Article

Evaluation of Toxic Gas Generation, Smoke Generation, and Flammability in Flame-Retardant Plywood Combustion Tests

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Abstract: The objective of this study was to evaluate toxic gas generation, smoke generation, and flammability during the combustion of flame-retardant plywood manufactured with a flame-retardant resin impregnated in it according to International Maritime Organization (IMO) standards. Wood and wood-based materials have been restricted in their application as structural building materials or finishing materials, because they are combustible materials. It has been reported that an enormous amount of the damage to humans in the event of a fire is due to the inhalation of toxic gases generated during combustion, rather than due to direct contact with flames. In recent years, fire-safe flame-retardant wood has been developed through various studies. The results of this study are expected to assist in ensuring that flame-retardant wood and wood materials are safer and freer from fire and toxic gases and are recognized as building materials that can minimize damage to humans while providing a safe residential environment.

As shown in the results of this study, seven out of the eight kinds of the toxic gases generated during the combustion of flame-retardant plywood as identified by the IMO, other than CO, were not detected. Even CO gas was detected in quantities smaller than the thresholds under three test conditions. Therefore, unlike synthetic resin products, flame-retardant plywood was determined to be a finishing material that could reduce the damage due to toxic gases in the event of a fire.

In smoke generation tests, the mass reduction rate showed a tendency to increase as the test conditions became more severe, with values of 13%, 18%, and 20%. In addition, as the test conditions became more severe, the average of the maximum specific optical density showed values of 75.70, 81.00, and 191.20. However, all of the values satisfied the standard given by the IMO, which is below 200, indicating that flame-retardant plywood is applicable as a finishing material.

Flammability was evaluated and the results showed the values of critical flux at extinguishment (CFE) as 49.5kW/㎡ on average, total heat release (Qt) as 0.21MJ on average, and peak heat release rate (Qp) as 0.66kW on average, which satisfied all of the thresholds presented by the IMO. Therefore, flame-retardant plywood, of which the flame-retardant performance was evaluated, was determined to be an applicable finishing material that satisfies fire safety standards.

Keywords: flame-retardant plywood; flammability; toxic gas generation; smoke generation

1. Introduction

Wood and wood-based materials have been widely used as structural and finishing materials for construction, but due to their material properties (constituents), they are vulnerable to combustion when exposed to fire [1].

There were 40,103 cases of fires in South Korea in 2019, and it was reported that there were 2,515 casualties and property damage of 858.4 billion won. The number of large-scale fires has also been increasing every year, and among the places where fires occurred, residential facilities were the most frequent with 11,058 cases (27.6%), followed by industrial facilities with 5,429 cases (13.5%). Among the initial igniting materials, paper, wood, and hay were the most frequent with 9,484 cases (23.6%), followed by electricity and electronics (20.5%), garbage (11.3%), synthetic resins (11.2%), and food (7.9%). In addition, it was reported that about 75.8% of deaths (216 people) and about 79.7% of injuries (1,777 people) occurred in residential and non-residential facilities [2]. The increase in the occurrence of fires and damage from fires is due to the increase in energy consumption and combustible interior materials following economic growth and it is expected to intensify further in the future [3]. Following technological and industrial development, various finishing materials have been developed and applied to buildings, and these are known to emit large amounts of toxic gases while burning. The main cause of harm to humans in the event of a fire is the phenomenon of asphyxiation from the inhalation of toxic gases rather than harm due to direct contact with the flames, and the harm caused by this asphyxiation is known to be very serious [4]. The risk is also increasing as gradually increasing quantities of combustible synthetic polymer materials are being used. Various studies have been conducted on the types and characteristics of toxic gases generated in fires and the effects of these gases on the human body [3, 5, 6, 7]. The International Maritime Organization (IMO) has determined eight kinds of toxic gases from fire that can cause fatal damage to the human body, which are CO (carbon monoxide), HBr (hydrogen bromide), HCL (hydrochloric acid), HCN (hydrogen cyanide), HF (hydrogen fluoride), NO (nitric oxide), NO2 (nitrogen dioxide), and SO2 (sodium sulfite), and has set thresholds for these toxic gases to strictly manage them [8]. In addition, the IMO has presented methods for the evaluation of the flammability of interior finishing materials and standards for safer finishing materials, and enacted regulations so that only products that have passed the standards are used for construction [9].

In addition, the following regulations were stipulated for the interior finishes and trims of buildings in the United States: "interior finish materials that give off smoke or gases more dense or more toxic than that given off by untreated wood or untreated paper under comparable exposure to heat or flame shall not be permitted" [10].

Wood is a combustible material that has been used as an important energy source, and wood and wood products have also been used as major components of buildings [7]; however wood emits a variety of compounds when combusted [11]. In an evaluation of the combustion behavior of building flooring and resultant gas toxicity, Lee. et al. (2008) [12] reported that emissions of CO and CO2 from wood-based MDF flooring were less than those from polyester flooring and PVC flooring. In addition, the gas hazards from flame-retardant wood and the toxicity index of combustion gases were evaluated. Based on the results, it was reported that the gas toxicity index of untreated wood was 0.183 and that of flame-retardant wood was 0.196 to 0.251, which were lower than that of PVC at 4.13 and urethane flooring at 7.2 [13].

Wood and wood-based materials are sometimes restricted in use because they are vulnerable to fire, and it is determined that consideration of fatal toxic gas generation upon combustion, which is a more serious problem, and the resultant harm to humans were insufficient. Various studies to lessen the vulnerability of wood and wood-based materials to combustion have been conducted worldwide [1, 14, 15, 16] and currently, flame-retardant wood that does not burn has also been developed and produced. Therefore, it was determined that studies on the types and amounts of the toxic gases generated when flame-retardant wood is combusted should be conducted, so that the evaluation and perception of wood and wood-based materials that are safer and can reduce harm to humans can be changed.

This study was conducted to evaluate the flammability, toxic gas generation, and smoke generation of flame-retardant plywood manufactured and applied as finishing materials for construction in South Korea with a combustion test. The results of this study are expected to contribute to the development of wood-based finishing materials with higher levels of safety and reduced harm to humans upon combustion.

 doi:10.20944/preprints202209.0003.v1

2. Materials and Methods

2.1. Testing materials

2.1.1. Flame retardant resin

The flame-retardant resin used in the manufacturing of flame-retardant plywood was manufactured by SAMHWA PAINTS Industrial Co., Ltd. It is a water-soluble, flame-retardant resin (NF200+) manufactured in Korea. The main components of the flame-retardant resin are ammonium phosphate polymer (APP), guanyl urea phosphate (GUP), phosphonic acid, acrylamide acrylic acid-N-{3-(dimethylamino)propyl} methacrylamide copolymer, 2-benzisothia zo lin-3-one, and a minor amount of additives.

2.1.2. Flame-retardant plywood

The flame-retardant plywood used for the evaluation of toxic gas and smoke generation, and combustion characteristics was prepared by vacuum pressure impregnation with flame retardant resin (NF200+) at 17kgf/㎠ pressure for 20min. The impregnation amount of the flame retardant was more than 300kg/㎥, and a type of flame-retardant plywood that passed the flame retardant performance according to the KSF ISO 5660-1 standard was used [17]. The specifications of the test specimen are shown in Table 1.

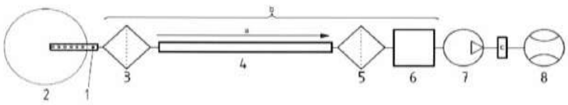
Table 1. Specifications of test specimen (IMO. 2010a, b).

Construction	Uses	Dimensions (mm)	Number of specimens	Flame-retardant resin
Plywood	Bulkheads, Linings, Ceilings	W75 x L75 x T24 (Toxic gas and smoke generation)	9	NF200$^+$ (SAMHWA PAINTS Industrial Co., Ltd.)
		W155 x L800 x T24 (Flammability)	3	

2.2. Testing methods

2.2.1. Test equipment

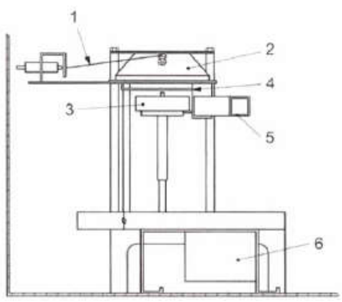
Toxic gas generation was measured by the Korea Marine Equipment Research Institute according to the test regulations of IMO Res. MSC. 307(88): 2010/ANNEX 1/Part 2[8]. Figure 1 shows a block diagram of the test equipment applied to the measurement of toxic gas generation and Fig. 2 shows a block diagram of the test equipment applied to the measurement of smoke generation.



Key: 1. Probe, 2. Exhaust duct (fire model),
3. Filter 1, 4. Transfer line, 5. Filter 2,
6. FTIR gas cell, 7. Pump, 8. Flow meter

Figure 1. Test equipment arrangement for toxic gas generation.

Key: 1. Thermocouple



2. Radiator cone
3. Specimen holder
4. Radiator shield
5. Heat flux meter holder
6. Spark ignition housing

Figure 2. Test equipment arrangement for smoke generation.

2.2.2. Test methods

Toxic gas and smoke generation of flame-retardant plywood fires were measured according to the test regulations of IMO Res. MSC. 307(88):2010/ANNEX 1/Part 2[8]. Before measurement, the test specimens were conditioned at a temperature of 23±2 ℃ and relative humidity of 50±5% for 816 hr. The test methods and procedures for toxic gas and smoke generation upon combustion are shown in Table 2. In addition, the flammability of flame-retardant plywood was measured according to the test regulations of IMO Res. MSC. 307(88):2010/ANNEX 1/Part 5[9]. Before measurement, the test specimens were conditioned at a temperature of 23±2 ℃ and relative humidity of 50±5% for 72 hr.

Table 2. Test methods of toxic gas and smoke generation upon combustion**.**

Procedure	**Test methods**	
	Toxic gas generation	**Smoke generation**
1	Remove all dirty layers and particles in the test chamber, and clean the internal probe for FTIR sampling.	**Prepare the test chamber** Prepare the test chamber with a cone set at 25kw/m² or 50kw/m². The distance between the cone heater and the specimen shall be 50 mm and the pilot burner must be positioned 15 mm down from the bottom edge of the cone heater.
2	Maintain the filter, gas sampling line, and valves and gas cell at a temperature of 150 °C to 180 °C for at least 10 min. prior to test.	**Tests with pilot flame** For tests with a pilot flame, with the burner in its correct position, turn on the gas and air supplies and ignite the burner, and check the flow rates.
3	The wavelength resolution of the spectrometer must be 4cm⁻¹ or better. Set the Mid-IR whole spectral region for collection between 650cm⁻¹ and 4,500cm⁻¹.	**Preparation of photometric system** Set the zero and then open the shutter to set the full-scale 100% transmission reading. Recheck the 100% setting. Repeat the sequence of operations until accurate zero and 100% readings are obtained on the amplifier and recorder when the shutters are opened and closed.
4	Close the chamber door and introduce the air in the chamber into the gas cell of the FTIR. Wait for 1 min. and record the background spectrum	**Loading the specimen** Place the holder and specimen on the supporting framework below the radiator cone. Remove the radiation shield from below the cone and simultaneously start the data recording system and close the inlet vent. The test chamber door and the inlet vent must be closed immediately after the start of the test.
5	During the smoke density test, the sampling must start by turning the sampling valve to introduce the gas in the chamber into the sampling line at maximum smoke density sampling time.	**Recording of light transmission** Record the percentage of light transmission and time continuously from the start of the test.
6	-	**Termination of test** The initial test in each of the test conditions must last for 20 min to verify the possible existence of a second minimum transmittance value.
7	-	**Conditioning of specimens** Before preparing the specimens for testing, they must be conditioned to a constant mass at 23±2 °C and 50±2% relative humidity.

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3. Results and Discussion

3.1. Toxic gas generation

In the event of a building fire, a wide variety of toxic gases are generated in the form of a single gas or a mixed gas depending on the materials being combusted [4, 6]. The International Maritime Organization (IMO) determined eight kinds of toxic gases that may be generated when finishing materials for bulkheads, linings, or ceilings are combusted, and applied the minimum generation criteria for each kind of toxic gas. Table 3 shows the eight kinds of toxic gases, their minimum criteria, and the characteristics of the effects of each toxic gas on the human body.

Table 3. Kinds and characteristics of toxic gases generated upon combustion and the associated minimum criteria**.**

Toxic gas	Characteristics	Criteria (ppm)
CO (carbon monoxide)	Suppresses oxygen transport, causing dizziness and headaches, etc.	≤1,450
HBr (hydrogen bromide)	Irritates the mucous membranes of the eyes, skin, and respiratory system, leading to death with the inflammation of the respiratory organs, and swelling and contraction of the larynx.	≤ 600
HCl (hydrogen chloride)	This is very toxic, irritates the airways and eyes, and when inhaled at a high concentration, leads to death due to the occurrence of lung edema.	≤ 600
HCN (hydrogen cyanide)	Poisoning with this leads to death due to dyspnea along with pain as if the chest is tightened.	≤ 140
HF (hydrogen fluoride)	With a pungent smell and potent toxicity, this penetrates through the skin, leading to severe pain, blindness, and even death.	≤ 600
SO_2 (sulfur dioxide)	Strongly irritates the eyes and respiratory system and leads to death when inhaled in large quantities.	≤ 120
NO (nitric oxide)	Toxic gas that is very harmful to the human body.	≤ 350
NO_2 (nitrogen dioxide)	Invades the respiratory tract or lungs and leads to death of more than half of animals at concentrations exceeding 100 ppm.	≤ 350

[3, 6, 13].

In this study, toxic gas and smoke generation from flame-retardant plywood upon combustion were measured according to the fire safety standards for interior finishing materials (bulkhead, lining, and ceiling materials) presented by the IMO. Table 4 shows the appearance of the specimens before and after the test under each of the test conditions.

The amount of toxic gases that were generated by each kind of toxic gas under three conditions (irradiance of 25 kw/㎡ in the absence of a pilot flame, irradiance of 25 kw/㎡ in the presence of a pilot flame, and irradiance of 50 kw/㎡ in the absence of a pilot flame) are shown in Table 4. The results of the evaluation of toxic gas generation of flame-retardant plywood that were tested in this study are shown in Table 5. Eight kinds of representative toxic gases generated by fire were evaluated and, according to the results, only CO (carbon monoxide) was detected, and the remaining toxic gases were not detected. The amounts of carbon monoxide (CO) gas generated that were detected in the three test conditions were 232 ppm, 293 ppm, and 1,444 ppm, respectively, which were smaller than the threshold of 1,450 ppm set by the IMO. Therefore, the flame-retardant plywood tested in this study was determined to be applicable as a finishing material for ships. In

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comparison to the study findings for polyurethane when it was combusted, 704 ppm of CO, 663.6 ppm of NO, 11 ppm of SO2, 63 ppm of HCl, and 70 ppm of HF were emitted, and 1,830 ppm of CO, 232.3 ppm of NO, 7.0 ppm of SO2, 282 ppm of HCl, and 70 ppm of HF were emitted when PVC was combusted [17]. Therefore, plywood made of wood can be said to be a safer material, because seven toxic gases, except for CO gas, were not detected, and even the amount of CO generated was shown to be below the threshold.

Table 4. Tested materials in each test condition.

Division		Before the test	After the test
Toxic gas and smoke generation (IMO, Part 2)	Irradiance of 25 kw/㎡ in the absence of pilot flame		
	Irradiance of 25 kw/㎡ in the presence of pilot flame		
	Irradiance of 50 kw/㎡ in the absence of pilot flame		
Flammability (IMO, Part 5)	Heat flux : ·50.5kW/㎡ (at the 50 mm position) ·23.9kW/㎡ (at the 350 mm position)		

As is well known, the main components of wood are carbon (C, 50%), oxygen (O, 44%), and hydrogen (H, 6%). Consequently, when wood is combusted, only carbon monoxide (CO) and carbon dioxide (CO2) are generated and, basically, the other toxic gases are not generated. In addition, the flame-retardant resin used in the manufacturing of the flame-retardant plywood in this study was water-soluble, and its main components were ammonium phosphate polymer (APP), guanyl urea phosphate (GUP), phosphonic acid, acrylamide acrylic acid-N-{3-(dimethylamino) propyl} methacrylamide copolymer, 2-benzisothiazolin-3-one, and a small amount of additives [16]. In the combustion test of the flame-retardant plywood in which such water-soluble, flame-retardant resins were vacuum pressure impregnated, no toxic gases other than CO gas among the eight kinds of toxic gases presented by the IMO were detected, and it was determined that CO gas was emitted in quantities below the threshold. Therefore, the emission of toxic gases due to the flame-flame-retardant resin treatment in addition to the gases generated when wood is combusted were determined to not be big enough to be problematic.

The results described above should be considered to evaluate the effects of building finishing material fires on the environment and human bodies when selecting these materials, and in the case of wood and wood products, those that emit the smallest possible amount of toxic materials should be selected, so that people can escape with minimal harm upon combustion of these materials [7]. The use of wood should not be restricted just because it is a combustible material. Since various types of flame-retardant wood and flame-retardant plywood, etc., are currently being developed and produced, it is expected that fire-retardant products can be applied to provide buildings and residential environments that are freer and safer from the toxic gases generated by fire and can minimize harm to humans.

Table 5. Toxic gas generation of flame-retardant treated plywood upon combustion.

Division	Irradiance of 25 kw/㎡ in the absence of a pilot flame	Irradiance of 25 kw/㎡ in the presence of a pilot flame	Irradiance of 50 kw/㎡ in the absence of a pilot flame	Criteria (ppm)
CO	232	293	1,444	1,450
HBr	0	0	0	600
HCL	0	0	0	600
HCN	0	0	0	140
HF	0	0	0	600
SO_2	0	0	0	120
NO	0	0	0	350
NO_2	0	0	0	350

Notes: 1) These criteria are for bulkheads, linings, or ceilings.
2) Each value is an average of three tests in each test condition.

3.2. Smoke generation

In this study, smoke generation during the combustion of flame-retardant plywood was measured according to the fire safety standards for interior finishing materials (bulkhead, lining, and ceiling materials) given by the IMO. Table 6 shows the average values for the three test pieces measured in each test condition.

The mass loss and maximum specific optical density (Dm) of the test specimens were evaluated under each test condition. As for the mass loss, the average mass of the test specimens before the test was 87.48 g and it was 72.30 g after the test, with showed a decrease by an average of 15.18 g under each test condition. Therefore, the average mass reduction rate was shown to be 17% (Fig. 3). As shown in Fig. 3, the mass reduction rate tended to increase with values of 13%, 18%, and 20% as the test conditions became more severe step by step.

Table 6. Smoke generation of flame-retardant treated plywood upon combustion.

Division	Irradiance of 25 kw/㎡ in the absence of pilot flame	Irradiance of 25 kw/㎡ in the presence of pilot flame	Irradiance of 50 kw/㎡ in the absence of pilot flame	Criteria
Initial mass (g)	85.92	86.70	89.82	Dm must not exceed 200 in any test condition
Final mass (g)	74.38	71.02	71.49	
Mass loss (g)	11.53	15.68	18.33	
Average of the maximum specific optical density (Dm)	75.70	81.00	191.20	

Notes: 1) These criteria are for bulkheads, linings, or ceilings.
2) Each value is an average of three tests in each test condition.

The average of the maximum specific optical density (Dm) showed different values under the three test conditions: 75.70 under the condition of irradiance of 25 kw/㎡ in the absence of a pilot flame, 81.00 under the condition of irradiance of 25 kw/㎡ in the presence of a pilot flame, and 191.20 under the condition of irradiance of 50 kw/㎡ in the absence of a pilot flame.

The IMO applied the standard that the average of the maximum specific optical density (Dm) should not exceed 300 under the three test conditions. The results of the measurements carried out in this study showed that the average of the maximum specific optical density (Dm) did not exceed 200, indicating that the flame-retardant treated plywood satisfied the IMO's smoke generation standard (Fig. 4).

When flame-retardant treated plywood was applied as a finishing material, the maximum specific optical density and toxic gas generation were determined to satisfy the finishing material standards presented by the IMO. Therefore, flame-retardant treated plywood is expected to contribute to the reduction of the enormous amount of harm to humans that occurs due to the generation of toxic gases and smoke upon combustion.

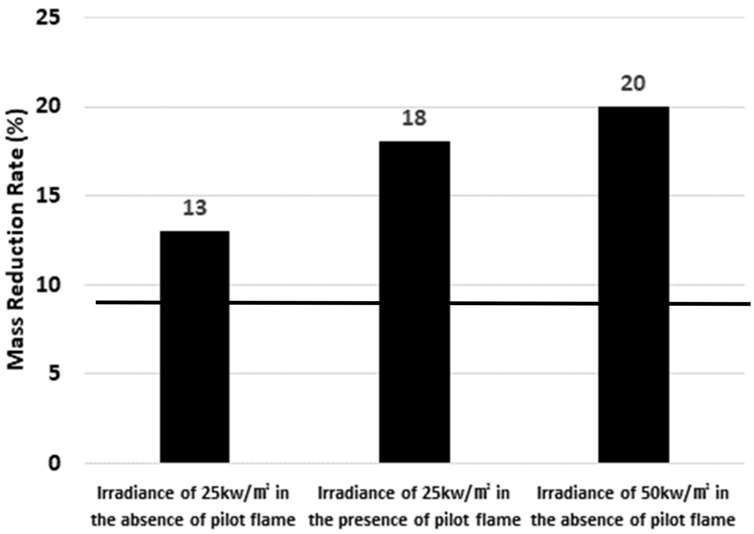

Figure 3. Mass reduction rate in each test condition.

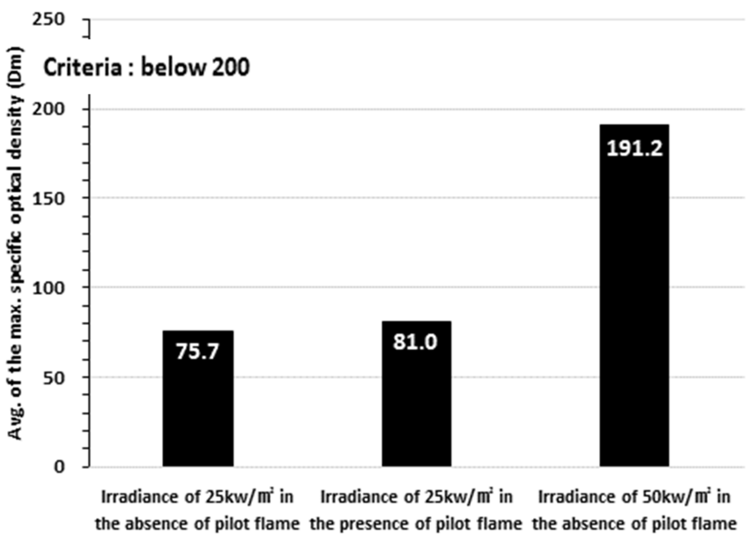

Figure 4. Average of the maximum specific optical density (Dm) in each test condition.

3.3. Flammability

The flame-retardant plywood flammability test results obtained in this study according to the standard under IMO Res. MSC. 307(88):2010/ANNEX 1/Part 5 [9] are shown in

Table 7. The test specimens were the flame-retardant plywood manufactured with vacuum pressure impregnation of the water-soluble flame-retardant resin mentioned in section 3.1. The test was intended to review the applicability of the foregoing material as a finishing material for ships.

Table 7. Flammability of flame-retardant plywood.

Specimen no. / items	1	2	3	Average	Surface flammability criteria1)
Average heat for sustained burning, **Qsb (MJ/㎡)**	-	-	-	-	≥ 1.50
Critical flux at extinguishment, **CFE (kW/㎡)**	49.5	49.5	49.5	49.5	≥ 20.0
Total heat release, **Qt (MJ)**	0.30	0.16	0.17	0.21	≤ 0.70
Peak heat release rate, **Qp (kW)**	0.87	0.53	0.58	0.66	≤ 4.00
Burning droplets	None	None	None	None	Not produced

Notes: 1) These criteria are for surface materials (bulkhead, wall, and ceiling linings).
「-」 Since there was no ignition, no values were recorded.

As shown in the table above with the results for the measurement of the flammability of flame-retardant plywood, the critical flux at extinguishment (CFE) was shown to be 49.5 kW/㎡ on average, which was more than twice the threshold of 20 kW/㎡ given by the IMO. The total heat release (Qt) was shown to be 0.21 MJ on average, which indicated excellent performance corresponding to about 30% of the upper threshold of 0.70 MJ presented by the IMO, and the peak heat release rate (Qp) was shown to be 0.66 kW on average, which was about 17% of the upper threshold of 4.0 kW given by the IMO, indicating excellent flame-retardant performance. The results of a previous study reported the evaluation of the flame-retardant performance of Korean pine wall panels treated with the same flame-retardant resin as that used in this study by vacuum pressure impregnation according to the test method under ISO 5660-1, and the total heat release (THR) values were shown to be 6.24 MJ/㎡ and 4.18 MJ/㎡, which were below the upper threshold of 8.0 MJ/㎡ for acceptance and satisfied the flame-retardant performance standard [14].

In conclusion, the test specimens in this study were considered to meet the requirements for low flame spread according to the recommendation for fire test procedures for the surface flammability of surface materials specified in IMO Resolution MSC 307(88)[9].

4. Conclusions

Experimental results of evaluation of toxic gas generation, smoke generation, and flammability in Flame-Retardant Plywood Combustion Tests are summarized as follows;

Based on the result of the toxic gas generation, there are seven out of the eight kinds of the toxic gases generated during the combustion of flame-retardant plywood as identified by the IMO, other than CO, were not detected. Even CO gas was detected in quantities smaller than the thresholds under three test conditions.

In smoke generation tests, the mass reduction rate showed a tendency to increase as the test conditions became more severe, with values of 13%, 18%, and 20%. In addition, as the test conditions became more severe, the average of the maximum specific optical density showed values of 75.70, 81.00, and 191.20. However, all of the values satisfied the standard given by the IMO, which is below 200.

Flammability was evaluated and the results showed the values of critical flux at extinguishment (CFE) as 49.5kW/㎡ on average, total heat release (Qt) as 0.21MJ on average, and peak heat release rate (Qp) as 0.66kW on average, which satisfied all of the thresholds presented by the IMO.

In the case of flame-retardant plywood, the applicability was confirmed as a finishing material suitable for IMO flame safety standards of toxic gas generation, smoke generation, and flammability.

Through this study, it is expected that flame-retardant wood materials will be recognized and widely used as a building finishing material that is safer from fire and can minimize human damage.

Author Contributions: H.-J.P. : conceptualization, investigation, methodology, writing—original draft, writing—review and editing. S.-U.J. : experiment, data analysis, supervision, writing—original draft, writing—review and editing. All authors have read and agreed to the published version of the manuscript.

Funding: This study was carried out with the support of ′R&D Program for Forest Science Technology (Project No. "FTIS 2020223B10-2222-AC02", "FTIS 2022457B10-2224-AC02")′ provided by Korea Forest Service (Korea Forestry Promotion Institute).

Acknowledgments: This study was carried out with the support of ′R&D Program for Forest Science Technology (Project No. "FTIS 2020223B10-2222-AC02", "FTIS 2022457B10-2224-AC02")′ provided by Korea Forest Service (Korea Forestry Promotion Institute).

Conflicts of Interest: The authors declare no conflict of interest.

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