

Intumescence in fire retardancy of lignocellulosic panels

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Abstract

At present there is growing interest in using wood and wood-based products for applications in various industries (mainly building and transportation). One of the limitations to use of wood is its flammability. The most usual way to improve the fire performance of wood is by chemical treatment with flame retardants. The “classical” mechanisms for flame retardancy of wood require relatively high load of flame retardants in the wood, which was connected with some problems (durability, higher sensitivity to moisture etc.). This paper deals with the intumescent concept of flame retardancy of wood based panels. Particleboard with improved fire performance was prepared in a laboratory conditions. Flame retardant formulations were used with the ability to form an intumescent layer during exposure to heat. This way it was possible to decrease the content of flame retardants in the body of the board. The heat release rate and the mass loss rate characteristics were measured by cone calorimetry. The results were promising in comparison to the classical way of flame retardancy of wood.

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1. Introduction

At present there is growing interest to use wood and wood based products for applications in various industries (mainly building and transportation). There are several reasons for this trend. Wood and other lignocellulosics are CO₂ neutral and renewable raw materials. Exploitation of wood if done properly does not lead to the devastation and/or endangering of nature. There is renewed interest for the industrial use of yearlings for non traditional applications, so there is great potential for the industrial use of lignocellulosic raw materials. The recycling of these products increase the possibilities for exploitation of lignocellulosic materials. They are natural biodegradable polymers, which is nowadays considered as an important property for polymer materials as well. The traditional good properties of wood (its easy processability, good physical and mechanical properties, low density, warm and healthy material etc.) are also of

great advantage. All these aspects make wood and lignocellulosic materials in general, attractive raw materials with high potential for future use.

Wood composite materials currently used in the furniture, construction and transport industries are in the form of boards and panels. However, one of the main limitations for the use of wood is its flammability. Ordinary particleboards belong in Slovakia for example to the flammability class C2 according to the standard STN 730862 which is second most flammable class. Flame retardant modified wood based panels belong to the class B according to the same standard. The lowered flammability of wood based panels enables them to be used in high performance applications. Therefore lowering of the ease of burning of wood materials has been practised for many years. The chemical treatment of wood with flame retardants (FR) is considered at this moment as the most usual and common way to improve its fire performance.

There are three main methods of application of flame retardant to wood and wood products: by impregnation of wood with the solution of flame retardant, incorporation of the flame retardant into the glue system, or surface treatment of the product. The impregnation of

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the wood is usually done with a water borne system, by water solution of the inorganic salts, quite often Lewis acids [1]. The most usual and effective flame retardants are ammonium salts of phosphoric acid, boron acids, zinc chloride, salts of sulfuric acid etc. It was however, found that these chemicals have prodegradation effect on wood strength properties [2] and they decrease the temperature of the beginning of the main process of thermal decomposition in comparison to the untreated wood [3,4]. LeVan and others found that of the FR chemicals used typically for wood, phosphoric acid has the most negative effect on the strength properties. A less severe effect was found for monnoammonium phosphate [5].

A model to predict thermal degradation at elevated temperatures (typically present in construction applications of wood panels) was proposed by Winandy and others [6]. Typical flame retardants can be leached out from the wood if they are exposed in humid conditions and they may increase the hygroscopicity of wood itself [7,8].

The other option for the improvement of the fire performance of wood is surface treatment of the wood product by a flame retardant coating system. Most effective are the intumescent coatings which form the foam during heat exposure. In this case the fire resistance may be improved as well as the reaction to fire, as demonstrated by White [9] and Richardson and others [10]. In this case the flammability of wood itself is not changed, the coatings may be subject to the damage during manipulation and their durability may be limited [8]. In composite materials the flame retardant can be incorporated into the adhesive, or as a mixture with the wood particles or fibres.

Kozłowski and others recently developed composite lignocellulosic mineral board with significantly improved fire performance [11]. They used the vermiculite to improve the flammability. Besides the improvement of heat release and mass loss characteristics, the physical and mechanical properties remained the same as for the untreated boards and the FR itself did not release toxic gases or smoke [11]. Magnesium hydroxide and the combination of monoammonium phosphate with aluminium hydroxide and boric acid were used for the preparation of FR treated particleboard in another study [12].

The common feature of the flame retardancy of wood is that relatively high loadings are needed to reach required improvements. Thus the price of the resulting product is significantly higher than that of the original untreated product. In the present study FR treated particleboard was prepared. Flame retardants with the ability to produce an intumescent layer were used. The effects of intumescent layer typical for the coating systems were combined with the application of flame retardant into the bulk of the material. Thus the

advantages of the protection in the bulk of material over the surface treatment and the protective intumescent layer were combined. The potential of the intumescent layer to decrease the required loading of flame retardants were evaluated. The flammability parameters were measured on a cone calorimeter using oxygen consumption calorimetry [13,14].

2. Experimental

Ordinary wood particleboard was prepared on a laboratory scale. The particleboard had three layers. Two types of chemicals were used as the flame retardants for the preparation of the FR treated particleboards i.e. a phosphate based flame retardant—ammonium polyphosphate (PB) and an expanding char layer forming flame retardant (IFR)—expandable graphite. The former was a standard flame retardant with known efficient flame retarding properties. The latter was chosen since it has char layer forming abilities at elevated temperatures.

Several FR loadings were used from 5 to 25% calculated on the dry particle weight. Melamine urea formaldehyde condensate was used as the adhesive. The moisture content of wood particles was 3%. For the FR loadings up to 13% the flame retardant was mixed with the glue prior to blending with the wood particles. For higher loadings the flame retardants were partly mixed with the wood particles and the remaining part was added to the glue prior to the blending of wood particles. In these cases the process was modified in order to obtain good processability of the glue since the flame retardants increased the viscosity of glue-FR mixture. The blend of wood particles FR and glue was pressed cold, followed by hot pressing at 180 °C.

The overall loadings of the flame retardants and densities of prepared particleboards are given in the Table 1.

The reaction to fire parameters were measured on a cone calorimeter. The materials were conditioned to equilibrium at 55% RH and 23 °C prior to testing. The dimensions of the samples were 100 × 100 mm. The thickness of the test specimens was 17.5 mm. The edge frame was used to minimize the side and edge effects.

Table 1
FR loadings and densities for prepared particleboards

Specimen	Untreated	PB	IFR
Loading (%)	Density (kg m ⁻³)	Density (kg m ⁻³)	Density (kg m ⁻³)
–	587	–	–
5	–	630	580
10	–	690	570
13	–	770	–
20	–	680	–
25	–	790	840

The specimen was placed on a low density ceramic fibre blanket, backed by the high density ceramic fibre board. The materials were tested in the horizontal orientation, at an external irradiance 50 kW/m² which represents severe heat exposure.

3. Results and discussion

Cone calorimeter measurements are useful for the small scale assessment of several reaction to fire parameters and this method is used in a great deal for the development of new materials. The heat release rate (RHR) data correspond directly to the intensity of fire. The heat release rate as a function of time is shown for untreated and flame retardant treated specimens in Fig. 1. The untreated particleboard has a double-peak RHR curve, typical for wood and wood-based products. During burning wood forms a char layer which slows down the heat transfer to the surface and thus the process of thermal decomposition and volatile formation also slows [15]. Therefore shortly after the ignition the heat release rate decreases as the char layer forms.

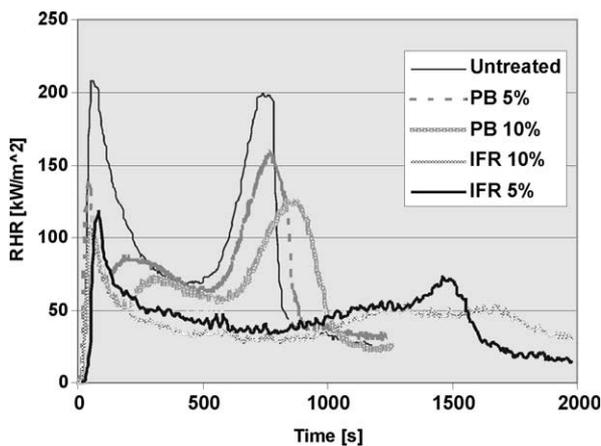


Fig. 1. RHR as a function of time for untreated and FR treated particleboard.

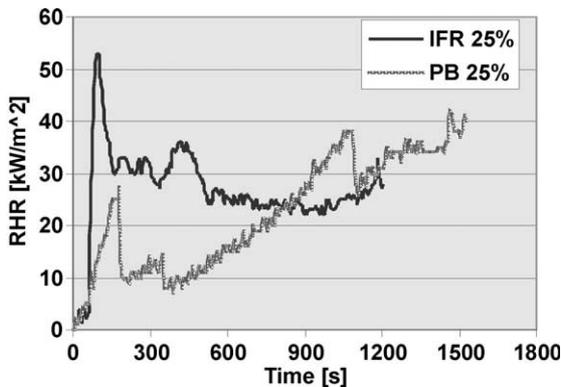


Fig. 2. RHR as a function of time for FR treated particleboard.

The cone calorimeter setup of the experiment is approximately one-dimensional. The external heat flux reaches the burning surface and the heat transfer into the specimen is mostly from the exposed surface. The difference in the peak value of RHR and the middle part of the RHR curve is remarkable. In the case of a sufficiently thick specimen the RHR reaches an approximately steady value in the middle part [16]. The second peak corresponds to the rear end effects of the experiment setup [15].

For the particleboard treated with the phosphate based flame retardant the double peak shape of RHR curve remained except for the high FR loadings (see Fig. 2). The second peaks remained about the same order as the corresponding first peaks similar to untreated particleboard. This observation suggests that the preheating of the specimens prior to thermal decomposition also took place for the phosphate-based modified particleboard. Furthermore the middle part of RHR curves for the uptakes 5 and 10% reached approximately the same values as that for the untreated particleboard.

Different behaviour of RHR curves was observed for the IFR modified particleboards. The first peak values decreased significantly even for low FR loadings both for the PB modified particleboard and IFR particleboards (Fig. 3). However the middle part of the RHR curves for the IFR flame retardant showed remarkably lower values than the untreated material and the PB treated particleboards as well. The second RHR peaks for IFR treated particleboards were almost eliminated even for low FR loadings (Fig. 1).

It can be seen in Fig. 3 that the values of the first RHR peaks were similar for both modifications in the whole range of FR loadings. The average values of RHR for the IFR modified particleboard were much lower than the untreated board even for lowest FR loading. With 5% loading of IFR the average RHR was only 44 kW/m² while for the untreated particleboard the

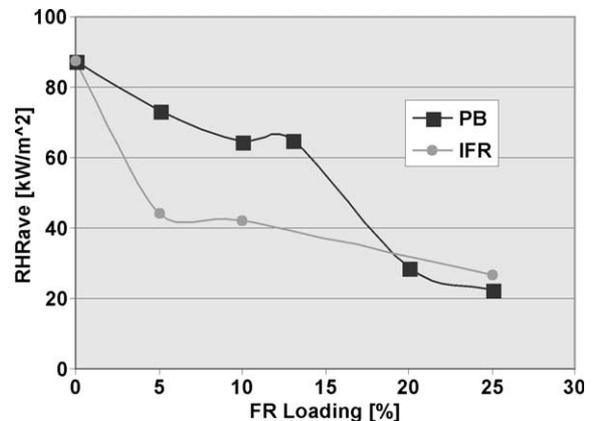


Fig. 3. First Peak RHR as a function of FR loading for particleboard.

average RHR was 87 kW/m². Further decrease of average RHR was not so strong for IFR flame retardant but it reached 27 kW/m² at 25% loading. Much weaker FR effect was observed for phosphate-based flame retardant at the low loadings regarding the average RHR values. For high loadings (around 25%) the values of average RHR were similar for both FR systems. The average mass loss rate measurements (Fig. 4) show similar effects as for average RHR and confirm the above findings. The mass loss rate for the PB flame retardant was very high up to 13% loading. Significantly lower mass loss rate was measured for high FR loadings.

The values for total mass loss of both types of modifications were practically the same for the 5 and 10% loadings and were just 8–13% lower than that of untreated material. This means that in both cases the flammable part of the material was burned out, however the burning time for material with IFR flame retardant doubled in comparison to the untreated material and low FR loadings PB modified materials. The CO yield

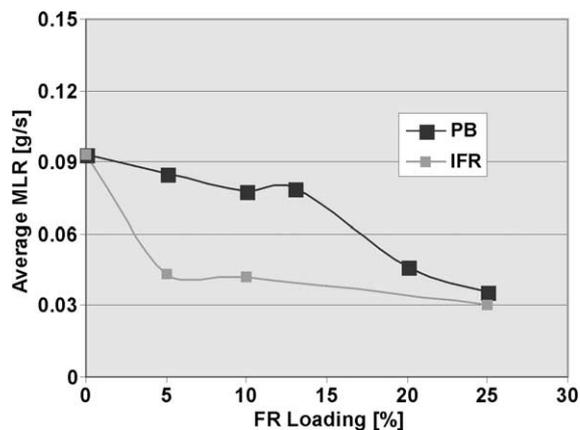


Fig. 4. Average mass loss rate as a function of FR loading for particleboard.

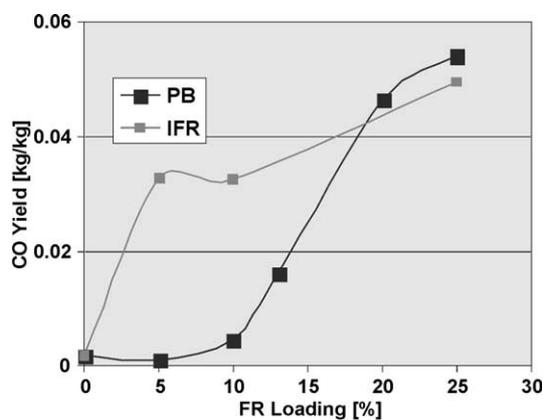


Fig. 5. CO yield vs. FR loadings for particleboard with intumescent systems.

differed for both types of intumescent systems (Fig. 5). For the expandable graphite the CO yield increased significantly even for lowest loadings of flame retardant in comparison to the untreated particleboard. However for the PB system it increased significantly for the FR loadings 13% and higher.

Ignition times, t_{ig} (s) for untreated and FR treated particleboards for the various FR loadings are listed in Table 2. It can be seen that the time to ignition did not change significantly for both FR systems up to 13% loading. For the phosphate based flame retardant at high loadings (20–25%) ignition did not occur, while for the IFR modified particleboards the ignition took place in 66 s from the beginning of heat exposure. However, the flame burning was very short as can be seen on the RHR curve (see Fig. 2).

The selected char retardant formulations formed a protective char layer during the exposure to heat. The charring foam for PB flame retardant was observed only at loadings of 13% and higher while for IFR flame retardant the expanding char layer was formed for all loadings. In the recent study by Duquesne and others [17] the comparison of the effect of expandable graphite and ammonium polyphosphate on the flame retardancy of polyurethane was reported. The rheological and mechanical destruction properties were studied. The ammonium polyphosphate modified polyurethane showed low blowing in comparison to the expandable graphite modified polyurethane [17] which is in agreement with our findings measured at low FR loadings.

A strong decrease of the average RHR and the average MLR was observed for loadings higher than 13% in PB modified particleboard. Ammonium polyphosphate is well known as a strong flame retardant while the IFR flame retardant acts mostly as the physical barrier for heat transfer to the body of the material [17]. As stated earlier, the adhesive used contained melamine which is a typical expanding agent used in intumescent systems. The ammonium polyphosphate probably acts as an acid source [18,19] and wood particles probably act as the carbon supplier.

Due to the strong char layer on the IFR modified particleboards the average mass loss rate as well as the average RHR was much lower than the values of these

Table 2

Time to ignition, t_{ig} (s) for untreated and FR treated particleboards

FR loading (%)	PB t_{ig} (s)	IFR t_{ig} (s)
0	32	32
5	25	31
10	33	26
13	41	
20	Not ignited	
25	Not ignited	66

parameters measured for the PB modified particle-boards at low loadings. Therefore the difference in effectiveness of the two flame retardants at low loadings can probably be explained by the difference in the presence of the expanding charring foam layer on the pyrolysing surface. In both types of modifications the flame retardants were incorporated into the body of the material and therefore as the material was gradually burning through the thickness the flame retardants acted as well. It is interesting to note that even for the lowest loadings the peak RHR values decreased to 55–66% of the original untreated material. However, the ignition times i.e. the time of the start of the sustained flame burning were not changed. This observation can be explained by the fact that the surface itself i.e. the flammable particles of wood on the surface were not directly protected.

4. Conclusions

Char layer forming flame retardants have a strong flame retarding effect. The forming char has a distinctive effect in the performance of the material when exposed to external heat in comparison to the same flame retardant without a char layer. By comparison of the flame retardant systems used in this work it can be concluded that the IFR system worked more effectively than the PB system regarding the heat release rate and mass loss rate characteristics at low loadings. The results suggest that the IFR system has potential to be used to improve the reaction to fire performance and the fire resistance as well of wood-based composite materials.

References

- [1] Košík M, Reiser V, Doležal J, Mihálik P. *Drevársky výskum* 1977;3:175–83.
- [2] LeVan SL, Winandy JE. *Wood and Fiber Science* 1990; 22(1):113–31.
- [3] Košík M, Reiser V, Doležal J, Uhlár J. *Drevársky výskum* 1979; 4:61–7.
- [4] Grexa O, Horváthová E. Flame retardants '96, *Inter Science Communication*; 1996. p. 125–32.
- [5] LeVan SL, Ross RJ, Winandy JE. *Res. Pap. FPL-RP-498*. Madison (WI): USDA FPL; 1990. p. 24.
- [6] Winandy JE, LeVan SL, Ross RJ, Hoffman SP, McIntyre CR, *Res. Pap. FPL-RP-501*. Madison (WI): USDA FPL; 1991. p. 21.
- [7] White RH, Sweet MS. In: Menachem L, editor. *Recent advances in flame retardancy of polymeric materials: proc. 3rd annual BBC conference on flame retardance*, Stamford, CT. Norwalk (USA); 1992.
- [8] Östman B, Voss A, Hughes A, Hovde J, Grexa O. *Fire Mater* 2001;25:95–104.
- [9] White RH. *Journal for Testing and Evaluation* 1986;14(2):97–108.
- [10] Richardson LR, Cornelissen AA. *Fire Mater* 1987;11:191–4.
- [11] Kozłowski R, Mieleniak B, Helwig M, Przepiera A. *Polym Degrad Stab* 1999;64(3):523–8.
- [12] Grexa O, Lubke H. *Polym Degrad Stab* 2001;74:427–32.
- [13] Hugget C. *J Fire Flamm* 1980;12.
- [14] Janssens M, Parker WJ. *Oxygen consumption calorimetry*. In: Babrauskas V, Grayson S, editors. *Heat release in fires*. UK: ESP; 1993. p. 31–59.
- [15] Janssens M. *Fundamental thermophysical characteristics of wood and their role in enclosure fire growth*. PhD dissertation thesis, NFPA, Washington, DC, 1991. p. 6.1–23.
- [16] Mikkola E. *Charring of wood*. VTT report. Technical Research Center of Finland; 1990.
- [17] Duquesne S, Delobel R, Le Bras M, Camino G. *Polym Degrad Stab* 2002;77:333–44.
- [18] Bugajny M, Bourbigot S, Le Bras M, Delobel R. *Polym Int* 1999; 48:264–70.
- [19] Le Bras M, Bourbigot S, Revel B. *J Mater Sci* 1999;34:5777–82.