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Comparative Assessment of Fire Effluent Toxicity of Flame-Retardant Coatings and Films

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Abstract

Flame-retardant coatings and films are widely used to delay flame spread on interior finishing and wood-based materials; however, their fire effluent toxicity has not been sufficiently characterized, and direct comparisons between these product types remain scarce. This study evaluated three commercial flame-retardant coatings and three flame-retardant films using the KS F 2271 gas toxicity test, NES 713 toxicity index test, and Py-GC/MS and HS-GC/MS analyses. Representative coating and film products were also applied to medium-density fiberboard (MDF) to assess average incapacitation time, total smoke release (TSR), and total heat release (THR). All tested specimens, including the 1 coat/layer, increased-loading, and MDF-applied conditions, satisfied the Korean gas toxicity criterion of 9 min. However, increased loading affected the two product groups differently; the intumescent coating showed a marked reduction in average incapacitation time, whereas the films remained relatively stable. The coatings produced higher toxicity indices and more diverse detected gases and pyrolysis products than the films. In MDF-based specimens, flame-retardant treatment increased average incapacitation time and reduced TSR and THR. These findings show that fire effluent toxicity differs between coatings and films and should be considered together with flame-retardant performance.

Keywords: flame-retardant coating; flame-retardant film; fire effluent toxicity; gas toxicity test; toxicity index test; total smoke release; total heat release

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1. Introduction

Despite continuous advances in fire protection systems and fire suppression technologies, fires in residential occupancies remain a persistent life-safety problem. According to the 2025 Statistical Yearbook of the National Fire Agency of Korea, fires in houses and apartments continued to occur at a substantial annual frequency during 2020–2024, with no clear reduction in associated casualties [1]. In Korea, an analysis of fatal apartment fires also reported that most deaths were associated with smoke and toxic gas inhalation, whereas deaths caused only by burns accounted for a much smaller fraction [2]. Therefore, in addition to active fire protection systems, passive and material-based fire safety strategies are needed to reduce fire growth, smoke exposure, and toxic fire effluent hazards during the early stage of fire. In this context, Korea has been considering broader application of flame-retardant products as one practical measure to reduce fire damage and improve fire safety in buildings and fire-vulnerable occupancies.

Smoke and toxic fire effluents are critical factors in fire safety because they can reduce visibility, impair evacuation, and cause incapacitation before direct flame exposure occurs. Stec emphasized that fire toxicity remains a major contributor to injury and death in unwanted fires, particularly under under-ventilated conditions where toxic product yields can increase substantially [3]. Purser also noted that toxic hazards in enclosed fires depend on fire scenario, ventilation, fire growth, and time-dependent toxic product yields, rather than on the intrinsic toxicity of a material alone [4]. These studies indicate that the fire safety of interior materials should be evaluated not only by their ability to resist ignition or flame spread but also by the characteristics of smoke and toxic gases generated during combustion.

The use of flame-retardant products is one practical approach to improving fire safety in residential buildings, multi-use facilities, and high-occupancy spaces. Compared with the installation of new active fire protection systems, flame-retardant surface treatments can be applied relatively easily to existing interior materials and combustible products. Flame-retardant coatings and surface treatments can improve fire performance by forming protective surface layers, promoting char formation, reducing heat transfer, or suppressing the release of combustible volatile products [5,6]. In particular, intumescent systems are designed to expand under heat exposure and form a thermally insulating char layer, which can delay heat and mass transfer between the flame and the underlying substrate [7,8]. A recent review has emphasized the continued development of intumescent and flame-retardant coating systems to improve flame resistance and address broader fire safety concerns, including heat release, smoke generation, and toxic effluents [9].

However, the fire safety of flame-retardant products cannot be evaluated solely on the basis of flame-retardant performance. Flame-retardant formulations contain various components, including binder resins, solvent or dispersing media, plasticizers, additives, fillers, and flame-retardant ingredients. These components can affect thermal decomposition pathways and the chemical composition of fire effluents [5]. In polymeric materials, the type of polymer and flame-retardant formulation can strongly influence pyrolysis behavior and volatile product distributions. For example, recent TG-FTIR-GC/MS analysis of flame-retardant PVC and PE showed that flame-retardant PVC generated HCl and aromatic compounds during dehydrochlorination and main-chain scission, whereas flame-retardant PE mainly produced alkenes and alkanes [10]. Similarly, a study on polyurethane-based materials reported that flame-retardant addition can delay flame propagation while also affecting heat release characteristics and gaseous combustion products, including toxic or irritating gases [11].

This issue is particularly relevant for surface-applied flame-retardant products such as flame-retardant coatings and flame-retardant films. Although both product types are used to protect combustible substrates, their application forms and thermal decomposition behavior differ. Flame-retardant coatings are applied as liquid formulations that form dried coating layers after curing or drying, whereas flame-retardant films are polymer sheets with adhesive layers attached to substrate surfaces. Therefore, the two product types may differ in decomposed components, volatile organic compounds, char-forming behavior, smoke release, heat release, and fire effluent toxicity.

In Korea, the performance evaluation of flame-retardant target products is mainly based on combustibility-related indices such as after-flame time, after-glow time, charred area, charred length, and maximum smoke density [12]. These criteria are useful for determining whether a product can suppress flame spread, but they do not directly evaluate the toxicity of fire effluents generated during combustion. By contrast, for building interior materials, the KS F 2271 gas toxicity test is applied in the classification of non-combustible, semi-non-combustible, and flame-retardant building materials under the Korean quality

recognition and management system for building materials [13]. However, this gas toxicity criterion is not generally incorporated into the performance evaluation of flame-retardant target products, including those applied to interior finishing materials and wood-based substrates. Therefore, additional information on fire effluent toxicity is needed to better understand the fire safety characteristics of flame-retardant products.

Previous studies on flame-retardant materials have mainly focused on heat release rate, ignition time, flame spread, char formation, smoke production, and flame-retardant mechanisms [5–11]. These studies have shown that flame-retardant treatments can modify combustion behavior and delay fire development. In addition, a recent study has combined cone calorimetry with gas toxicity or gas analysis methods to evaluate fire risk factors and toxic gas emission characteristics of polymeric materials [14]. These studies demonstrate the usefulness of combining heat release, smoke, and gas toxicity indicators. However, comparative information on the fire effluent toxicity of flame-retardant coatings and flame-retardant films remains limited. Hansen-Bruhn and Hull investigated the smoke toxicity of fire-protecting timber treatments using untreated, pressure-impregnated, and surface-coated plywood under different fire conditions [15]. Nevertheless, their study focused on timber treatment systems, and direct comparisons between commercially used flame-retardant coatings and films remain limited. The behavior of these products before and after application to combustible substrates has also received relatively little attention.

In this study, commercial surface-applied flame-retardant coatings and flame-retardant films used in Korea were selected to compare their fire effluent toxicity. The flame-retardant coatings included an acrylic water-based emulsion coating, a chlorinated rubber solvent-based coating, and a phosphorus–nitrogen intumescent coating. The flame-retardant films were PVC-based adhesive polymer films. The specimens were evaluated using the KS F 2271 gas toxicity test, the NES 713 toxicity index test, and Py-GC/MS and HS-GC/MS analyses to identify major pyrolysis products and headspace volatile compounds. In addition, representative coating and film products were applied to medium-density fiberboard (MDF) to compare changes in average incapacitation time, total smoke release (TSR), and total heat release (THR) before and after flame-retardant treatment.

The objective of this study is to compare the fire effluent toxicity of flame-retardant coatings and films, examine the effects of coating/layer loading on average incapacitation time and toxicity index, and interpret the results in relation to chemical characterization and MDF-applied fire behavior. This study aims to clarify how product type, loading, chemical composition, and substrate application influence the fire effluent toxicity characteristics of surface-applied flame-retardant products.

2. Materials and Methods

2.1. Materials

Three commercial flame-retardant coatings and three flame-retardant films commonly used in Korea were selected to compare the fire effluent toxicity of surface-applied flame-retardant products. The flame-retardant coatings were designated as C1, C2, and C3, while the flame-retardant films were designated as F1, F2, and F3. The coatings represented different formulation types, including an acrylic water-based emulsion coating, a chlorinated rubber solvent-based coating, and a phosphorus–nitrogen intumescent coating. The flame-retardant films were PVC-based adhesive polymer films with different surface patterns or textures. The classification and main characteristics of the specimens are summarized in Table 1.

Medium-density fiberboard (MDF) was selected as a representative combustible wood-based substrate to examine the effect of flame-retardant treatment under substrate-applied conditions. The MDF-based specimens were classified as untreated MDF, MDF-Coating,

and MDF-Film. MDF-Coating was prepared by applying C1 to the exposed surface of MDF, and MDF-Film was prepared by attaching F2 to the exposed surface of MDF. C1 and F2 were selected as representative coating and film products because their gas toxicity and toxicity index results were located within the intermediate range among the tested coating and film products, respectively. Table 2 summarizes the experimental design, including the test methods, specimen types, main test conditions, and principal outputs used to evaluate fire effluent toxicity, smoke release, heat release, and chemical characteristics.

Table 1. Classification and product characteristics of the flame-retardant coating and film specimens used in this study.

Sample	Category	Main Base Material	Product-Level Characteristics
C1	Coating	Acrylic emulsion resin	Water-based transparent flame-retardant coating
C2	Coating	Chlorinated rubber resin	Solvent-based transparent flame-retardant coating
C3	Coating	Ammonium polyphosphate-containing resin	Water-based white intumescent-type flame-retardant coating
F1	Film	PVC-based adhesive film	Light marble-type design; eco-friendly building material certified
F2	Film	PVC-based adhesive film	Wood-type design; eco-friendly building material certified
F3	Film	PVC-based adhesive film	Linen-type design

Table 2. Summary of the fire toxicity, combustion, and chemical characterization tests used in this study.

Test	Specimens	Main Condition	Main Output
KS F 2271 Gas Toxicity Test	C1–C3, F1–F3, MDF-based specimens	6 min heating, 15 min observation	Average incapacitation time
NES 713 Toxicity Index Test	C1–C3, F1–F3	Combustion of approximately 1 g specimen	Toxicity index and detected gases
ISO 5660-1 Cone Calorimeter Test	MDF-based specimens	50 kW/m^2, 10 min	TSR and THR

2.2. Preparation of Test Specimens

For the KS F 2271 gas toxicity test [16], product-only specimens were prepared by applying the flame-retardant coatings or films to aluminum plates. Aluminum plates were used as inert supporting substrates to minimize the influence of combustible base materials and to focus on the relative fire effluent toxicity of the applied flame-retardant product.

For the coating specimens, 0.5 mm thick aluminum plates were used as supporting substrates. C1, C2, or C3 was applied to the exposed surface using a roller and dried sufficiently at room temperature before testing. After drying, the measured dry film thicknesses of the 1-coat specimens were 0.74 mm for C1, 0.57 mm for C2, and 0.21 mm for C3. The 1 coat/layer condition thus represents a practical single-application loading according to manufacturer-provided conditions, rather than a strictly equal-thickness or equal-mass comparison among different products. To examine the effect of coating loading, specimens were prepared under 1-coat and 5-coat conditions.

For the film specimens, F1, F2, or F3 was attached to the same type of 0.5 mm thick aluminum plate using the adhesive side of each film. The nominal thickness of the PVC-based adhesive film used for F1–F3 was 0.22 mm. After attachment, the specimens were conditioned at room temperature for at least 24 h to stabilize adhesion. Specimens showing lifting, delamination, or poor adhesion were excluded and re-prepared. To examine the effect of film loading, specimens were prepared under 1-layer and 5-layer conditions.

The gas toxicity test specimens were prepared with three through-holes of 25 mm diameter from the front surface to the back surface in accordance with KS F 2271 [16]. For the substrate-applied specimens, 18 mm thick MDF was used, and MDF-Coating was prepared by applying C1 once to one exposed surface of MDF using a roller according to the manufacturer's recommended dry film thickness. MDF-Film was prepared by attaching one commercial layer of F2. The treatments were conducted under practical application conditions for each product type rather than with strictly controlled identical thickness or applied mass per unit area. The MDF-based specimens were cut to the required dimensions for the KS F 2271 gas toxicity test and the KS F ISO 5660-1 cone calorimeter test [16,17].

2.3. Gas Toxicity Test

The biological effect of fire effluents was evaluated using the KS F 2271 gas toxicity test [16]. In this test, laboratory animals are exposed to combustion gases generated from a specimen under specified heating conditions, and the relative biological effect of the fire effluents is evaluated based on the time to incapacitation. The test result is expressed as the average incapacitation time, where a longer average incapacitation time indicates a relatively lower biological effect of the generated combustion gases.

Each specimen was installed in the heating furnace and heated using an LPG propane flame and an electric heater. Heating was initiated using the LPG propane flame, followed by additional heating with the electric heater. The total heating duration was 6 min, and the total observation period was 15 min. Combustion gases generated during heating were transferred into the exposure chamber. The movement of laboratory animals was recorded using a rotating wheel and sensor system, and the time at which the last movement was detected was defined as the incapacitation time. The average incapacitation time of 9 min, which is used as the gas toxicity criterion for Korean building finishing materials, was applied as a relative evaluation criterion in this study [13,16].

2.4. Toxicity Index Test

The toxicity index test was conducted in accordance with NES 713 to identify and compare major toxic gaseous components generated from the flame-retardant coatings and films [18]. NES 713 evaluates the toxicity of combustion products by measuring selected small-molecular toxic gases generated during combustion and summing their contributions relative to specified reference concentrations. The gases commonly considered in the NES 713 toxicity index include CO_2, CO, H_2S, NH_3, HCl, HBr, HF, HCN, NOx, SO_2, formaldehyde, phosgene, acrylonitrile, and phenol [18]. In this study, the analysis was performed for the detectable gases among the target gases specified in the test method.

The specimens were prepared to have a mass of approximately 1 g. For the coating specimens, the flame-retardant coating was repeatedly applied and dried on aluminum plates until the target mass was obtained. For the film specimens, the films were cut or layered to meet the target mass. The prepared specimens were combusted under a high-temperature flame condition. After combustion, the gases in the chamber were mixed, and the concentrations of individual gas components were measured using gas detection tubes.

The toxicity index was calculated by summing the ratio of the converted concentration of each gas component (Cθ) to its reference concentration (Cf). Cθ was calculated by converting the measured concentration to the concentration corresponding to the combustion of 100 g of material in a 1 m^3 volume, taking into account the specimen mass and chamber volume [18]. In addition to the total toxicity index, the Cθ/Cf contribution of each gas component was compared to identify the dominant toxic contributors for the coatings and films.

2.5. Cone Calorimeter Test

The smoke release and heat release characteristics of the MDF-based specimens were evaluated using a cone calorimeter test in accordance with ISO 5660-1 [17]. The specimens tested were untreated MDF, MDF-Coating, and MDF-Film. The cone calorimeter test was conducted to examine how flame-retardant treatment changed the combustion behavior of MDF under controlled external heat flux conditions.

The test was performed at an external heat flux of 50 kW/m^2. Each specimen was placed in the specimen holder beneath the cone heater, and ignition was initiated using an igniter positioned between the cone heater and the specimen. The test duration was 10 min. During the test, the exhaust gas flow rate, oxygen concentration, carbon dioxide concentration, carbon monoxide concentration, and pressure were measured. These data were used to calculate TSR and THR. TSR represents the cumulative smoke release during the test, whereas THR represents the cumulative heat released, calculated based on the oxygen consumption principle. ISO 5660-1 specifies the cone calorimeter method for assessing heat release rate and dynamic smoke production rate, with heat release rate determined from oxygen concentration and the flow rate in the combustion product stream [17].

2.6. Py-GC/MS and HS-GC/MS Analysis

GC/MS analyses were performed to characterize the pyrolysis products and headspace volatile compounds generated from the flame-retardant coatings and films. Py-GC/MS analysis was conducted using a Frontier Lab PY-3030D pyrolyzer coupled with an Agilent 7890B/5977A GC/MS system. This analysis was used to identify major organic compounds generated when the specimens decomposed under high-temperature pyrolysis conditions. C1–C3 and F1–F3 were analyzed and compared to evaluate differences in major pyrolysis products among the coating and film specimens.

HS-GC/MS analysis was conducted using an Agilent headspace sampler coupled with an Agilent 7890B/5977A GC/MS system to identify volatile compounds present in the headspace of the specimens. C1–C3 and F1–F3 were analyzed, and the major headspace volatile compounds detected from each specimen were compared. GC/MS-based analysis provides qualitative information on complex volatile organic compounds generated during heating or pyrolysis and can complement toxicity index data, which are limited to specific target gases.

2.7. Data Interpretation

The KS F 2271 gas toxicity test results were interpreted based on average incapacitation time. A longer average incapacitation time was interpreted as indicating a relatively lower biological effect of the fire effluents generated from the specimen, whereas a shorter average incapacitation time was interpreted as indicating a relatively higher biological effect. Because the tests were conducted within the repetition requirements of the relevant standards, the results were interpreted mainly in terms of relative trends and consistency among product groups rather than statistical significance.

The NES 713 test results were interpreted by considering both the total toxicity index and the detected gas components. A higher toxicity index does not simply indicate a larger total amount of gas generation; it may also indicate that highly toxic components with low reference concentrations contributed significantly to the overall index. Therefore, the Cθ/Cf values of individual gases, particularly NOx, HCN, and acrylonitrile, were examined to compare the toxic contributions of the flame-retardant coatings and films.

The MDF-based specimen results were interpreted by integrating average incapacitation time, TSR, and THR. Average incapacitation time was used as an indicator of the biological effect of fire effluents, TSR was used as an indicator of smoke generation, and

THR was used as an indicator of cumulative heat release. This integrated interpretation was used to compare the overall fire safety tendency of untreated and flame-retardant-treated MDF-based specimens.

3. Results and Discussion

3.1. Gas Toxicity of Flame-Retardant Coatings and Films

The gas toxicity test was first conducted under the 1 coat/layer condition to compare the baseline fire effluent toxicity of the flame-retardant coatings and films. In this study, a longer average incapacitation time was interpreted as indicating a relatively lower biological effect of the combustion gases generated from the specimen. Because the Korean government gas toxicity criterion for building finishing materials is based on an average incapacitation time of 9 min, this value was used as a relative evaluation criterion in the present study [13,16].

Figure 1 shows photographs of the specimens before and after the gas toxicity test. All specimens lost their original appearance and exhibited carbonized or thermally damaged surfaces after exposure to the test heating condition. However, visual observation alone was not sufficient to distinguish the relative gas toxicity of the specimens. Therefore, the average incapacitation time was used as the primary indicator for comparing the biological effect of the generated fire effluents.

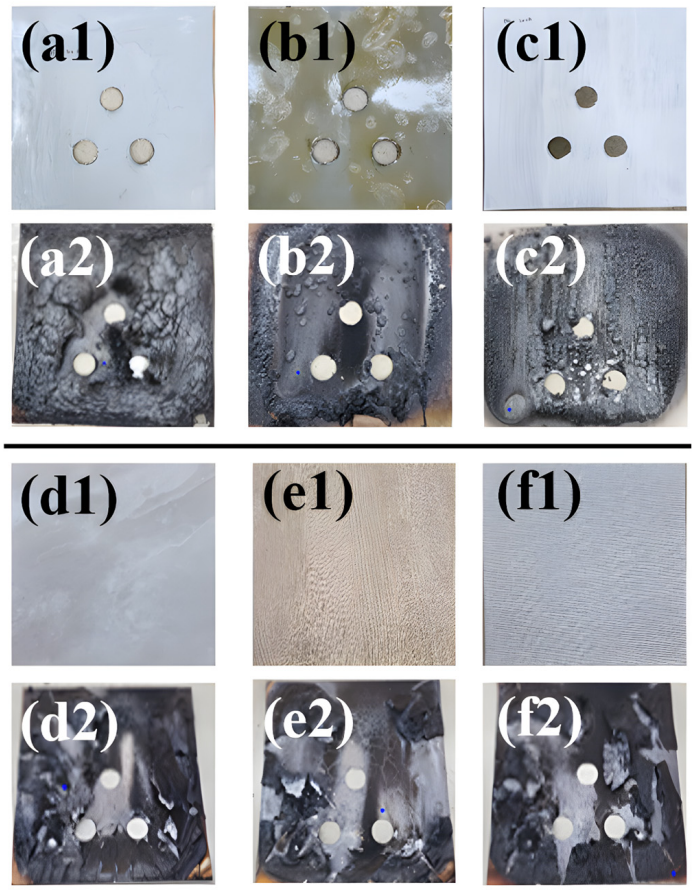


Figure 1. Photographs of the flame-retardant coating and film specimens before and after the KS F 2271 gas toxicity test. Panels (**a**–**c**) show the coating specimens C1, C2, and C3, respectively, and panels (**d**–**f**) show the film specimens F1, F2, and F3, respectively. For each specimen, images labeled "1" represent the specimens before testing, and images labeled "2" represent the corresponding specimens after the gas toxicity test: (**a1**,**a2**) C1, (**b1**,**b2**) C2, (**c1**,**c2**) C3, (**d1**,**d2**) F1, (**e1**,**e2**) F2, and (**f1**,**f2**) F3. The three through-holes in each specimen were prepared in accordance with KS F 2271.

Table 3 summarizes the average incapacitation times of C1–C3 and F1–F3 under the 1 coat/layer condition. The average incapacitation times of C1, C2, and C3 were 14:07, 13:39, and 14:43, respectively. All three coating specimens exceeded the criterion of 9 min, and the differences among the coatings were relatively small under the single-coat condition. C2 showed a slightly shorter average incapacitation time than C1 and C3, but the difference was limited to within approximately 1 min. Therefore, under the 1 coat condition, the differences in coating formulation did not produce a marked difference in the gas toxicity test results.

Table 3. Average incapacitation times of the flame-retardant coatings and films under the 1 coat/layer condition.

Specimen	Category	Coating/Layer Count	Average Incapacitation Time (min:s)
C1	Coating	1	14:07
C2	Coating	1	13:39
C3	Coating	1	14:43
F1	Film	1	14:54
F2	Film	1	14:54
F3	Film	1	13:55

The average incapacitation times of F1, F2, and F3 were 14:54, 14:54, and 13:55, respectively. F1 and F2 showed long average incapacitation times close to the end of the observation period, and F3 also showed a value close to 14 min. Compared with the coating specimens, the film specimens showed similar or slightly longer average incapacitation times under the 1 layer condition. This result indicates that the PVC-based flame-retardant films did not generate fire effluents that caused rapid biological incapacitation under the present test condition.

Under the 1 coat/layer condition, the coatings exhibited average incapacitation times of 13:39–14:43 min, which were similar to those of the film specimens (13:55–14:54 min). This similarity does not imply identical fire effluent chemistry between the coatings and films. Rather, it reflects the dominance of the initial physical barrier effect at practical single-application loadings. At these thicknesses, both product types effectively delayed heat penetration and oxygen diffusion to the inert aluminum substrate before substantial thermal decomposition occurred. The coatings formed thin protective layers through different mechanisms (acrylic barrier in C1, gas-phase inhibition in C2, and initial char formation in C3), while the PVC-based films acted as uniform polymeric sheets. Although PVC can undergo dehydrochlorination to release HCl and aromatic compounds [10], and coatings contain multi-component binders and additives that influence decomposition pathways [5], the integrated biological response of the KS F 2271 test (whole fire effluent exposure) did not produce rapid incapacitation. Most specimens showed average incapacitation times close to the upper limit of the 15 min observation period. Formulation-dependent differences became more evident at higher loadings (Section 3.2).

When only the 1 coat/layer condition is considered, all flame-retardant coatings and films tested in this study satisfied the Korean gas toxicity criterion. However, this result should be interpreted as a relative evaluation under a controlled laboratory condition. In practical applications, fire effluent toxicity and combustion behavior may vary depending on applied amount, dry film thickness, number of film layers, curing or drying condition, substrate type, and heat exposure condition. Therefore, the effect of increased coating/layer loading and the contribution of individual toxic gases were further examined to compare the fire effluent toxicity characteristics of the coating and film products.

3.2. Effect of Coating/Layer Loading on Gas Toxicity

In practical use, the applied amount of a flame-retardant coating or the number of attached film layers may vary depending on the product, substrate, and installation condition. Therefore, the 1 coat/layer and increased-loading conditions were compared to examine how increased loading affected the average incapacitation time of the coating and film specimens.

Figure 2 compares the average incapacitation times under the 1 coat/layer and increased-loading (5 coats/layers) conditions. For the coating specimens, C1 decreased from 14:07 to 12:47, and C2 showed only a small change from 13:39 to 13:31. In contrast, C3 showed a more pronounced decrease from 14:43 to 10:12 under the 5 coats condition. This difference can be attributed to the distinct flame-retardant mechanisms and formulation characteristics of the coatings. C1 is an acrylic barrier-type coating, and C2 is associated with chlorinated-rubber-based gas-phase flame-retardant action. In contrast, C3 is a phosphorus–nitrogen intumescent coating. Increased loading of an intumescent coating can increase not only the potential char-forming contribution but also the amount of thermally decomposable formulation components, including binder, additives, and intumescent ingredients. In the present product-only test using an aluminum plate as an inert supporting substrate, the measured fire effluent was mainly influenced by the thermal decomposition of the coating layer itself, rather than by the suppression of pyrolysis from an underlying combustible substrate. Therefore, coating-derived volatile and gaseous products could contribute more directly to the fire effluent as the C3 loading increased. The marked decrease observed for C3 was thus interpreted as a formulation-dependent loading effect. In comparison, the barrier and gas-phase mechanisms of C1 and C2 appeared less sensitive to increased loading under the present test conditions [5,9,19]. Although C3 still exceeded the Korean gas toxicity criterion of 9 min, the reduction in average incapacitation time indicates that increased coating loading can increase the relative biological effect of fire effluents for certain coating formulations.

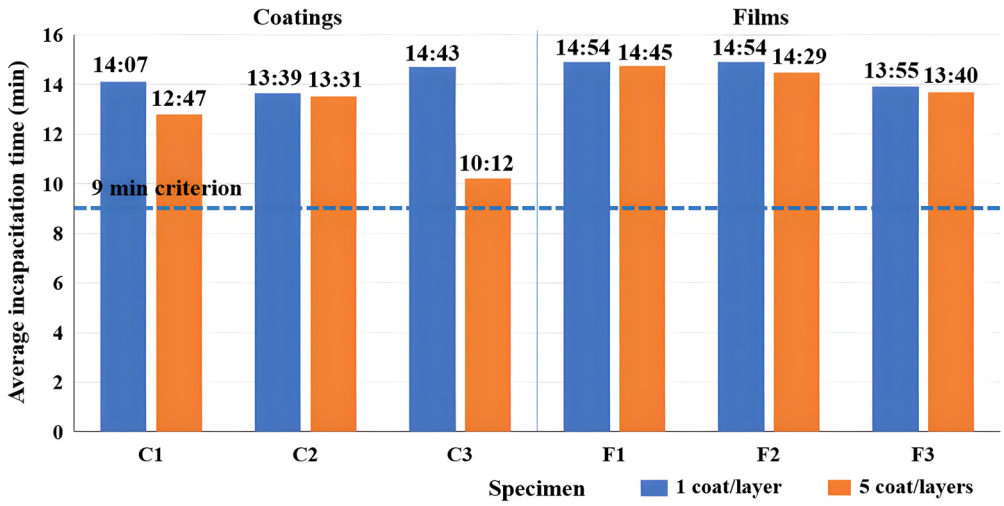


Figure 2. Effect of coating/layer loading on average incapacitation time. The 1 coat/layer and increased-loading conditions are compared for flame-retardant coatings and films. The dashed line indicates the 9 min criterion of the KS F 2271 gas toxicity test.

The different loading behavior between coatings and films can also be explained by their different material structures. The coating specimens were liquid-applied multi-component systems, whereas the film specimens were preformed polymeric sheets. For the film specimens, F1, F2, and F3 showed only limited decreases in average incapacitation time under the 5-layer condition, from 14:54 to 14:45, 14:54 to 14:29, and 13:55 to 13:40,

respectively. Thus, the films maintained relatively stable gas toxicity test results even when the number of layers increased. On a group-average basis, the coating specimens showed a decrease in average incapacitation time from approximately 14:10 to 12:10, corresponding to a reduction of about 14.1%. In contrast, the film specimens decreased only from approximately 14:34 to 14:18, corresponding to a reduction of about 1.9%. This quantitative comparison indicates that the loading effect was more pronounced for the coating specimens than for the film specimens under the present test conditions. Compared with liquid-applied coatings, layer-by-layer film application may produce less variability in applied mass and surface morphology, which may partly explain the relatively small changes observed for the film specimens under increased loading.

However, because the films used in this study were PVC-based polymer films, their thermal decomposition behavior should not be generalized only from the gas toxicity test results. Previous TG-FTIR-GC/MS studies have shown that flame-retardant PVC materials can undergo dehydrochlorination and subsequent main-chain scission, producing HCl, aromatic hydrocarbons, and other volatile products depending on formulation and pyrolysis condition [10]. Therefore, the relatively stable gas toxicity test results of the film specimens should be interpreted together with the toxicity index and GC/MS results discussed in the following sections.

3.3. Toxicity Index and Detected Gaseous Components

The toxicity index results obtained using NES 713 are summarized in Table 4. Unlike the KS F 2271 gas toxicity test, which evaluates the integrated biological response to the whole fire effluent, the NES 713 toxicity index is calculated from selected target gases and their corresponding reference concentrations. Therefore, the toxicity index provides analytical information on the relative contribution of specific gaseous toxicants, but it does not necessarily show a direct correlation with average incapacitation time.

For the coating specimens, the toxicity indices were 1.24 for C1, 0.53 for C2, and 0.80 for C3. C1 showed the highest toxicity index among the coating specimens, mainly due to the contribution of NOx. In C1, CO_2, NOx, SO_2, and acrylonitrile were detected, whereas HCN was not detected. In C2 and C3, CO_2, NOx, SO_2, acrylonitrile, and HCN were detected. However, the contribution of HCN to the toxicity index was relatively limited under the present test conditions. These results indicate that the toxicity index of the coating specimens was influenced not only by the presence or absence of HCN, but also by the combined contributions of NOx, SO_2, acrylonitrile, CO_2, and HCN.

For the film specimens, the toxicity indices were 0.14 for F1, 0.18 for F2, and 0.22 for F3. These values were lower than those of the coating specimens. In the film specimens, CO_2, NOx, and SO_2 were mainly detected, whereas acrylonitrile and HCN were not detected under the present test conditions. This result suggests that the selected target gas contributions measured by NES 713 were more limited for the film specimens than for the coating specimens. On average, the toxicity index of the coating specimens was approximately 4.8 times higher than that of the film specimens, indicating a clear quantitative difference between the two product groups under the present test conditions. The SO_2 detected in Table 4 was likely derived from minor sulfur-containing additives, fillers, stabilizers, or processing residues present in the commercial flame-retardant films and coatings.

The toxicological importance of the detected gases differs depending on their chemical nature and exposure scenario. Fire smoke toxicity is generally governed by both asphyxiant gases and irritant gases, and analytical gas data provide important information for interpreting toxic potency, although biological response and fire scenario effects should also be considered [3,20,21]. HCN is widely recognized as an important toxicant in fire smoke, particularly when nitrogen-containing materials are involved, because it can contribute

to rapid incapacitation in combination with other fire gases [22]. Acrylonitrile is also a toxic nitrile compound, and its toxicity has been associated with both intrinsic biological reactivity and cyanide-related metabolic pathways [23,24]. NOx and SO_2 are irritant gases that can contribute to respiratory effects, and their contribution can be emphasized in toxicity index calculations because of their relatively low reference concentrations in NES 713 [18].

Table 4. Toxicity indices and detected gaseous components of the flame-retardant coatings and films.

Specimen	Toxicity Index	Detected Component	Cθ (ppm)	Cf (ppm)	Cθ/Cf
C1	1.24	CO_2	14,285	1,000,000	0.01
		NO_x	236	250	0.94
		SO_2	18	400	0.05
		Acrylonitrile	7	400	0.02
C2	0.53	CO_2	19,580	1,000,000	0.02
		NO_x	162	250	0.65
		SO_2	5	400	0.01
		Acrylonitrile	5	400	0.01
		Hydrogen cyanide (HCN)	5	150	0.03
C3	0.80	CO_2	13,043	1,000,000	0.01
		NO_x	144	250	0.58
		SO_2	11	400	0.03
		Acrylonitrile	4	400	0.01
		Hydrogen cyanide (HCN)	9	150	0.06
F1	0.14	CO_2	7,306	1,000,000	0.01
		NO_x	6	250	0.02
		SO_2	18	400	0.05
F2	0.18	CO_2	12,978	1,000,000	0.01
		NO_x	3	250	0.01
		SO_2	16	400	0.04
F3	0.22	CO_2	14,757	1,000,000	0.01
		NO_x	6	250	0.02
		SO_2	18	400	0.05

When the gas toxicity test (Table 3) and toxicity index test (Table 4) results are compared, some differences are observed, particularly for the coating specimens. The KS F 2271 gas toxicity test evaluates the integrated biological response to the whole fire effluent, expressed as average incapacitation time, whereas the NES 713 toxicity index quantifies the contribution of selected target gases based on their measured concentrations and reference toxicity values. Therefore, the two methods provide complementary but not directly interchangeable information. Although the coating specimens generally showed higher toxicity indices than the film specimens, their average incapacitation times remained relatively long under the present test conditions. In particular, C1 showed the highest toxicity index mainly due to the NOx contribution, but it did not show the shortest incapacitation time in the KS F 2271 test. This indicates that the toxicity index can be strongly affected by selected gases with low reference concentrations, whereas the biological response is influenced by the combined and time-dependent effects of the whole fire effluent, including gas release timing, dilution, and possible synergistic or antagonistic interactions. In contrast, the film specimens showed more consistent behavior across the two methods, with relatively low toxicity indices and long average incapacitation times. Thus, the gas toxicity test and toxicity index test should be interpreted together to understand the toxicity characteristics

of flame-retardant coatings and films. The relationship between these target-gas results and the organic compounds identified by GC/MS is discussed in the following section.

3.4. Chemical Characterization of Pyrolysis Products and Headspace Volatiles

Py-GC/MS and HS-GC/MS analyses were conducted to provide additional chemical information on the thermal decomposition products and volatile components of the flame-retardant coatings and films. While the NES 713 toxicity index test quantifies selected target gases specified in the test method, GC/MS analysis identifies organic compounds generated during pyrolysis or released into the headspace. Therefore, GC/MS provides complementary information for interpreting the chemical characteristics of the fire effluents. However, because the TIC profiles were compared using relative intensity normalized to the maximum peak of each sample, the GC/MS results should be interpreted qualitatively rather than as direct quantitative evidence of toxic potency.

Figure 3 shows the representative Py-GC/MS total ion chromatograms of the coating and film specimens, and Figure 4 shows the representative HS-GC/MS total ion chromatograms. The major compounds identified by Py-GC/MS and HS-GC/MS are summarized in Table 5.

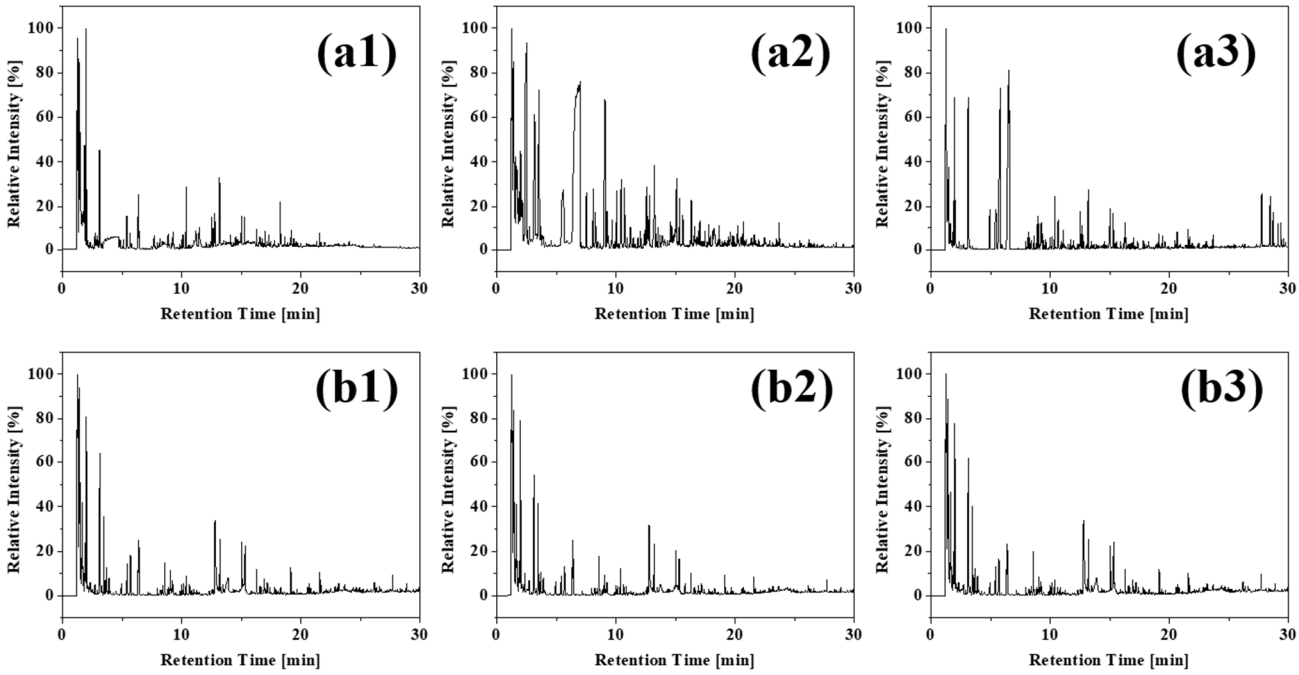


Figure 3. Representative Py-GC/MS total ion chromatograms of the flame-retardant coatings and films: (**a1**–**a3**) C1–C3 coating specimens and (**b1**–**b3**) F1–F3 film specimens. The chromatograms are presented as relative intensities normalized to the maximum peak of each specimen, and the major identified compounds are summarized in Table 5.

As summarized in Table 5, the coating specimens generated a wider range of organic pyrolysis products than the film specimens. C1 produced ethylbenzene, styrene, dichloroethane, phenol, toluene, and benzene as major pyrolysis products. C2 produced ethylbenzene, chlorobenzene, benzene, phenol, and toluene, whereas C3 produced pyridine, chlorobenzene, ethylbenzene, phenol, toluene, and ethylene glycol. These results indicate that the coating specimens generated various aromatic, chlorine-containing, and nitrogen-containing organic decomposition products depending on the coating formulation.

The film specimens showed comparatively simpler Py-GC/MS profiles. Because F1–F3 were all PVC-based flame-retardant adhesive films, their major decomposition products were broadly similar. The differences among the film specimens were less pronounced

than those among the coating specimens, although minor product-specific differences were observed. These differences may be associated with design type, surface texture, adhesive layer characteristics, and minor additives, but detailed formulation data were not available from the manufacturers.

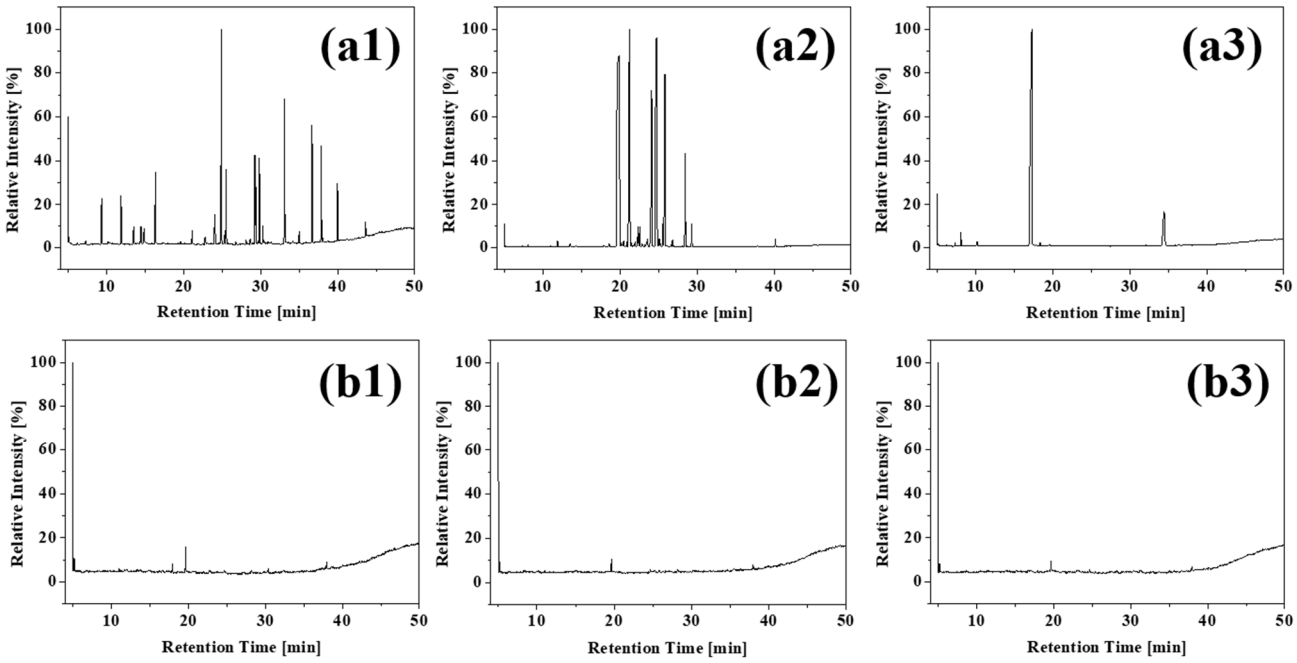


Figure 4. Representative HS-GC/MS total ion chromatograms of the flame-retardant coatings and films: (**a1**–**a3**) C1–C3 coating specimens and (**b1**–**b3**) F1–F3 film specimens. The chromatograms are presented as relative intensities normalized to the maximum peak of each specimen, and the major identified compounds are summarized in Table 5.

Table 5. Major pyrolysis products and headspace volatile compounds identified by Py-GC/MS and HS-GC/MS.

Analysis	Specimen	Major Compounds Identified
Py-GC/MS	C1	Ethylbenzene; styrene; dichloroethane; phenol; toluene; benzene
	C2	Ethylbenzene; chlorobenzene; benzene; phenol; toluene
	C3	Pyridine; chlorobenzene; ethylbenzene; phenol; toluene; ethylene glycol
	F1	Styrene; ethylbenzene; toluene
	F2	Styrene; toluene
	F3	Styrene; toluene
HS-GC/MS	C1	Methyl methacrylate; n-butyl acrylate; styrene
	C2	Toluene; ethylbenzene; styrene; xylene
	C3	Propylene glycol; diisobutyl adipate

The HS-GC/MS results also showed differences between coatings and films. The coating specimens released several volatile organic compounds, whereas the film specimens showed fewer major volatile components under the present headspace conditions. These results support the interpretation that the coating products had more diverse volatile and pyrolytic decomposition behavior than the film products.

The GC/MS results also help interpret the marked decrease in average incapacitation time observed for C3 under the 5-coat condition. C3 was a phosphorus–nitrogen

intumescent coating; however, under product-only test conditions, increasing the coating loading also increased the amount of coating material exposed to thermal decomposition. This may have enhanced the contribution of coating-derived volatile or gaseous products, even though the intumescent mechanism can provide heat shielding and flame-retardant effects [7–9].

In addition, the GC/MS results help to interpret the toxicity index results in Table 4, but the two analyses should be compared carefully because they target different chemical species and test conditions. NES 713 detects selected small-molecular gases such as CO_2, NOx, SO_2, acrylonitrile, and HCN under combustion conditions, whereas GC/MS mainly identifies organic pyrolysis products and headspace volatile compounds. Therefore, the absence of a specific organic compound in GC/MS does not necessarily contradict the detection of inorganic or small-molecular toxic gases in NES 713. For example, C1 showed a high NOx contribution in the toxicity index test, although major nitrogen-containing organic compounds were not prominent in the GC/MS results. Conversely, the detection of pyridine from C3 in Py-GC/MS and HCN in the NES 713 test suggests that nitrogen-containing formulation components may contribute to toxic gas generation during thermal decomposition. Therefore, the GC/MS results provide qualitative chemical support for the differences observed in the toxicity index results, while confirming that coatings and films have different decomposition characteristics.

Overall, the Py-GC/MS and HS-GC/MS analyses suggest that the differences in fire effluent toxicity between the coating and film specimens are related to both chemical composition and thermal decomposition behavior. The coating specimens generated more diverse pyrolysis products and headspace volatile compounds, whereas the film specimens showed a relatively limited distribution of major detected compounds under the present analytical conditions. Thus, GC/MS-based chemical characterization provided chemical context for interpreting the gas toxicity and toxicity index results, particularly when comparing flame-retardant products with different formulation types.

3.5. Gas Toxicity, Smoke Release, and Heat Release of MDF-Based Specimens

The results obtained from the flame-retardant products themselves do not fully represent the fire behavior that may occur when these products are applied to combustible substrates. Under product-only conditions, the test results mainly reflect the contribution of the flame-retardant layer itself to fire effluent generation. In contrast, under substrate-applied conditions, the observed fire behavior reflects the combined effect of the flame-retardant layer and its interaction with the underlying combustible substrate. Therefore, MDF was selected as a representative combustible wood-based substrate, and the coating and film were applied to MDF to compare changes in gas toxicity, TSR, and THR before and after flame-retardant treatment. This approach is relevant because the fire performance of flame-retardant-treated wood-based materials can be influenced by both the treatment layer and the substrate response [25,26].

Figure 5 shows the average incapacitation time, TSR, and THR of the MDF-based specimens. The average incapacitation time of untreated MDF was 12:05, whereas those of MDF-Coating and MDF-Film were 13:48 and 14:24, respectively. Thus, both flame-retardant-treated MDF specimens showed longer average incapacitation times than untreated MDF, with the longest value observed for MDF-Film. This result indicates that, under the present gas toxicity test condition, the flame-retardant treatments reduced the overall biological effect of the fire effluents generated from the MDF-based specimens.

MDF is a combustible wood-based composite containing wood fibers and resin components, and it can generate smoke, heat, and toxic combustion products during heating or combustion [25,27]. When a flame-retardant layer is applied to the MDF surface, it

may delay heat transfer to the substrate, suppress rapid substrate pyrolysis, and reduce the release of combustible or toxic volatile products. This substrate-protection effect provides a plausible explanation for the increased average incapacitation time observed for MDF-Coating and MDF-Film compared with untreated MDF. Similar improvements in fire performance have been reported for MDF or other wood-based panels treated with flame-retardant additives or coating strategies, particularly in terms of reduced heat release and smoke production [26,28,29].

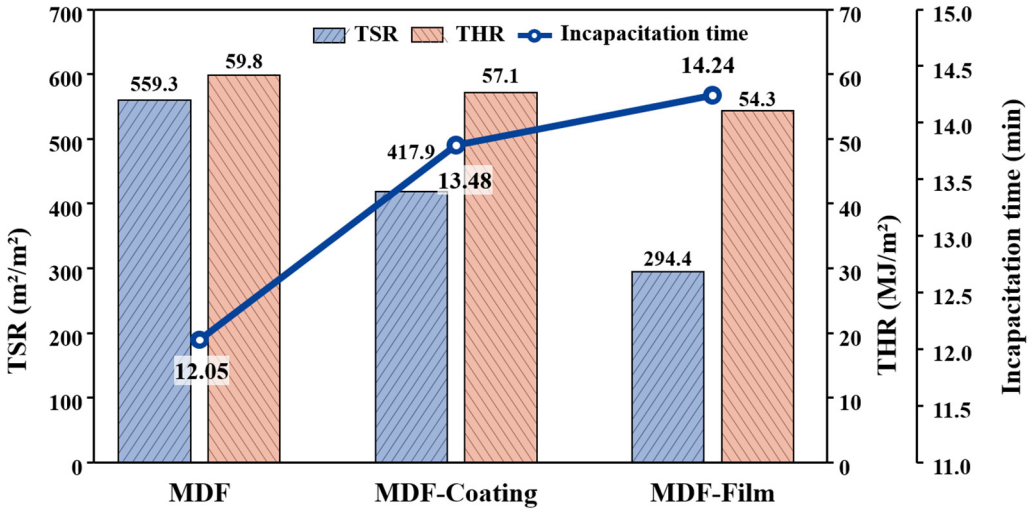


Figure 5. Comparison of average incapacitation time, total smoke release (TSR), and total heat release (THR) of MDF-based specimens.

The difference between MDF-Coating and MDF-Film can be interpreted in terms of the balance between the contribution of the treatment layer and its protective effect on the substrate. MDF-Film showed a greater increase in average incapacitation time than MDF-Coating. This may be related to the film layer physically covering the MDF surface and delaying heat transfer and volatile release from the substrate. MDF-Coating also increased the average incapacitation time compared with MDF, but the improvement was smaller than that observed for MDF-Film. A coating can form a protective surface layer, but the dried coating layer is also a formulation composed of binder, additives, fillers, and flame-retardant ingredients. Therefore, during heating, the coating layer itself may undergo thermal decomposition and contribute volatile or gaseous products, while simultaneously providing a protective function to the substrate [5,26]. These results suggest that the fire effluent toxicity of flame-retardant-treated substrates should be interpreted as the net outcome of both treatment-layer contribution and substrate-protection effect.

The smoke and heat release characteristics of the MDF-based specimens were evaluated using the cone calorimeter test in accordance with ISO 5660-1 [17]. The TSR of untreated MDF was 559.3 m^2/m^2, whereas those of MDF-Coating and MDF-Film were 417.9 m^2/m^2 and 294.4 m^2/m^2, respectively. THR also decreased after flame-retardant treatment. These results indicate that both flame-retardant treatments reduced cumulative smoke release and cumulative heat release from MDF under the present cone calorimeter condition, with the reduction being more pronounced for MDF-Film. Comparable reductions in heat release and/or smoke production have also been reported for MDF or wood-based panels treated with flame-retardant additives or coating strategies in cone calorimeter tests [28,29].

When the gas toxicity test and cone calorimeter results are considered together, the MDF-based specimens showed a consistent trend: average incapacitation time increased, whereas TSR and THR decreased after flame-retardant treatment. This suggests that

the reduced smoke and heat release may have contributed to the improved gas toxicity performance observed for the MDF-based specimens. In particular, reductions in TSR and THR are important because they reflect decreases in cumulative smoke release and cumulative heat release, respectively. Reduced smoke release can lessen smoke-related tenability concerns, while lower cumulative heat release can contribute to reduced fire growth potential. This interpretation is consistent with international fire safety guidance in which toxicity, heat, and smoke-related effects are considered important components of fire threat to people [17,30,31].

Overall, MDF-Film showed the most favorable combined trend among the MDF-based specimens in this study, with the longest average incapacitation time and the lowest TSR and THR. However, this result should be interpreted within the scope of the present experimental conditions. The observed tendency may depend on the specific MDF substrate, product formulation, applied amount, adhesion condition, heat flux, and test duration. Therefore, the present MDF-based results should not be interpreted as a general ranking of all flame-retardant coatings and films, but rather as a comparative case showing that substrate application can change the overall fire effluent toxicity, smoke release, and heat release behavior of flame-retardant-treated materials. Further studies using various substrates, product formulations, application amounts, installation conditions, and aging conditions are needed to verify the generality of these trends.

4. Conclusions

This study compared the fire effluent toxicity of commercial flame-retardant coatings and films using the KS F 2271 gas toxicity test, NES 713 toxicity index test, Py-GC/MS and HS-GC/MS analyses, and MDF-applied fire behavior tests. All tested specimens, including the 1 coat/layer, increased-loading, and MDF-applied conditions, satisfied the Korean gas toxicity criterion of 9 min. However, considerable differences in fire effluent toxicity were observed depending on product type, applied amount, chemical composition, and substrate application. Under the 1 coat/layer condition, both coatings and films showed relatively stable gas toxicity results. Under increased-loading conditions, however, the intumescent coating showed a marked decrease in average incapacitation time, whereas the film specimens remained relatively stable. The coating specimens also showed higher toxicity indices and more diverse detected gases and pyrolysis products than the film specimens. In the MDF-applied tests, MDF-Film showed a longer average incapacitation time and lower TSR and THR than MDF-Coating, indicating that the fire safety performance of flame-retardant-treated substrates can vary depending on the type and structure of the applied treatment layer.

Future studies should further clarify the mechanisms of toxicity generation from flame-retardant products under controlled loading and substrate conditions. In particular, the relationship between flame-retardant properties and toxic emissions should be investigated using time-resolved gas analysis, various product formulations, different substrate materials, and realistic application or aging conditions. Such studies will be important for understanding how flame-retardant mechanisms, toxic gas generation, smoke release, and heat release interact and ultimately influence fire safety outcomes. In addition, further research is needed to develop and validate alternative fire effluent toxicity assessment methods that can reduce or replace animal exposure while still providing scientifically reliable information on toxic gas generation and fire safety outcomes.

Overall, this study provides useful baseline information for evaluating the combustion-related toxicity characteristics of different flame-retardant coatings and films. The findings suggest that flame-retardant products should not be assessed only by conventional flame-retardant performance, but also by fire effluent toxicity, toxicity index, chemical

characterization, TSR, and THR. In practical material selection and fire safety assessment, both flame-retardant performance and product-specific toxicity behavior under expected loading and substrate conditions should be considered. By examining the hazardous characteristics of various flame-retardant products during combustion, this study may help support the development and selection of flame-retardant products whose toxicity under fire conditions is more carefully evaluated. These efforts may ultimately contribute to the broader application of safer flame-retardant products in various practical settings and to the reduction in fire-related life-safety risks.

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