Effects of Fire Retardants on the Combustion Behavior of High-Density Fiberboard

Ferhat Özdemir* and Ahmet Tutuş

The objective of this study was to determine the effects of certain fire-retardant chemicals on the combustion resistance of high-density fiberboard (HDF). Borax (BX), boric acid (BA), ammonium polyphosphate (APP), and alpha-x (AX) were added into the fibers made from 50% scots pine (*Pinus sylvestris* L.) and 50% beech (*Fagus orientalis* Lipsky) woods at 3%, 6%, and 9% levels based on oven-dry fiber weight. HDF panels were produced in 6.5 mm thickness. HDF panels' combustion behavior was explored. To detect combustion resistance, the panels were tested according to the ASTM-E 69 and thermogravimetric analysis (TGA) tests. It was determined that fire-retardant (FR) chemicals enhanced the combustion resistance of the panels to varying degrees. As a result, the FR chemicals' type and concentrations are effective for determining the combustion resistance of HDF panels.

Keywords: High-density fiberboard; Borax; Boric acid; Combustion,

Contact information: Department of Forest Industry Engineering, Faculty of Forestry, Kahramanmaras Sutcu Imam University, Kahramanmaras 46060, Turkey;
**Corresponding author: ferhatozd@hotmail.com*

INTRODUCTION

Wood components are degraded as hemicelluloses, cellulose, and lignin (Tutus *et al.* 2010). Wood and wood-based materials are composed of carbon and hydrogen components; therefore, when the temperature reaches about 275^{o}C, combustion behavior is observed (Hakkarainen *et al.* 2005). Between 500^{o}C and 800^{o}C, carbonization takes place. Wood combustion can be life threatening when it affects habitable structures and their wood-based contents. In order to reduce flammability and provide safety, wood can be treated with fire-retardant chemicals (Baysal *et al*. 2007).

In wood and cellulosic materials, fire-retardant chemicals are frequently used to stop combustion (Lyons 1970). Meanwhile, it has been recommended that boron-based products should be used individually or jointly due to their smoke-suppressor features (Le Van and Winandy 1990). Fire-retardant chemicals used for wood products typically contain phosphorus and nitrogen (Le Van and Winandy 1990; Ayrılmış 2005).

Boric acid and borax are largely used in the wood protection industry (Baysal 1994), as they diminish the flames' spreading on wood's surface once exposed to extreme heat; they also have a low melting point and form a glassy film layer on the surface (Nussbaum 1988). Borax eliminates the spread of flames, while boric acid enhances carbonization, which is why they should be mixed when used (Yalınkılıç *et al.* 1996; LeVan *et al.* 1990; Baysal 2002).

Ammonium polyphosphate creates a carbon layer effect on the combustion behavior. Such a layer blocks the access of oxygen and heat, which inhibits combustion and reduces the amount of smoke. Fire-retardant chemicals do not completely stop combustion of wood, but they create a fire-retardant effect.

When fire-retardant (FR) chemicals are applied to wood, they retard combustion by releasing phosphoric acid esters, wood polysaccharides, and water. These releases influence the dehydration reactions of wood (Grexa *et al.* 1999). In wood and wood-based materials, boron components are preferred due to their thermal resistant features (Tsunoda *et al.* 2002). For this reason, predicting the models for thermal degradation of untreated and treated wood with FR chemicals is very important.

The main objective of this study was to determine the optimum chemical type and concentration against combustion resistance of structural HDF panels treated with selected fire retardants.

MATERIALS AND METHODS

Raw Materials

Wood fibers (a 50:50 blend) consisting of pine and beech species were obtained from a commercial MDF plant, SFC Integrated Wood Company, in Gebze, Turkey. The moisture content of the fibers, as determined by oven-dry weight, was found to be 3% to 4% prior to treatment. Four fire retardants used in the treatments were BX, BA, APP, and a commercial product known as alpha-x (density: 0.84 g cm-^{3}, pH: 3.74, boiling point: 380^{o}C) obtained from Ozen Chemical Company, Istanbul, Turkey. The powdered chemicals were then added to the blender at target contents of 3%, 6%, and 9% based on the oven-dry fiber weight. A commercial liquid UF resin, with a 55% solid content and a specific gravity of 1.23 was used. All the chemicals were determined in proportion to the oven-dry fiber weight.

Panel Manufacturing

Fire retardants – BX, BA, APP, and AX chemicals – were added homogeneously in ratios of 3%, 6%, and 9% within glued fibers, and then they were mixed. Density profiles of HDF panels produced using 400 x 400 x 150 mm size cold-pressing were checked via GreCon™ device. The three panels for each group were pressed under 3.5 MPa pressure applied over the panels in a Cemil Usta SSP 125 press machine. Thickness was set at 8 mm by using thickness wedges. Using rubbing, 980-1000 kg/m^3 density HDF panels were cut to 6.5±2 mm thick. Production conditions and ingredients for the HDF panels are displayed in Table 1 and Table 2, respectively.

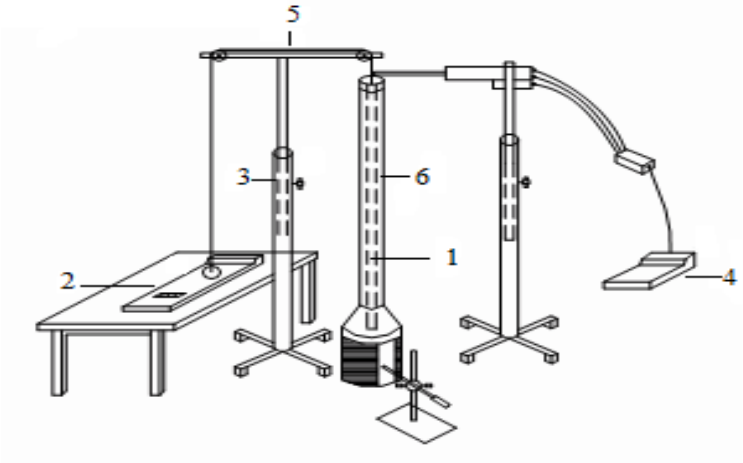
Table 1. Hot Pressing Parameters for HDF Production

Parameters	HDF
Temperature (^{o}C)	183
Press (MPa)	3.5
Time (s)	18

Fire-Resistance Test Method (ASTM-E 69)

In order to detect weight loss in the test samples, according to ASTM E 69, the combustion test method was implemented. Control and test samples were acclimatized prior to the combustion procedure under 20±2^{o}C temperature and 65±5% relative humidity conditions (Sweet and Winandy 1996). Weight loss was measured using a digital scale every 30 seconds. Flame source combustion (4 min) and without flame source combus-tion (6 min) values were detected. The experiment was finished after 10

min. The FS and WFS weight losses were noted. For each test group, tests were repeated 6 times. The combustion test apparatus is exhibited in Fig. 1.



1 – Combusion test specimen
2 – Electonic scales
3 – Support frame
4 – Gas analyzer
5 – Wire
6 – Fire tube

Fig. 1. Combustion test apparatus

Thermogravimetric Analysis

From each panel group, small samples were taken and ground into flour-form using a Wiley mill. Flour were screened to 100-mesh-size and thermogravimetric analysis was performed using a Perkin Elmer thermogravimetric analyzer. The amount of each sample was 8 mg to 10 mg. The sample was heated under a stream of dry N_2 gas with a flow rate of 100 mL min^{-1} at a temperature ranging from 20°C to 700°C with a heating rate of 10°C min^{-1}. Weight losses occurred while heating high-density fiberboard samples, for which thermal characteristics were analyzed and detected via a computer program. Three repetitions were implemented for each test group.

Interfacial Morphologic Analysis

The interface of test specimens was observed using a scanning electronic microscope (SEM, Jeol-5600) under an acceleration voltage of 10 kV. Each specimen was exposed to a vacuum prior to scanning. Particle size and dispersion of the chemicals between the wood strands were viewed.

Statistical Analysis

The SPSS statistical package program was used. Data from the fire test were analyzed using a computerized statistical program to perform an analysis of variance (ANOVA) and by carrying out the Duncan test at a $P \leq 0.05$ confidence level. Homogeneous groups with small letters were indicated with a superscript.

RESULTS AND DISCUSSION

Combustion Weight-Loss Values

In Table 2, results of combustion with flame source (FS) and without flame source (WFS) are presented, with weight-loss amounts of the HDF panel test samples treated with FR chemicals. The smallest weight loss was observed at a 9% concentration of FS in BX (22.4%) chemical. In the case of WFS the smallest value was observed when using the APP chemical (47.3%).

Table 2. Average Weight Loss Values of High-Density Fiberboards Obtained Using Some Fire Retardant Chemical Substance

Group	Chemicals	Concentration (%)	FS Weight Loss (%) (Mean ±SD)	WFS Weight Loss (%) (Mean ±SD)
1	CONTROL	0	42.6 ± 3.5^{a}	84.3 ± 3.4^{a}
2		3	27.8 ± 3.6^{cd}	81.4 ± 4.3^{ab}
3	BX	6	25.6 ± 1.7^{def}	71.6 ± 4.6^{e}
4		9	22.4 ± 1.8^{g}	66.5 ± 1.7^{fg}
5		3	34.5 ± 1.7^{b}	82.6 ± 4.9^{ab}
6	BA	6	29.0 ± 1.0^{c}	76.2 ± 3.9^{cd}
7		9	23.6 ± 1.8^{efg}	66.2 ± 3.3^{fg}
8		3	26.5 ± 2.6^{cde}	72.8 ± 3.5^{de}
9	APP	6	24.2 ± 3.5^{efg}	63.8 ± 4.2^{g}
10		9	22.9 ± 2.2^{fg}	47.3 ± 3.2^{h}
11		3	34.0 ± 2.4^{b}	79.9 ± 1.9^{bc}
12	ALPHA–X	6	28.6 ± 1.5^{c}	76.4 ± 1.4^{cd}
13		9	24.4 ± 1.2^{efg}	70.2 ± 2.1^{ef}

FS: Flame source; WFS: Without flame source
Homogeneity groups with small letters are given as superscript from highest to lowest in the order of letters (a-h) and indicate significant difference by Duncan's mean separation test.
SD: Standard Deviation

A – Weight-Loss Values of Flame Source Combustion

In the first 4 min, the first stage of the experiment, combustion in all the samples occurred at different proportions. The results obtained from FS revealed that test panels treated with chemicals provided better results in comparison to the control sample. With respect to the 3% concentration ratio of all chemicals, the 9% concentration ratio provided further protection. In regard to mass loss, when compared to the control sample, the best effect was obtained with the 9% concentration. The best protection was provided from the samples that included BX (22.4%), APP (22.9%), BA (23.6%), and alpha-x (24.4%) chemicals. Similar results were reported by Ustaömer (2008) for medium-density fiberboard panels. Yalınkılıç *et al*. (1998) stated that the decrease of the weight loss of Douglas fir specimens treated with a Bx+Ba mixture increased at the end of combustion.

Fire-retardant chemicals decreased the pyrolysis temperature and, by increasing carbonization, diminished weight loss. Due to the increased carbonization, the emission of flammable gases was lessened (Wang *et al.* 2005).

In Fig. 2, different chemicals used to enhance fire resistance and the effect of varying concentrations on the weight-loss values of test panels (%) are comparatively exhibited.

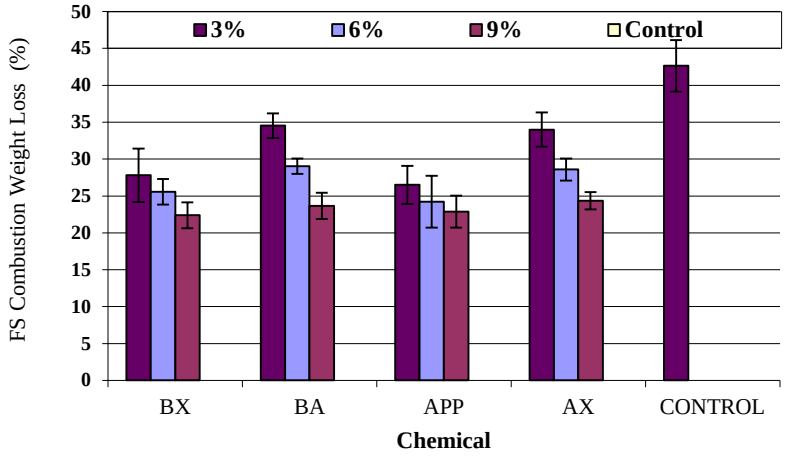


Fig. 2. Average weight loss values in FS

B – Weight-Loss Values of Without Flame Source Combustion

The highest mass loss was obtained from the control specimens (84.3%). The lowest value occurred when using the APP (47.3%) chemical and the second stage of combustion after the movement of flame source from the fire tube. Higher concentrations enhanced the effects of the chemicals. Compared to the control sample, weight losses decreased. In the WFS samples at the 9% concentration, relative to the control samples, the added fire-resistance chemicals (APP 47.3%, BA 66.2%, BX 66.5%, and AX 70.2%) were found to be effective. The effects on WFS weight-loss values of different types and concentrations of the FR chemicals are shown in Fig. 3.

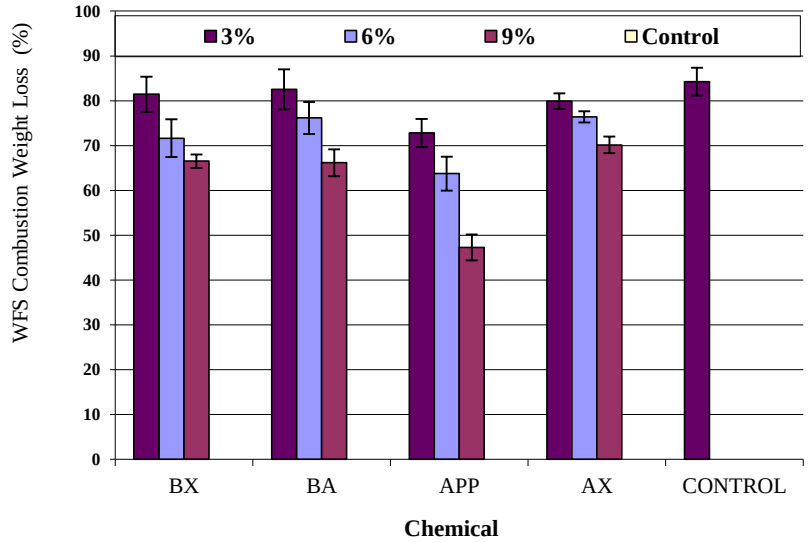


Fig. 3. Average weight-loss values in FS

Due to the fire source, in the first 4 min, at the first stage of the combustion test, an increase in weight loss occurred in all the samples at nearly the same time. The weight loss continued to slowly decrease when the fire source was moved away from the fire tube, but burning velocity fell. The highest mass reduction (84.3%) was observed in control specimens during the second stage of combustion after the movement of the flame source from the fire tube. Based on comparisons with the control samples, it can be said that impregnated chemicals exhibited effectiveness as fire retardants. Similar results have been found in previous studies (Yapıcı *et al.* 2011; Kurt and Uysal 2009).

At the FS stage of the combustion, an increase occurred in weight loss due to the flame source, and a decrease occurred as a result of the flame source's being moved far away from the fire tube.

The Duncan test was applied in order to determine which of the differences from variance analysis were significant ($P \leq 0.05$), and the results were shown as different homogenous groups in Table 2.

Thermogravimetric Analysis

The effects of fire-retardant chemicals on the combustion resistance of HDF panels are depicted in Fig. 4.

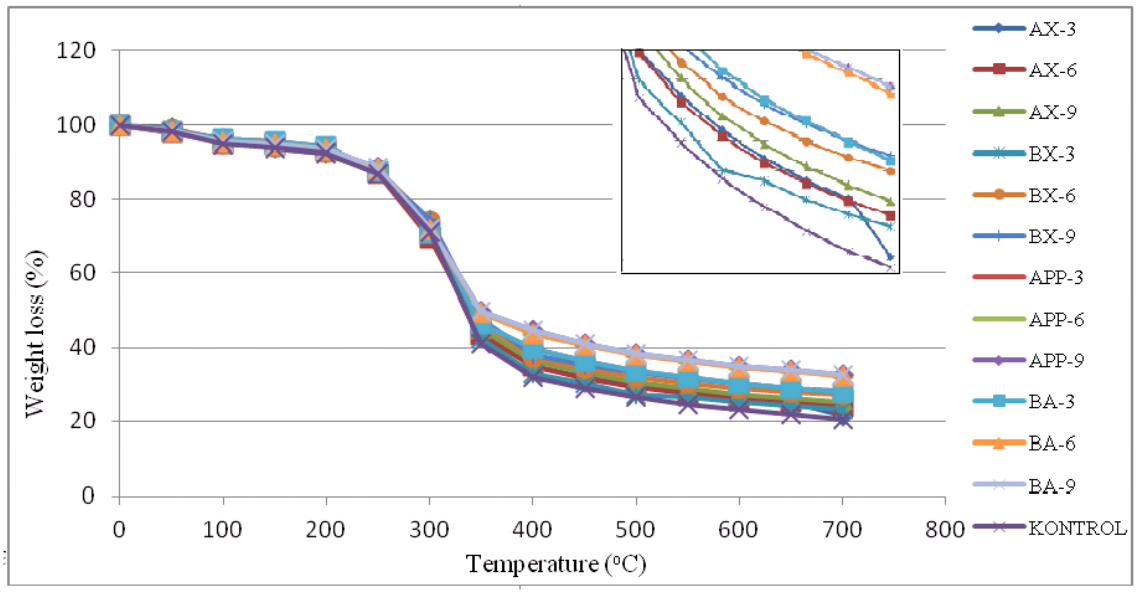


Fig. 4. TGA weight-loss values of HDF specimens treated with FR chemicals

The correlation between temperature rise of HDF test panels through the TGA method and emerging loss of weight was analyzed. It was found that under 700°C, compared to the control samples, all FR chemicals showed a positive effect. In FR-added panels, compared to the control sample, the temperature increase caused smaller weight losses.

The observed reductions in losses are related to the chemical's enhanced effect on the fire resistance of the panel and positive effect against combustion. Sun *et al.* (2012) reported that the treated MDF with FR chemicals showed a lower weight loss than the untreated control specimens. Thermal degradation temperatures and the weight loss of fire-retardant chemicals are given in Table 3.

Table 3. Amount of Total Weight Loss at Thermal Degradation Temperatures

Fire retardant chemicals	% wt	Initial temperature of thermal degradation (^{0}C)	Moisture content (%)	End temperature of thermal degradation (^{0}C)	Thermal degradation weight loss (%)	Total weight loss (%)
CONTROL	0	283.3	7.9	361.0	68.0	79.0^h
BX	3	282.1	7.0	359.4	64.7	76.2^g
	6	287.6	8.6	361.6	61.6	72.7^d
	9	289.3	9.3	361.2	60.3	71.2^c
BA	3	281.7	7.9	362.9	63.5	75.9^g
	6	292.5	10.6	348.3	53.1	69.4^b
	9	291.4	9.9	348.3	52.3	67.0^a
APP	3	260.8	5.6	344.9	56.3	71.0^c
	6	262.1	6.2	342.3	52.4	67.5^a
	9	266.6	6.4	336.2	50.6	67.1^a
AX	3	274.6	6.5	361.0	63.0	75.2^f
	6	254.3	6.0	358.5	62.2	75.1^f
	9	258.0	5.6	361.4	60.6	73.9^e

At less than 700^{o}C, the greatest mass loss was observed in the control sample (79.0%). Among all FR chemicals, in the 9% concentration, the most effective ones detected were BA (67.0%), APP (67.1%), BX (71.2%), and AX (73.9%).

In relation to the increase in the ratios of FR chemicals, a decrease in weight loss occurred. FR chemicals increase carbonization at relatively low temperatures and boost thermal isolation (Kollman and Cote 1968).

In comparison to the control sample, in the 9% concentrations, BA (15.19%), APP (15.06%), BX (9.87%), and AX (6.46%) had a positive effect on greater TGA weight loss.

A relationship was identified between the combustion test and TGA. The combustion test is conducted in the presence of oxygen, but TGA is conducted in nitrogen without oxygen. The proportion of oxygen in air is normally 21%. Thus, it was shown that the treated samples had burned poorly because of the decrease in the oxygen ratio. Impregnation chemicals were seen to be effective as fire retardants. Therefore, as the amount of FR chemicals increase, the combustion ratio decreases.

The experimental results were evaluated with ANOVA and Duncan test ($P \leq 0.05$). The test results revealed significant differences among the groups. Different homogeneity groups are indicated with small letters shown as superscript letters from lowest to highest in alphabetical order (a-h). The results indicate significant differences based on the Duncan's mean separation test.

Morphological Observation

FR chemicals were used in particle sizes ranging from 35 to 100 mesh. The small particle size of chemicals positively affected homogeneity dispersion. The samples were examined using a scanning electron microscope. The distribution of fire-retardant chemicals sprinkled into the fibers is shown in Fig. 5.

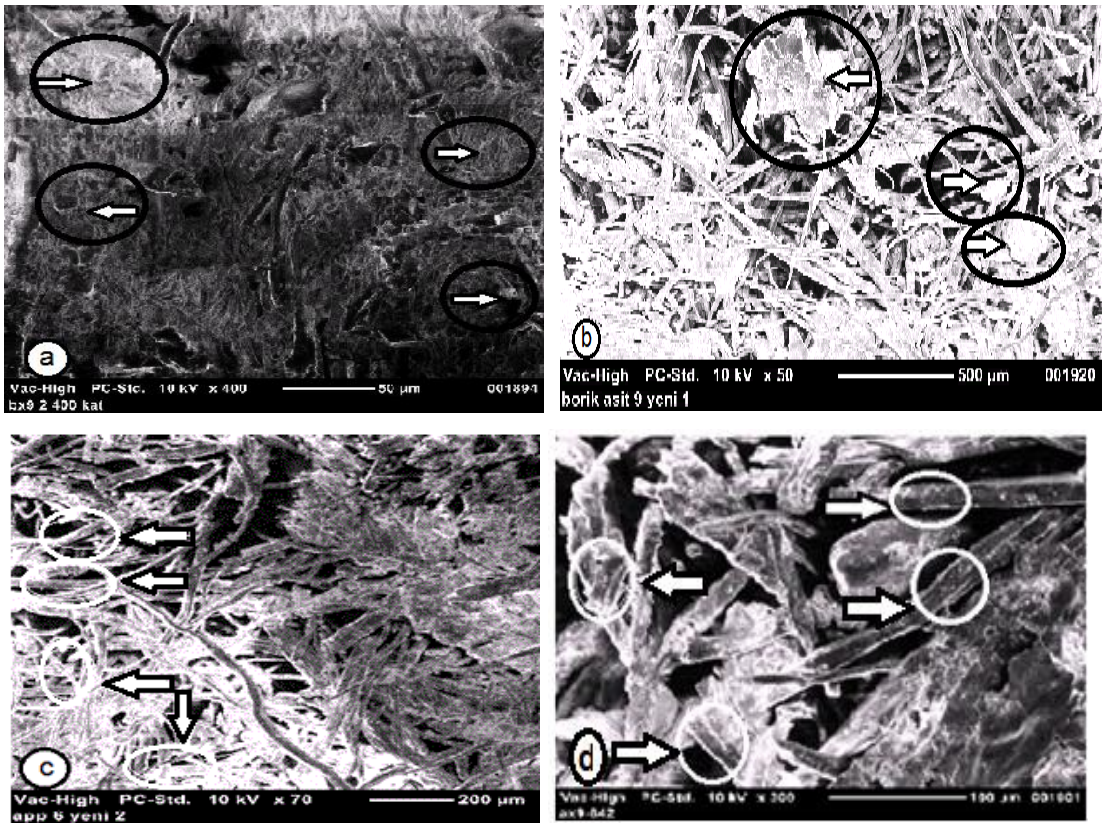


Fig. 5. SEM micrographs of BX(a), BA(b), APP(c), and AX(d) fire-retardant chemicals added into the fibers

CONCLUSIONS

1. According to the results, fire-retardant (FR) chemicals have a positive effect on combustion resistance and provide a certain amount of protection against combustion.
2. In relation to the rise in the concentration of FR chemicals, high-density fiberboard (HDF) panels were further protected against combustion, and there was a decrease in weight loss. In flame source (FS) tests, the best protection was obtained using borax (BX) treatment. In tests without flame source (WFS), the best protection was obtained using ammonium polyphosphate (APP).
3. The results of thermogravimetric analysis (TGA) revealed that weight losses when using FR chemicals were lower than the control sample. Increasing the concentration ratio resulted in a decrease in the combustion amount. Boric acid (9%) and APP (6%, 9%) were identified as the most effective chemicals.
4. FR chemicals affected the combustion mechanism. By causing carbonization upon exposure to high temperature, they acted as isolation materials, diminished the emission of flammable gases, and increased combustion resistance against high temperatures. In comparison to the control sample, there was a smaller weight loss when using FR chemicals at a sufficiently high concentration ratio.

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