toxicity index of the flue gas for all specimens (Fig. 8) was calculated by applying the gas detection value shown in Table 2 to Eq. 2. The toxicity index of the untreated specimen was 1.22. The toxicity index of the FRTW-pressure specimen was 1.48. The limit value of the toxicity index specified by the NES 713 standard is 5, and it is determined that the combustion gas of the material is safe when the value of toxicity index is less than 5.

However, according to previous studies, when the toxicity index limit is applied to housing, if the toxicity index is greater than 1.5, the risk of injury or disability from a fire is higher in humans that are exposed to that environment for more than 30 min (Chauhanm *et al.* 2012; Seo and Son 2015). Overall, the toxicity index results demonstrated that the FRCs developed in this study had a low risk for indoor use. Based on these findings, the FRTW-pressure specimen also seemed to improve the fire safety of these building materials. However, further research is needed to reduce the amount of NO<sub>x</sub> emissions in the FRTW-pressure specimen. A further goal should be to decrease the toxicity index.

## CONCLUSIONS

1. The FRCs were found to work based on the SEM images of the specimens after the combustion test. Foams, which can form a char on the material surface, were formed after heat was applied during combustion. The foam was well adhered to the inside of the wood pits even after burning. This form is due to the formation of HPO and PO, which were formed by the pyrolysis of the phosphate species were belonging the FRCs. They can play a role in improving the flame retardant performance.
2. Based on the FT-IR spectrums for FRTW-pressure specimen of before and after combustion test, it has been confirmed that there was a decreased content of hydroxyl groups resulting from the evaporation of water due to combustion and an increase of NH<sup>+</sup> due to pyrolysis of phosphate.

3. The TG analysis confirmed that the FRCs used in this study were responsible for delaying the pyrolysis of the wood and for forming the char, thereby reducing the mass loss rate of wood by more than 30%.
4. The FRCs produced very little noxious gas after combustion. The NO<sub>x</sub> was among the noxious gas components detected, but analysis of the overall toxicity index demonstrated that this was not problematic. Nevertheless, further studies are needed to determine the composition and composition ratios of FRCs.

## ACKNOWLEDGMENTS

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