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ORIGINAL

Evaluation and fireproofing treatment of wooden heat-insulating/acoustic absorbing materials

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Abstract Heat insulating and acoustic absorbing materials were endued with fireproof performance and subjected to quantitative evaluations assuming a variety of uses and situations. Fireproofing was performed by spraying an aqueous solution of flame retardants on raw materials (wood shavings, kenaf bast fibers, and core-sheath-type fibers) during the fiber spreading and mixing step. It was possible to treat the materials homogeneously and without increasing manufacturing workload. Flame retardancy was evaluated using vertical flame testing, horizontal flame testing, the 45° Meckel's Burner method, and a cone calorimetry method in accordance with UL94, JIS D 1201, JIS L 1091, and JIS A 9521, respectively. It was possible to achieve results that satisfied each of these standards, along with quantifying the relations between treatment conditions and performance.

1 Introduction

Industrial materials distinguished by their low cost, such as glass wool, are used heavily in a wide range of industries, including construction, household equipment, and automobile industries. Due to enhanced environmental consciousness and changing awareness of the use of wooden resources, wooden heat insulation and acoustic absorbing materials have however in recent years also seen distribution in the marketplace. One reason for this distribution in Japan is thought to be the enactment of laws that promote the utilization of wood in public buildings.

In response to this demand, wooden heat insulation and acoustic absorbing materials have been developed using wood flakes (wood shavings) produced by an auto-planer as the primary raw material (Fukuta et al. 2010b, 2012). For heat insulation materials whose primary use is assumed to be in residential housing, Japan stipulates no specific performance provisions related to flame retardance performance per se; rather, the fire resistance of housing is evaluated by means of flame exposure testing of the wall body surface. In this respect, fire-resistance testing of wall bodies that utilize wood chips as heat insulation material has shown fire resistance performance superior to glass wool, achieved by the evaporative latent heat of water which wood contains controlling temperature increases, and by the maintenance of packing properties (Taniuchi et al. 2008). As a variety of situations can be envisaged to affect the inside of the wall body both at the time of construction and afterwards, heat insulation and acoustic absorbing materials with consistent flame retardance (fireproof) performance are however preferable and in great demand. Consistent fireproof properties will also be required regarding the likely wide expansion in the use of these types of materials in the future.

With respect to the flame retardant treatment of wood, numerous reports concerning flame retardant substances and methods for their treatment as well as evaluations of their performance are available. In a report concerning flame retardant treatment of particle board, Pedieu et al. (2012) discussed recent research examining wood and wood-based materials. Although there are studies such as the report by Grohe (2003) who investigated low-density

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wooden materials such as heat insulation materials, there are however comparatively few. This may be because the manufacturing processes of the materials and the evaluation methods of their flame retardance performance have not yet been generalized.

In the present study, fireproofing treatments on wooden heat insulation and acoustic absorbing materials developed by the authors were performed and quantitative evaluations were also conducted that took into account a variety of uses in order to determine appropriate treatment conditions. The fireproofing treatment used in the present study was found not to increase the manufacturing workload of the materials, and to impart fireproofness in a simple and efficient manner.

2 Materials and methods

2.1 Wooden heat insulation and acoustic absorbing materials

As previously reported (Fukuta et al. 2010b), the manufacturing method for heat insulation and acoustic absorbing materials developed by the authors uses wood shavings and kenaf bast fibers as well as core-sheath-type fibers (binder component) as raw materials. The wood shavings used as primary raw materials were produced from air-dried Japanese cedar (*Cryptomeria japonica*) of 0.35–0.40 g/cm^3 density using an auto-planer. The wood shavings were slightly curled flakes with a maximal local thickness of ~0.2 mm and a width and length of 5–20 mm. Although variability was large, all flakes were used without screening. Kenaf bast fibers 80 mm in length were used that had been roughly spread from kenaf bast. The core-sheath-type fibers were synthetic fibers composed of two layers of resin with differing qualities. ESC (ES Fibervisions) fibers were used, comprising a polypropylene core and polyethylene sheath, with a length of 5 mm and a fineness of 2.2 dtex. The decitex unit (dtex) is an international standard that shows the thickness of fiber, where 1 dtex is 1 g per 10 km of fiber. For the fiber material spreading and mixing of these heterogeneous raw materials, an air blower equipped with a chamber was used in which the raw materials could be suspended simultaneously. To this end a dust collector generally utilized in e.g. wood processing plants was modified (Fig. 1). It sucks up raw materials through a separate dust-collecting inlet and circulates it inside the machine. Its structure allows re-introduction of material from the dust collecting chamber into the air blower. Core-sheath and kenaf fibers were first circulated until the fibers separated sufficiently, then the wood shavings were sucked up and all the raw materials mixed. The fusion capability of the core-sheath fibers was employed for mat thermoforming. By heating the fibers to a temperature at which only the polyethylene sheaths melt, the wood shavings and kenaf fibers can be integrated at the same time as the core-sheath fibers fuse together. The thickness of the mixed raw materials was controlled by placing them into a mold of inner dimensions 310 × 310 mm^2 in a quantity corresponding to the target density and thickness. Although it is efficient to utilize a heat-through dryer in thermoforming, this process was carried out by heating the mixture, mold by mold, in a general oven dryer at 160 °C for 2 h. Mixtures were then left to cool to room

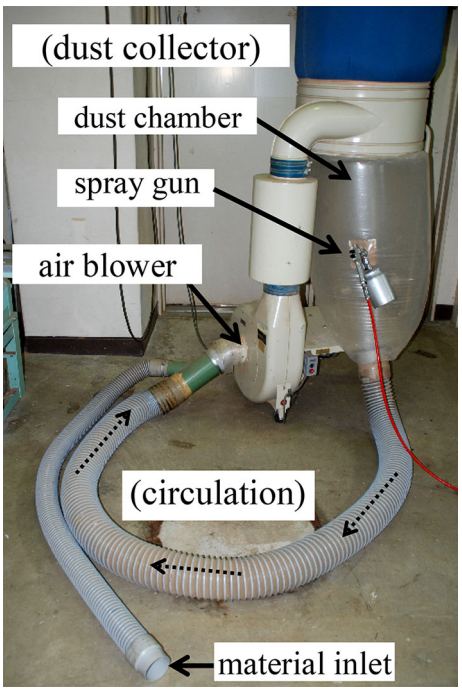

Fig. 1 Raw material fiber spreading/mixing and fireproofing treatment technique

Fig. 2 Wooden heat insulation and acoustic absorbing material

temperature and taken out of the molds. Figure 2 shows the appearance of the wooden heat insulation and acoustic absorbing materials generated by this method. The present study used mats prepared with a bulk density of 0.080 g/cm^3 and a thickness of 25 mm for testing.

2.2 Flame-retardant substances

Flame-retardant substances were created using SOUFA (Koubou-Saiga), a borate-based flame-retardant treatment substance with sodium polyborate as its primary component (hereafter referred to as borate-based), and W2-50 (Marubishi Oil Chemical), a phosphate-based flame-retardant treatment substance with ammonium carbamylpolyphosphate as its primary component (hereafter referred to as phosphate-based). Both are water-soluble flame retardants used generally in flame retardant treatments of wood. Treatment conditions were manipulated to regulate the percent rate of weight gain (WG) of the sample according to the treatment type, and to adjust the amount and concentration of the aqueous flame retardant solution according to target WG. The WG standards used were 1, 2, 4, 7.5, and 10 %.

2.3 Fireproofing treatment

In the production of the wooden heat insulation and acoustic absorbing materials, the flame retardant aqueous solutions noted above were sprayed into the chamber by a spray gun during the fiber-spreading and mixing process of the raw materials (Fig. 1).

2.4 Flame testing methodologies

For flame testing, flame retardance was evaluated in accordance with the four testing methodologies described below.

2.4.1 Vertical flame testing

Vertical flame testing is intended to evaluate the vertical progress of combustion and the presence or absence of combustion products. The test specimens were 25 mm in thickness and 20 mm in width, in accordance with UL 94 (2013). The testing can in general terms be considered to equate the effects of fire generated by electrical wiring or electrical malfunctions, or unexpected fires starting during construction in the wall body interior. An overview of the method is shown in Fig. 3. A flame of specified characteristics is applied for 10 s to the end of a vertically held test specimen 125 mm in length, and the combustion's progress is evaluated. Remaining combustion time after removal of the burner is measured; in the event that combustion stops within 30 s, the operation is repeated. Performance is thus judged based on remaining flaming and glowing combustion time, on whether the sample burns up to the clamp, and on whether the sample ignites the cotton placed underneath via falling flaming particles. In this study, performance was evaluated in terms of the sum total of flaming and glowing combustion times, of whether the combustion extended to the clamp, and of whether flaming particles fell.

Fig. 3 Vertical flame testing

2.4.2 Horizontal flame testing

This test is intended to determine combustion speed in the horizontal direction in accordance with JIS D 1201 (Japan Standard Association 1998). The test specimens evaluated had dimensions of 350 × 100 mm and were 25 mm in thickness. It should be noted that the technical parameters of this standard are equivalent to those of ISO 3795 and FMVSS No. 302 (Federal Motor Vehicle Safety Standards No. 302); performance can thus be compared with the felt-type acoustic absorbing materials found inside automobiles. An overview of the method is shown in Fig. 4. The combustion width is set at 50 mm by means of a metal frame, then a flame of specified characteristics is applied to the end of the horizontally-held test specimen for 15 s, and the speed of the flame's progress is evaluated. As the flame did not advance for almost all of the samples in the present research, combustion distance was measured instead.

2.4.3 Flame testing by the 45° Meckel's burner method

This method is intended to evaluate the progress of combustion by a relatively strong flame in accordance with the A-2 testing method (45° Meckel's burner method) in JIS 1091 (Japan Standard Association 1999). Test specimens with dimensions of 350 × 250 mm^2 and thickness of 25 mm were evaluated. Figure 5 depicts the testing method

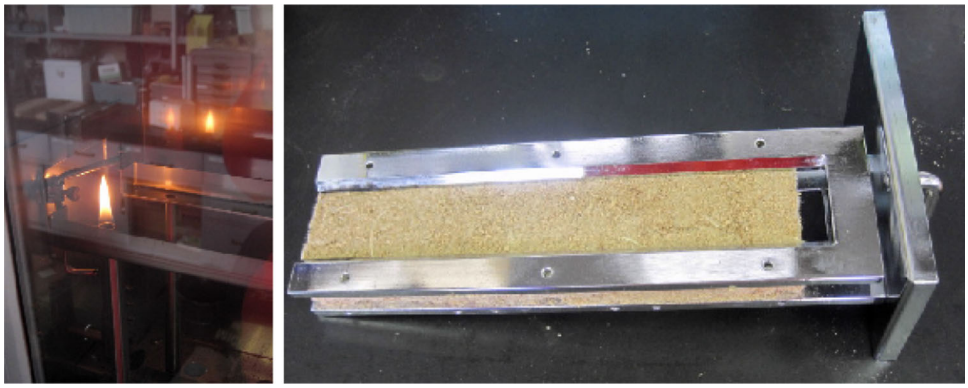

Fig. 4 Horizontal flame testing

in progress. This testing method is meant to be applied to textiles of thick fabric exceeding 450 g/m^2 that require fireproof performance. A flame 65 mm in length is applied via a Meckel's burner to a test specimen placed at 45° inside a given apparatus for 2 min, and the progress of the combustion is evaluated. In the present research, remaining flaming and glowing combustion times were evaluated.

2.4.4 Measurement of ignition and total heat release by cone calorimeter

In accordance with the fire acceleration testing method specified in JIS A 9521 (Japan Standard Association 2011), time to ignition as well as total heat release were tested using a cone calorimeter for a testing time of 5 min and a radiation heat value of 10 kW/m^2. Test specimen dimensions were 100 × 100 × 25 mm^3. Inorganic-based heat insulation materials and performance can be compared using this method. The testing is configured to apply constant radiant heat to the test specimen to thermally decompose the sample, and to test for the presence of ignition of the combustible gas generated. In addition, the heat release rate can be calculated from the change in oxygen concentration of the exhaust gas, enabling capturing not just combustion but other oxidation reactions not caused by the flame. In the present research, the mass increase due to the flame retardant was excluded from the mass of the test specimen when calculating total heat release per unit mass. By dividing total heat release by combustible mass, the effects exerted on the result by differences in test specimen mass were removed.

3 Results and discussion

3.1 Fireproofing treatment

Although the raw material mixture was damp after spraying with the flame retardant aqueous solution, the solvent (water) was evaporated by the subsequent thermoforming process, producing a shape identical to that of a non-treated mixture. Samples treated with phosphate-based flame retardant substances did however go brown after thermoforming, an effect that became more pronounced at higher WG values. Trends corresponding to WG for flame test specimens cut arbitrarily from materials formed into mat shape and subjected to the various flame tests were recorded. The present technique is considered to have achieved homogeneous fireproofing treatment. As no extra workload was added to the manufacturing process, the method can also be considered to be able to impart fireproofing both simply and efficiently.

3.2 Vertical flame testing

Table 1 shows experimental results and Fig. 6 shows samples after testing. The results demonstrate a general trend of fireproof performance increasing in proportion to WG. An almost complete inhibition of combustion progression was obtained at WG 4 %. However, this trend and the form of combustion differed depending on the flame retardant substance. For the phosphate-based retardant, the combustion progressed and almost completely destroyed low-WG samples on first ignition. For 4 % WG, combustion started at the lit end and progressed relatively quickly over the surface during ignition. After the burner was removed and the flame went out, carbonization continued primarily inside the material. Even after lighting of the sample for a second time, the flame extinguished immediately but glowing combustion continued for a relatively long time. For both the 7.5 and 10 % WG samples, neither flaming nor glowing combustion were present after removal of the burner. On the other hand, combustion was gentle even at low WG for the borate-based retardant, and glowing combustion ceased after a short period. However, some glowing remained in the lit portion even at high WG.

The appearance of the phosphate-based samples post-testing showed significant surface charring. However, this

Table 1 Results of vertical flame testing in accordance with UL 94

Flame retardant	Judgment criteria	WG 10 %	WG 7.5 %	WG 4 %	WG 2 %	WG 1 %
Borate-based	Remaining flaming + glowing combustion time (s)	13	11	13	17	<50
	Combustion extending to clamp	Absent	Absent	Absent	Present	Present
	Fallen flaming particles	Absent	Absent	Absent	Absent	Absent
Phosphate-based	Remaining flaming + glowing combustion time (s)	2	0	24	Disintegration by combustion	Disintegration by combustion
	combustion extending to clamp	Absent	Absent	Absent		
	Fallen flaming particles	Absent	Absent	Absent		

was a result of the combustion that progressed over the surface during the application of the flame only. After removing the burner, combustion stopped in a short period of time, during which the inner part of the sample carbonized slightly. Charring was also present on the surface of the borate-based samples; inside the material, there was almost no combustion.

3.3 Horizontal flame testing

Figure 7 shows the results of horizontal flame testing and specimens post-testing. As for vertical flame testing, the results showed an overall trend of fireproof performance increasing in proportion to WG. In addition, flames extinguished at the halfway point even in untreated material; progression of combustion as seen in felt-type materials of general synthetic fibers, when evaluated by the same standards, was absent. Since combustion did not proceed, an untreated specimen of low density was prepared and tested. It was found that although the combustion distance increased, burning occurred only on the sample surface; the interior did not burn. One reason for these superior results to felt-type materials even in untreated samples is thought to be the low calorific value of the wood raw materials. Differences between the flame retardant substances were very slight because the fire only spread over a short distance in all test specimens, however the borate-based retardant displayed slightly higher fireproof performance than the phosphate-based one.

Fig. 5 45° Meckel's burner method

3.4 45° Meckel's burner method

Table 2 shows the experimental results of the 45° Meckel's burner method and test specimens post-testing. Progression of combustion was considerable under the conditions not pictured, so tests for those specimens were pursued no further. In the illustrated conditions, the fire extinguished simultaneously with cessation of heating by the burner. In the WG 10 % borate-based sample, not even glowing combustion was generated. The interior of the WG 7.5 % sample showed some slight glowing for a limited time. Neither the WG 4 % borate-based sample nor the WG 10 % phosphate-based sample ignited, yet they were extinguished prematurely because remaining glowing combustion time exceeded 180 s. Differences in fire intensity and heating time were responsible for the larger extent of combustion. It is suggested that the differences in results cannot be regarded as arising solely from the exposure mode of the flame. In this test, the fireproof performance of the borate-based retardant was strikingly higher than that of the phosphate-based retardant.

3.5 Cone calorimetry

Figure 8 shows the relationship between WG and time to ignition, and Fig. 9 shows total heat release during the testing period (5 min). For both figures, each plot shows the average of three test specimens. For time to ignition, no delay could be observed for the phosphate-based retardant, while borate-based retardants showed a trend of elongated time to ignition with increasing WG. Some specimens of WG 7.5 and 10 % satisfied JIS A 9521, i.e., they were not

Table 2 Test results of 45° Meckel's burner method and specimens post-testing

Flame retardant	Judgment criteria	WG 10%	WG 7.5%	WG 4%
Borate-based	Remaining flaming combustion time (s)	0	0	0
	Remaining growing combustion time (s)	0	180	<180
	Test specimen			
Phosphate-based	Remaining flaming combustion time (s)	0		
	Remaining growing combustion time (s)	<180		
	Test specimen			

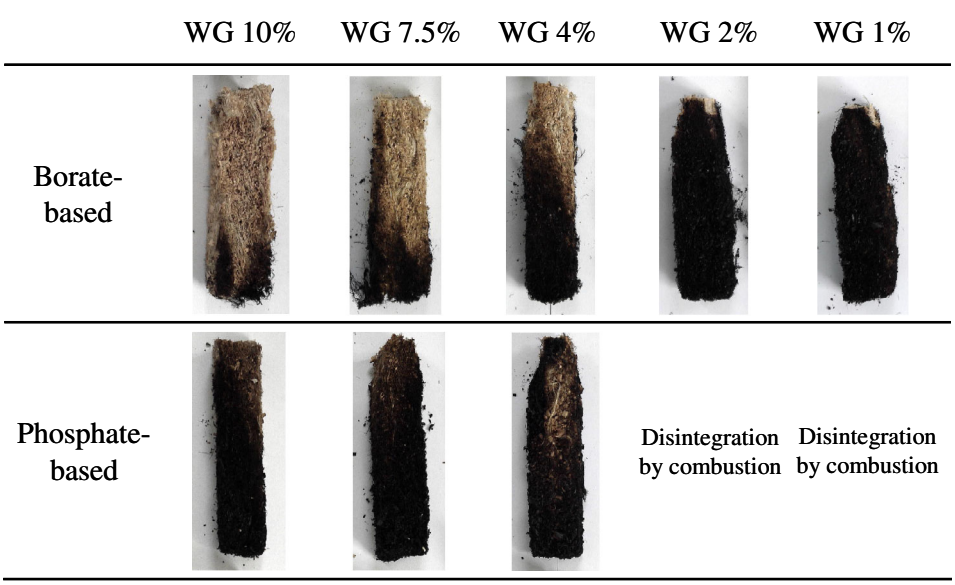

Fig. 6 Samples after vertical flame testing

	(0.08 g/cm³)	(0.06 g/cm³)	Borate-based					Phosphate-based				
WG	Untreated	Untreated	1 %	2 %	4 %	7.5%	10 %	1 %	2 %	4 %	7.5%	10 %
Combustion Distance (mm)	50	130	30	25	20	15	15	45	45	20	20	20

Fig. 7 Results of horizontal flame testing and specimens post-testing

seen to ignite within the 5 min time period. Depending on treatment conditions, the possibility to achieve performance that satisfies the standards of glass wool and rock wool was thus verified. Although an overall trend of total heat release decreasing with increasing WG was shown for both types of retardants, this was more striking for borate-based retardants. Based on these combined findings, the borate-based agent is believed to be effective in inhibiting fires. It should be noted that no differences in correlations of performance with WG between phosphate-based and borate-based retardants have been found in a past research concerning flame-retardance treatment of wood (Fukuta et al. 2010a). In the present study however, experimental conditions using high-intensity flames such as the 45° Meckel's burner method and cone calorimetry revealed the inferior tendencies of the phosphate-based retardants. The browning of the samples post-thermoforming is connected with this tendency, as the thermoforming process is predicted to negatively affect the performance of the substances. While it is intended to investigate the specific factors behind this in future studies, the authors suggest that the dehydration and carbonization reactions that are functions of the phosphate-based flame retardance have already initiated in the thermoforming step. For the thermoforming process, certain kinds of core-sheath-type fiber with the melting point of the sheath part lower (around 120 °C) than the present condition (160 °C) can also be used. By making use of these characteristics, a certain amount of improvement is possible even for phosphate-based flame retardant substances. Furthermore, an oven dryer was used for thermoforming instead of a heat-through dryer, which can treat samples in a shorter period of time, and the required long heating time could also be considered a factor. This is not predicted to present significant problems as industrial thermoforming is performed in a short period of time using a heat-through dryer.

4 Conclusion

Heat insulation and acoustic absorbing materials developed in past studies (Fukuta et al. 2010b, 2012) were endued

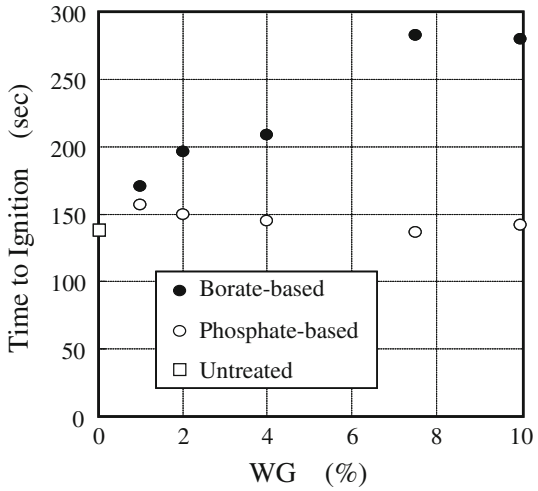

Fig. 8 Relation of WG and time to ignition in cone calorimeter testing

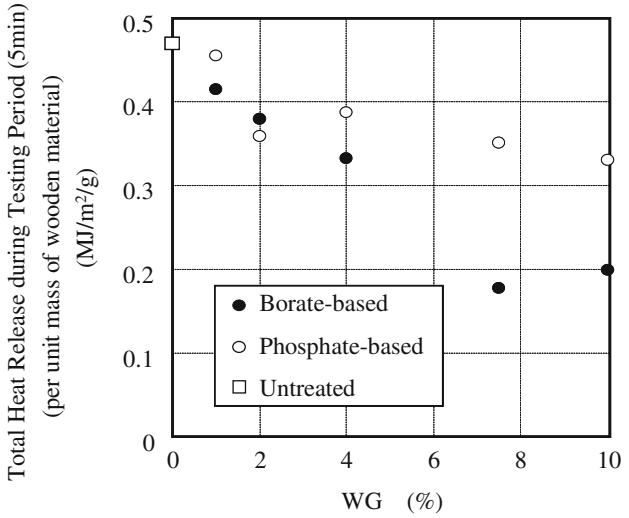

Fig. 9 Total heat release over the testing period (5 min) in cone calorimeter testing

with fireproof performance and subjected to quantitative evaluations that assumed a variety of uses and situations.

It was possible to perform fireproofing treatment homogeneously and without increasing manufacturing workload by using a method of spraying an aqueous solution of flame retardant on the raw materials (wood shavings, kenaf bast fibers, and core-sheath-type fibers as the binding component) during the fiber spreading and mixing step.

Flame retardancy was evaluated by vertical flame testing, horizontal flame testing, the 45° Meckel's burner method, and a cone calorimetry method (in accordance with UL94, JIS D 1201, JIS L 1091, and JIS A 9521, respectively), and the combustion modes of each experimental situation were investigated. By exploring the relationships between treatment conditions and performance, it was possible to obtain valuable information that will likely be useful in achieving widespread expansion and use of such materials.

In recent years, manufacturing techniques for wood-fiber heat insulation materials have been introduced to Japan from Europe, and further similar developments are expected in the future. Furthermore, the distribution of natural-based insulation materials such as wooden heat insulation materials is already progressing in Europe. Treatment with borate-based agents has become a widely-used technique for imparting antiseptic and termicidal performance at the same time as flame retardancy, and agents that suppress problematic moisture absorption in flame retardants have also been developed. Extensive applications of wood in this field can therefore be expected in the future.

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