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Improvement on flame retarding performance: Preparation and characterization of water-based indoor paints with addition of boric acid

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ABSTRACT

With the increasing population, a fire-protection of buildings has become necessary. Therefore, water-based indoor paints, which reduce the rate of the fire and offer the chance to intervene before the flames spread, have received attention; however, the performance and flame retardant property of these paints need to be improved. In this study, boric acid (H_3BO_3), which is the product of boron (B), was used as an additive for a flame retardant in the prepared water-based indoor paints. In order to investigate the effect of different weight ratios of H_3BO_3, the paints have been prepared with the addition of 5%, 10%, and 20% (w/w) of H_3BO_3. In addition, the synergistic effect of H_3BO_3 and melamine ($C_3H_6N_6$) on the flame retarding performance of the prepared paints was also investigated. The physicochemical properties, flammability characteristics and thermal properties were analyzed by Fourier transform infrared spectroscopy (FTIR), limiting oxygen index (LOI), and thermogravimetric analysis (TGA), respectively. The results demonstrated that the characteristic peaks of H_3BO_3 were observed in the prepared paints. When the flammability behavior of the paint samples were compared, the sample (P6) containing 20% (w/w) of H_3BO_3 showed the highest fire resistance property. Moreover, the results of the thermogravimetric analysis demonstrated that the prepared paints containing H_3BO_3 decomposes into B_2O_3 and water, which suppresses the fire. It can be concluded that the prepared paint (P6) can be used as a suitable alternative for the water-based indoor paints.

1. Introduction

With the developing technology and the increase in urbanization, the risk of a fire has been a growing threat. In order to diminish this possible serious risk, fire protection in construction materials has become crucial with the increasing requirements for safety regulations [1]. These regulations make the most urgent need to develop new materials that can provide flame retardancy at high temperatures in the construction industry.

Recent innovations on fire-protection of construction materials cost more in terms of labor, raw materials, and coating, etc. Therefore, in order to lower this burden, the current research has moved towards the development of new products with a sustainable design in construction [2]. The design of a fire-protection of the construction materials for buildings can be performed actively or passively. While the active strategy is composed of mechanical and electrical systems, the passive one refers to limit the spread of flames within the elements of the buildings such as columns, coating, etc. [3].

The paint and coating industry has been changing with technology. This industry is separated into three groups such as architectural coatings, industrial coatings, and maintenance coatings. The architectural coatings include all paints, varnishes, and lacquers [4]. During a fire, to extend the time of interfering with a building, indoor paints have taken attention among other elements [2].

Indoor paints are chemical materials that create protective and decorative thin layers at different surfaces with several application methods [5]. However, most of these paints, composed of halogen- or phosphor-

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*Corresponding author: ndilsiz@gazi.edu.tr based flame retardants in formulations, are inflammable, not ecologically friendly, and release toxic gases and smoke in case of fire [2]. Thus, considerable attention to phosphor-or halogen-free paints should be given to solve these disadvantages [6].

Turkey has the highest boron (B) reserves in the world with 73.4% of the total reserves [7,8]. Many countries that have taken it as a raw material from Turkey transform this raw material into remarkable refined boron products, and then, they sell it back into the global market with higher prices [9]. Therefore, there will always be an increasing need for boron demand [10], and in order to obtain higher economic benefits from these reserves, Turkey should sell refined boron products.

Current interest in flame retardants has focused on developing nontoxic and environmentally-friendly systems as well as preventing further spreading of fire [11]. It is well known that boron compounds like H_3BO_3 and borax are flame retardants and smoke suppressants [6]. During combustion, boron-based flame retardants generate a protective intermediate layer between a surface and a heat source. This impenetrable layer, named char, covers the surface and acts as an oxygen barrier preventing further fire propagation [12]. Moreover, boron compounds have low toxicity compared to toxic halogen-or phosphorus-based flame retardants and are also odorless and colorless [6]. However, there are fewer studies done on boron and its compounds. Xu et al. have found that the addition of 10% of H_3BO_3 as a flame retardant agent into their product increased the value of the limiting oxygen index (LOI) by 33.1% [13]. Uddin et al. have shown that the addition of H_3BO_3 into chitosan films enhanced the fire retarding property when compared to single chitosan films [14]. On the other hand, one of the flame retardants is melamine ($C_3H_6N_6$), which has low cost and is nontoxic during combustion [15].

The aim of this study is to improve the effect of H_3BO_3 on the flame retarding performance of water-based indoor paints. In order to compare its effect, $C_3H_6N_6$ and also calcium carbonate ($CaCO_3$) were utilized in the prepared paints. The physicochemical properties, flammability characteristics and thermal properties were investigated and characterized by Fourier transform infrared spectroscopy (FTIR), limiting oxygen index (LOI), and thermogravimetric analysis (TGA), respectively. The findings of this study contribute to the construction industry as a suitable option to halogen-based paints for the enhancement of the flame retardancy of the water-based indoor paints.

2. Materials and Methods

2.1. Materials

Water-based indoor paints (P1-P6) were prepared with the help of Ortaç Boya Ltd. Sti. (Ankara, Turkey) according to the standards of ISO and the Turkish Standard Institute (TSE). $CaCO_3$, used as filling material in the paint, was obtained from NİDAŞ A.Ş. (Niğde, Turkey) by 99.6% pure and at 3μ of the particle size. H_3BO_3 was kindly supplied from Eti Maden (Ankara, Turkey) as almost 99.9% pure. $C_3H_6N_6$ was purchased from OCI (Netherlands) by 99.8% pure.

2.2. Preparation of the Samples

Figure 1 represents the preparation procedure of the samples. Firstly, in order to prepare P1, 0.005 g of pH regulator, 0.04 g of hardness modifier, 0.015 g of thickener, 0.024 g of the other auxiliary chemicals, and 1.88 g of water were mixed thoroughly in the mixer at 25°C with a speed of 800 rpm for 6 min. Then, 1.18 g of TiO_2, which is used to provide the balance of the white color of the paints for the filling process, was added to the mixer with the addition of 0.1 g of the matting agent and 0.24 g of the stabilizer. After the speed of the mixer was reduced to 700 rpm, the inhibitors of mold/fungus and antifreezing agent were also added, and the solution was mixed for 8 min. In the last step, 1.33 g of the binder and 0.01 g of defoaming agent were added to the mixer whose speed was reduced to 600 rpm and the final mixture was mixed thoroughly for 6 min. This composition of the sample (P1) is the basic formulation, which the compositions and the speed of the mixer in each step were determined by Ortaç Boya Ltd. Sti. with respect to TSE and ISO standarts.

In order to prepare the other samples from P2 to P6, the same procedure was followed at 25°C. However, when the speed of the mixer was reduced to 700 rpm, $CaCO_3$, H_3BO_3 and $C_3H_6N_6$ were added to the mixer. This step was repeated for all the prepared samples at different percentages of H_3BO_3, 5%, 10%, 20%, respectively, and the mixture containing 10% H_3BO_3 and 10% $C_3H_6N_6$. In the last stage, 0.933 g of the binder and 0.01 g of the defoaming agent were again added to the mixer at 25°C with a speed of 600 rpm for 6 min. During the whole process, the temperature was kept constant at 25°C.

The basis of the compositions was determined according to the preliminary studies in Ortaç Boya Ltd. Sti. The value of the compositions of all the other chemicals except $CaCO_3$, H_3BO_3 and $C_3H_6N_6$ were kept constant for all the samples. However, in order to prepare from P2 to P6, the compositions of $CaCO_3$, H_3BO_3 and $C_3H_6N_6$ were changed according to the composition of H_3BO_3 used at different percentages of 5, 10 and 20% wt. In our preliminary stability tests performed at different temperatures (at room temperature and in the oven at 52±1°C), at above 20% wt. of H_3BO_3, the prepared paints showed more viscous nature and started to dry much fast. Therefore, the highest value of H_3BO_3 was chosen as 20% wt. Table 1 shows the

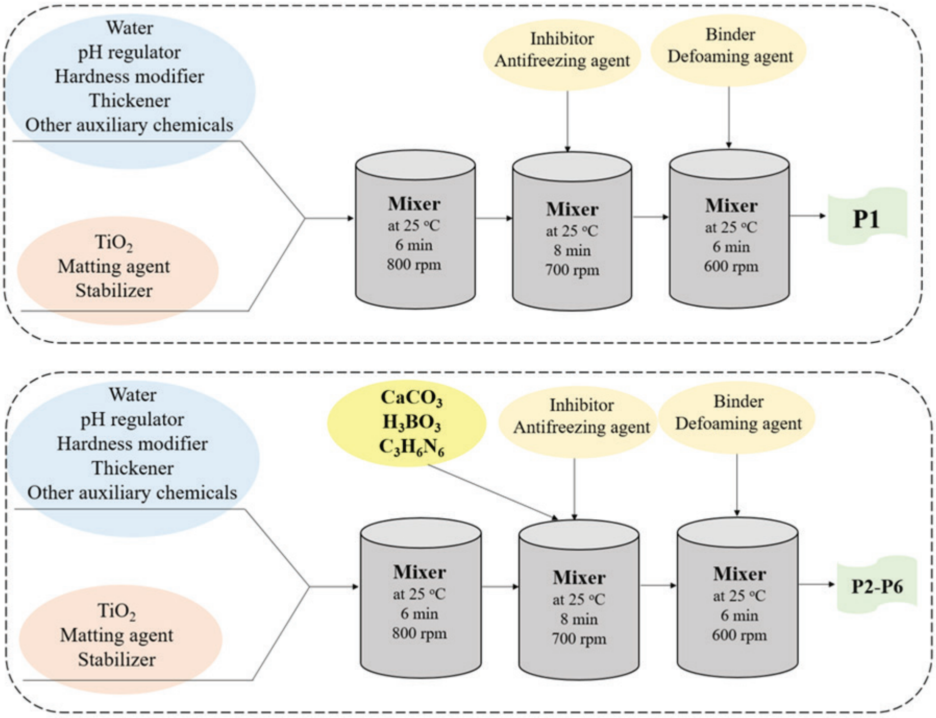

Figure 1. The preparation procedure of the samples.

compositions of each prepared sample, which gives 100% for each sample.

2.3. Characterization of the Samples

The mixing process of the paint and the additives were performed by Sozer/SM 1-130 machines (Sozer Makina, Kocaeli, Turkey), and then, the prepared paints were kept at KP-EY11 boxes for storage.

The chemical bonding of the samples was characterized by a Fourier-transform infrared spectroscopy (FTIR, Nicolet Avatar 370; Thermo Fisher Scientific, Inc., Waltham, MA, USA) in the range of 4000-400 cm^{-1} at room temperature.

Limiting oxygen index (LOI) measurements were performed using a Dynisco Polymer Test Limiting Oxygen Index Chamber (Dynisco Europe, Heilbronn, Germany) according to ASTM D 2863-19. In the test, each sample was dried and placed in templates of 10 cm x 1 cm x 0.4 cm according to TS EN ISO 4589-2 for three days at 35°C, and then gas flow was adjusted to confirm the standard test technique.

The thermal characterization of the samples was determined by thermogravimetric analysis (TGA, Perkin Elmer, Inc., Simultaneous Thermal Analyzer STA 6000, Waltham, MA, USA) in the temperature range of 25-800°C under nitrogen atmosphere at a heating rate of 10°C min^{-1}.

Table 1. The compositions of each prepared sample.

Composition (weight ratio %)	Sample Code					
	P1	P2	P3	P4	P5	P6
Water	38.5	17.6	17.6	17.6	17.6	17.6
TiO_2	23.6	15.7	15.7	15.7	15.7	15.7
Binder	26.6	19.0	19.0	19.0	19.0	19.0
Defoaming agent	0.2	0.2	0.2	0.2	0.2	0.2
Other auxiliary chemicals	11.1	11.1	11.1	11.1	11.1	11.1
$CaCO_3$	-	36.4	31.4	26.4	16.4	16.4
H_3BO_3	-	-	5.0	10.0	10.0	20.0
$C_3H_6N_6$	-	-	-	-	10.0	-

3. Results and Discussion

3.1. Characterization of the Samples

The chemical bonding of the prepared paints was characterized by FTIR from 4000 to 400 cm^{-1} in wavenumber in Figure 2. The FTIR spectra of pure paint (P1) without any additives or filling materials revealed that O-H stretching, C-H stretching vibrations, C=O stretching, C-H bending, C(=O)-O asymmetric stretching vibration was at about 3700 cm^{-1}, between 2900 and 2800 cm^{-1}, between 1800 and 1700 cm^{-1}, about 1400 cm^{-1}, between 1270 and 1200 cm^{-1}, respectively [16]. In case of P1, the stretching vibration peak at 3500-4000 cm^{-1}, which ascribed to OH bonds, likely owing to the absorption of moisture from the atmosphere, was more intense than the other paints. A broad and a sharp absorption bands owing to asymmetric stretching band of CO_3^{2-} between 1400-1500 cm^{-1} and at 877 cm^{-1}, respectively, were the specific peaks of $CaCO_3$ that was added in all the prepared samples [17]. The intensity of this peak was decreased with decreasing the addition amount of $CaCO_3$ in the prepared paints from P2 to P6. On the other hand, in the FTIR spectra of H_3BO_3, the broad band at 3200 cm^{-1} and the narrow band at 1190 cm^{-1} were referred to the absorption of B-OH; the band at about 1400 cm^{-1} was assigned to B-O vibration absorption [6,18]. In Figure 2, FTIR spectra of the samples containing only H_3BO_3 additive among with the sample without any additives or filling materials are shown. As seen in Figure 2, the intensity of characteristic H_3BO_3 peaks increased by increasing the amount of H_3BO_3 in the paints prepared.

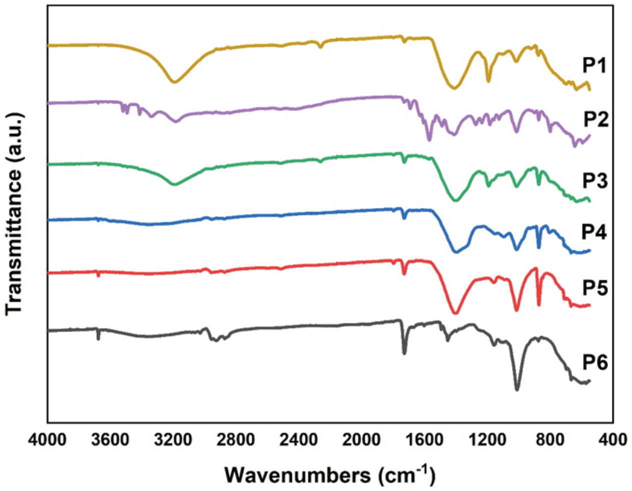

Figure 2. FT-IR spectra of the prepared samples (from P1 to P6).

3.2. LOI Test

Limiting Oxygen Index (LOI) is the most primary, fast and effective technique for investigating the flammability characteristics of the prepared samples [19]. The instrument of the limiting oxygen chamber determines the minimum oxygen requirement to burn these samples, and the results are displayed in Figure 3. The LOI value of P1, which contains no additives and no filling materials, was measured and found to be 24%, and it had the lowest value. When the filling material, $CaCO_3$, was only added to the pure paint, the LOI value of P2 increased and reached an LOI value of 27%. However, when the LOI value of the samples is lower than 28%, these samples are defined as flammable [19]. Thus, the sample P2 was found as flammable. On the other hand, besides $CaCO_3$, with the addition of 5% of H_3BO_3, the LOI value of P3 increased to 30%. In addition, with the increasing amount of H_3BO_3 from 5% to 10%, the LOI value of P4 rose from 30% to 33%. With the addition of 10% of H_3BO_3 and 10% of $C_3H_6N_6$, the LOI value of P5 demonstrated a moderate increase to 39%. Yildirim and Celik examined the flame retardant properties of their prepared composite coatings reinforced with H_3BO_3 according to the pure paint. They reported that whereas the combustion reaction was observed in the coating containing 1% of H_3BO_3, the other coatings including 3%, 5%, 10%, and 15% of H_3BO_3 were not shown any combustion reactions [20]. Furthermore, Figure 3 obviously represents that the LOI value of P6 displayed a surprising boom from 39% to 55%, which contains 20% of H_3BO_3. It can be concluded from the data that P6 is a highly effective flame retardant and can provide excellent flame retardancy to the water-based indoor paints at 20% of H_3BO_3 content. These findings are consistent with the literature where the fire resistance increased when H_3BO_3 is used as a fire retardant material [11,21].

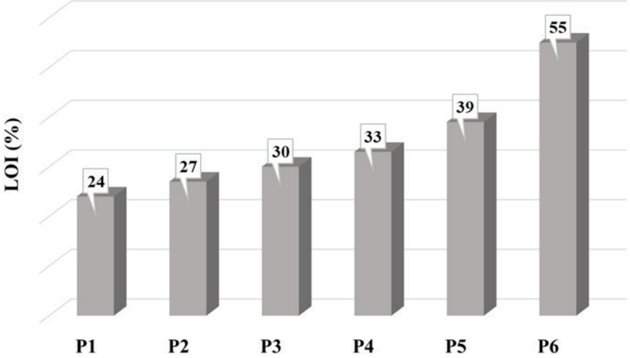

Figure 3. The results of LOI of the prepared paints (from P1 to P6).

3.3. Thermal Analysis

The thermal analysis of all the prepared samples was examined by the thermogravimetric analysis system. Figure 4 shows the TGA curve of P1, which contains no additives and no filling materials, and P2, which contains only $CaCO_3$ as a filling material.

As shown in Figure 4, the first prepared sample, P1, showed about 30% weight loss below 403.7°C; however, P2 sample showed three decomposition stages:

a weight loss of 2% at about 116.1°C by the evaporation of water in the matter, 15% at 400.7°C by losing volatile components in the paint, and a large weight loss of about 40% at about 772.6°C indicating the decomposition of $CaCO_3$ to CaO and CO_2. This was defined by Deepika et al. [22].

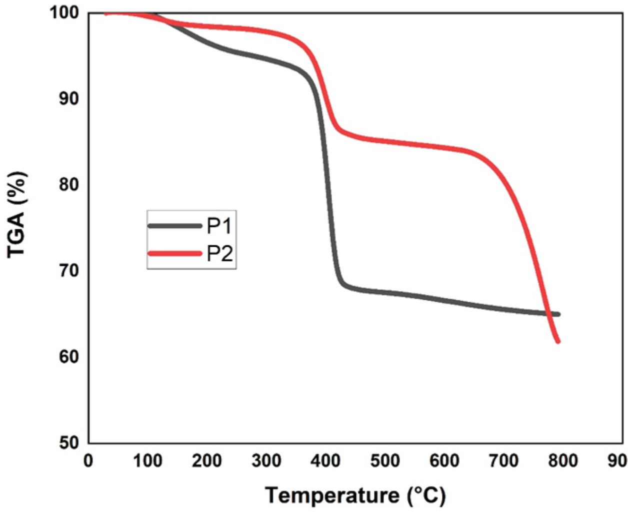

Figure 4. The thermal decomposition patterns for P1 and P2.

Figure 5 shows the TGA curve of the prepared samples from water-based indoor paints that contain different amounts of H_3BO_3 as an additive (P3, P4 and P6). There were three mass-losses in TGA profiles of all the prepared paints. However, the decomposition temperatures and the amounts of weight loss were different due to the different amounts of H_3BO_3 in the prepared paints. When compared to the thermal decomposition patterns of P3, P4 and P6, the first decomposition temperature was obtained as almost 1% at about 155.4°C for P3; however, the first weight losses of P4 and P5 were found at 116.2 and 112.5°C as 2% and 10%, respectively. This slightly weight reduction at an early stage was attributed to the desorption of physically bound water molecules [23]. Then, a sharp decrease in weight loss of P3, P4 and P6 were about 15%, 20% and 30% between 300-400°C, respectively due to the thermal decomposition of the auxiliary chemicals [22]. In addition, when the heating temperature was higher than 720°C, the TGA curve of all the samples demonstrated a final third weight loss due to the thermal decomposition of $CaCO_3$. It can be clearly seen from Figure 5 that the weight loss of the samples increased directly as the amount of H_3BO_3 increased. It can be explained that there might be the conversion reaction of H_3BO_3 to HBO_2 and then into boron oxide (B_2O_3). This result was confirmed by the study carried out by Uner and coworkers [24]. They reported that the H_3BO_3 is transformed into HBO_2 when there is due to dehydration happened below 150°C. Then, metaH_3BO_3 is transformed into B_2O_3 after all water is removed. Whereas crystalline boron oxide melts at a specific temperature (at 450°C), amorphous boron oxide does not, but only softens at 325°C [24]. Therefore, the peaks at 300-400°C may be defined as the transition of crystalline boron oxide to amorphous B_2O_3 [25].

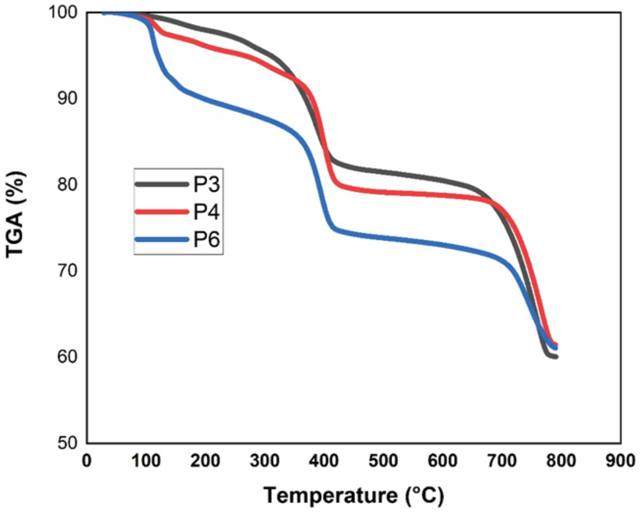

Figure 5. The thermal decomposition patterns for P3, P4 and P6.

On the other hand, Figure 6 illustrates the TGA mass-loss profiles of the prepared samples with H_3BO_3 (P4 and P6), and $H_3BO_3/C_3H_6N_6$ (P5). It can be seen that above 400°C, decomposition of $C_3H_6N_6$ occurs and releases ammonium and water, which then sublimes or decays [26]. Furthermore, the study of Ullah et al. [27] showed that the addition of $C_3H_6N_6$ increased the expansion of a char layer up to 1100% at 375°C while the addition of H_3BO_3 increased to 300%. Therefore, the addition of both additives (H_3BO_3 and $C_3H_6N_6$) as flame retardancy increased the decomposition stages of the samples. This result confirms that $C_3H_6N_6$ has the positive effect on the property of flame retardancy of indoor paints.

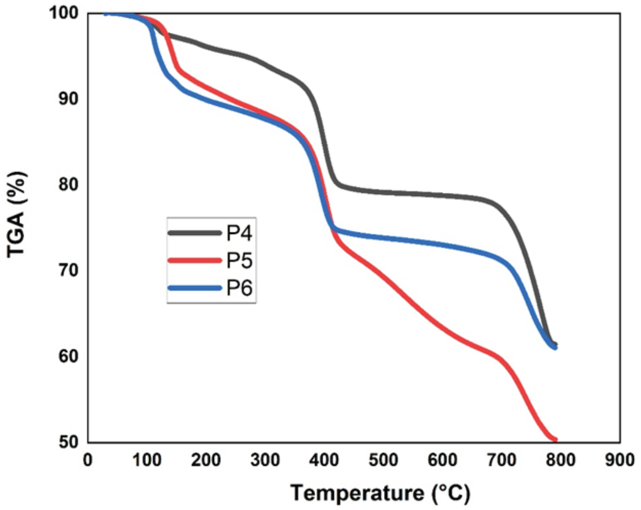

Figure 6. The thermal decomposition patterns for P4, P5 and P6.

Table 2. The maximum decomposition temperatures of each prepared sample.

Sample Code	Decomposition Temperature (°C)			
	Stage 1	Stage 2	Stage 3	Stage 4
P1	192.5	403.7	-	-
P2	116.1	400.7	772.6	-
P3	155.4	273.8	389.4	760.5
P4	116.2	304.3	400.5	764.8
P5	142.7	401.6	513.8	730.9
P6	112.5	147.2	394.6	739.0

4. Conclusions

The indoor paint samples containing $CaCO_3$, $C_3H_6N_6$, and H_3BO_3 were successfully prepared, and the characterization of the prepared paints was performed by FTIR. The addition of these potential flame retardant additives in the paints resulted in an enhancement in the flammability characteristic and thermal property characterized by LOI test and TGA, respectively. The characteristic peaks of $CaCO_3$, $C_3H_6N_6$, and H_3BO_3 were observed in the FTIR spectra of the prepared paints. When H_3BO_3 was added at 20% (w/w) weight fraction, the prepared paint showed more significant enhancement in flammability behavior from 24% to over 55% than that of containing $C_3H_6N_6$ and H_3BO_3. In addition, in the thermogravimetric analysis, the prepared paints containing H_3BO_3 decomposes into boron oxide and water, which suppresses the fire. Based on these results, it can be said that the prepared paint, P6, that contains 20% of H_3BO_3, can be used as a suitable alternative in the water-based indoor paints.

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