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Fire Characteristics of Selected Tropical Woods without and with Fire Retardant

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Abstract: The flammability of tropical woods and the effect of a selected fire protection coating were evaluated using a cone calorimeter at a cone radiancy of 35 kW/m^2. Three samples were from the South American continent (Cumaru, Garapa, Ipe), and two were from the Asian continent (Kempas and Merbau). Samples were treated with commercial fire retardant (FR) containing ferrous phosphate as an essential component. The untreated samples were used as reference materials that were of particular interest concerning their flammability. It was shown that there is unambiguous correlation between the effective heat of combustion (EHC) and total oxygen consumed (TOC) related to mass lost during burning for both the untreated and treated samples. In the case of Cumaru and Garapa, there exists an inverse relation between the amount of smoke and carbon residue. The decisive effect on the time of ignition was performed by the initial mass of the sample. This is valid for the spruce and the Cumaru, Ipe, and Kempas, both treated and untreated with retardant, while Garapa and Merbau were found to decline. According to the lower maximum average rate of heat emission (MARHE) parameter, a lower flammability was observed for the treated samples of wood, except for Garapa wood. Fire-retardant treated Garapa and Merbau also have a significantly lower time to ignition than untreated ones.

Keywords: tropical wood; flame retardant; cone calorimetry testing; heat release rate; total smoke production; amount of carbon residue; total oxygen consumed

1. Introduction

By the end of the 1970s, tropical and subtropical tree species started to be imported, processed, and sold in Central Europe. This became a growing trend, due to their positive physical and mechanical characteristics and innovative color finish in comparison with the native trees growing in a different climate zone. Exotic tree species, because of their good stability and lifespan, are used primarily as terraces, balconies, swimming pool flooring, etc., that is, in places with increased humidity and probability of sudden weather changes [1,2]. These tree species are used both in their natural and modified form commonly known as Thermwood [3].

Wood is an organic material, and therefore there is a wide range of substances that can be aimed at the protection of it from any undesirable impacts of the environment. From the fire-fighting perspective, many types of fire retardants have been developed and manufactured, putting stress on their efficiency, aging, and reaction to fire classification [4–6]. It is necessary to point out that lots of attention has been paid to the fire technical properties of common woods (flammable material), such as spruce, oak, beech, etc., and other materials, such as textiles and plastics [7–10]. Until now, less attention has been given to tropical woods, as they represent more complex systems [1,11,12]. The morphology, the different

content of hemicelluloses, cellulose, and lignin, as well as the characteristics and amount of inorganic and low molar mass organic substances may exert a certain influence on their fire behavior.

Three types of trees species that are chosen for our experiments originate in America (Cumaru, Garapa, Ipe) and two tree species in Asia (Kempas and Merbau). The samples come from the Slovak supplier DLH SLOVAKIA s.r.o. (Bratislava, Slovakia) in the form of boards without any surface treatment and have the same dimensions. Laboratory tests are carried out in a cone calorimeter with the aim to evaluate their flammability by comparing the series of parameters of the respective tropical wood as well as with those of the samples treated by coating with fire retardant containing ferrous phosphate. In the particular case of the selected tropical woods, Reference [11] deals with the burning of Merbau wood; however, the initial content of water was unexpectedly high, and thus the effective heat of combustion (EHC) measured at 50 kW/m^2 was very low (4 MJ/kg) when compared with the EHC of common woods (14–18 MJ/kg).

2. Materials and Methods

2.1. Materials

Figure 1 shows the cross-section and microscopic structure (type of microscope: TESCAN SEM Solutions) of the selected tree species—three of them originate in America: (a) Cumaru, (b) Garapa, (c) Ipe, and the remaining two in Asia, (d) Kempas, and (e) Merbau.

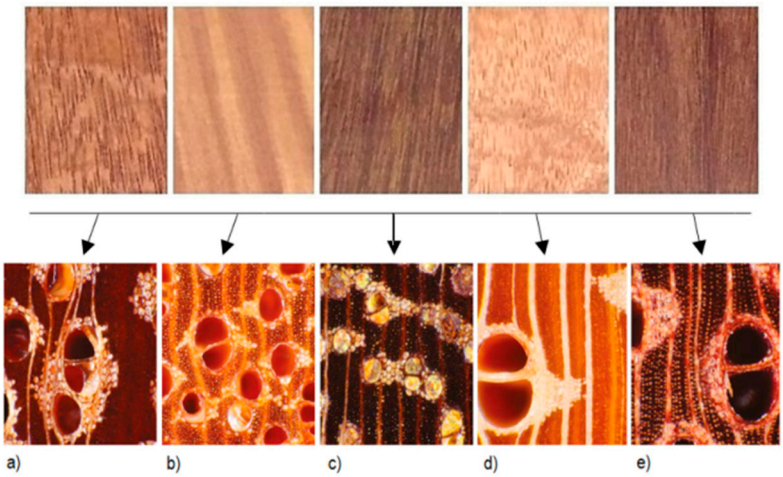

Figure 1. Microscopic structure (cross-section) of tropical tree species from listed as (**a**) Cumaru, (**b**) Garapa, (**c**) Ipe, and the two remaining in Asia, (**d**) Kempas, (**e**) Merbau.

Based on [13,14], Kempas and Merbau have the most distinct vessels, followed by Cumaru, Garapa and Ipe, in descending order. Ipe wood contains traces of lapachol, a substance having a protective function. Each tree type has single as well as double vessels. Garapa wood even has triple vessels. These trees also contain distinct medullary rays. Aside from the vessels and the rays, the pictures show the wood fiber of the particular tree species.

Cumaru (*Dipteryx spp.*) is a tree species with yellowish brown to reddish brown heartwood, clearly demarcated yellowish white sapwood, and with spiral, demarcated, and interlocked fibers of a darker color. Red-brown is the reference color. Sapwood is 2 to 3 cm thick [13,15].

Garapa (*Apuleia leiocarpa*) is a tree species with a yellowish beige to yellowish brown heartwood, yellowish white sapwood, and with straight and just a few spiral but clearly demarcated and interlocked fibers. Sapwood is 5 to 11 cm thick. Orange yellow is the reference color. As the wood ages, the yellow color changes to light brown [13,16].

Ipe (*Tabebuia spp.*) is a tree species with olive green to dark brown heartwood, yellowish white sapwood, and thin spiral and interlocked fibers. Traces of lapachol—which is liquid protecting the wood—can be found in the pores. Reference color is brown, and sapwood is 3 to 9 cm thick [13,17].

Kempas (*Koompassia malaccencis*) is a wood of roseate and gradually darkening reddish brown heartwood, light yellow to white sapwood, straight and just a few spiral interlocked fibers as well as frequent concentric layers of phloem. Reference color is red-brown, and sapwood is 2 to 5 cm thick [13,18].

Merbau (*Intsia bijuga*) is a tree species having reddish brown to dark brown heartwood, light yellow sapwood, and straight and only a few spiral interlocked fibers (so do Garapa and Kempas). If exposed to light without any surface protection for a longer period of time, the wood changes its color by becoming dark red or dark brown in color [13,19].

Samples of 100 × 100 × 20 mm ± 1 mm were used during the test. Four untreated samples and one sample coated with fire retardant (FR) were selected for each tree type described above. Chemical composition of the FR is in the Table 1.

Table 1. Chemical composition of FR.

Name
$FeHPO_4$
Citric acid
Polyoxyethylene stearyl alcohol
Water

The coating was applied twice using a paint roller with FR (minimum 300 g/m^2) on the surface of the sample. After drying at different intervals, there was no visible measurable coating layer on the surface of the samples, and therefore we assumed that the majority of it was absorbed by the material.

2.2. Methods

2.2.1. Test Equipment

The device is used to study the fire behavior of small-size samples made out of various materials in solid form. The peak release rate (PkHRR) is a key factor needed to assess the fire characteristics. The heat release rate from a cone calorimeter was obtained by monitoring the oxygen consumption based on the difference in the cone entrance and in a cone extraction pipe of air flows in a cone calorimeter system. It is assumed that an average of 13.1 MJ of energy is released per one kilogram of consumed O_2 [20]. The heat flow was set at 35 kW/m^2. Other readouts were as follows: ignition time, total burning time, weight loss, effective heat consumption (EHC), O_2 consumption, smoke release rate, etc. No CO detection was available. The device (the product of Fire Testing Laboratories Ltd., West Sussex, UK) consists of a number of parts, as seen in Figure 2.

Figure 3 shows the test procedure in a cone calorimeter. It starts by preheating the samples using a radiant heat source (heating coil), followed by the ignition of the combustible gaseous components evaporating from the sample. The fire starts to spread, and the charred layers are observed.

Figure 2. Cone calorimeter.

Figure 3. Samples tested in the cone calorimeter.

2.2.2. Measurement Procedure

The sample of the given dimensions was mounted into the steel frame in a horizontal position so that the only part of the sample exposed to heat is its surface, not its edge. In order to prevent the material from peeling and the chemical components from dripping, the sample was wrapped up into an aluminum foil from the bottom and the sides. The radiator, which has a shape of a truncated cone is placed above the sample (radiant heat source). It represents a constant radiation source that the sample was exposed to. The temperature was regulated using 3 thermocouples and a thermostat. Weight measurements were carried out using a load cell of a tensometer with a readability of 0.01 g. A 10 kV spark generator equipped with a safety shut-off mechanism is used to ignite the vapors of the test specimens. There is a steady flow of air of 24 l/h and a constant temperature of 770 °C [21].

In the tests, the sample is ignited using a spark. The timer is activated the moment the sample ignites and is turned off the moment the sample goes out spontaneously. Smoke and combustion gases from the sample are absorbed by the suction hood with a tube made out of stainless steel equipped with a fan of adjustable flow rate (0 to 50 g/s), a measuring probe for the products of combustion, a thermocouple, a pressure transmitter, and a parameter analyzer of O_2 (0–25%). The smoke release rate is determined using a laser with photodiodes. The heat release rate is calibrated using methane 99.5% [21].

3. Results

Attention has been given to a proper selection of samples with respect to their humidity and density. The samples were climatized under standard conditions (22 °C and 55% of air humidity) for 7 days before and after the application of the retardant. The content of water in the samples was controlled gravimetrically to a constant weight achieved by conditioning. The samples were selected from the cut boards (see Table 2) so that differences not higher than ± 5% kg/m^3 were allowed.

Table 2. The density of samples.

The Sample	Density [1] (kg/m^3)	Density [2] (kg/m^3)
Cumaru	1200	1070
Garapa	1050	790
Ipe	1300	1050
Kempas	1050	880

[1] The density of fresh wood and density; [2] the density at 12% of water content.

The amount of retardant absorbed is given in Table 3. As the properties of tropical woods differ due to different densities and thus porosities, the penetrability of the coating into the mass of the sample was rather different, as was the amount of retardant coated.

Table 3. The acceptance of wet retardant in grams.

The Sample	Cumaru	Garapa	Ipe	Kempas	Merbau
The acceptance (g)	2.4	2.5	6.5	8.4	2.6

The results are documented in the following section either in a tabular form (see Table 4) or graphically (see Figures 4–6). Figure 4 is a representation of four parallel HRR (heat release rate)–time runs for Merbau samples. One can see that repeatability of the respective experiments is quite satisfactory. Typical HRR–time runs involve a sharp increase of the heat released just after ignition. At that time, the flame is spread along the surface, and in its close proximity below, the flame then penetrates through the layer of carbonaceous residue where high molar mass products of the degradation are pushed into the bottom of the carbonaceous layer by the heat front and accumulate there. In the final stage, there appears a second maximum, which characterizes the burning of the accumulated products, followed by the extinction of the flame. The lines HRR–time are in fact a first derivative of mass loss runs, which may also be seen in Figure 4. It is of interest that the retardation effect on the Merbau samples performed by the FR is seen particularly at the final stages of burning. This is more or less distinct for all retarded samples. The prolongation of the time of burning is the most significant for Cumaru and Garapa. Garapa and Merbau samples showed a significant reduction in time to ignition (90 vs. 65 s, and 91 vs. 54 s). From the set of respective parallel runs, the average curves were determined, which may be seen in Figure 5 (Cumaru—C, Garapa—G, Ipe—I, Kempas—K, Merbau—M), as well as the standard deviations concerning the time of ignition, the time of burning, the amount of carbon residue in percentage, the peak of HRR, and MARHE (Table 4). The graphical results representing HRR curves for the samples treated with FR are in Figure 6.

Table 4. Parameters read from cone calorimetry measurements of untreated samples C (Cumaru), G (Garapa), K (Kempas), M (Merbau), and I (Ipe), and samples treated with FR.

Sample	Parameters								
	m (g)	m_{lost} (g)	Time to Ignition/Time of Burning (s)	EHC (MJ/kg)	Peak HRR (kW/m^2)	TOC (g)	TSR (m^2/m^2)	MARHE (kW/m^2)	% of Carbon Residue
C_{av}	163.3	121.5	45 ± 4/1067 ± 30	15.6	457.2 ± 42.3	115.9	662.2	176.5 ± 5.1	25.5 ± 0.3
C_{fr}	173.2	114.4	51/1514	13.4	264.4	93.5	189.0	98.1	33.9
G_{av}	180.0	118.5	90 ± 10/1439 ± 49	13.6	275.0 ± 28.2	98.4	205.7	107.4 ± 6.3	34.2 ± 0.5
G_{fr}	189.8	166.0	65/1731	15.9	342.5	161.0	923.0	148.6	24.5
K_{av}	139.9	112.7	37 ± 2/1200 ± 34	17.1	307.8 ± 9.8	115.8	385.8	155.6 ± 7.4	20.6 ± 1.7
K_{fr}	138.7	105.7	42/1237	17.5	268.1	112.8	426.8	145.0	23.8
M_{av}	253.3	183.0	91 ± 16/1676 ± 50	17.4	438.4 ± 32.8	194.8	1468.8	187.9 ± 8.4	27.7 ± 1.2
M_{fr}	253.7	183.4	54/1565	17.1	357.6	191.6	1924.1	170.1	27.7
I_{av}	227.0	171.9	89 ± 5/1340+-64	16.9	500.9 ± 17.9	177.6	1678.8	224.6 ± 8.6	24.4 ± 0.4
I_{fr}	230.6	170.2	98/1413	15.8	452.6	164.7	1745.9	187.8	26.2

Notes: m_{lost} = total loss of mass during burning; time to ignition/time of burning; time to ignition = time interval from insertion of the sample below the cone heater to ignition (piloted ignition by spark); time of burning = time of the flame burning of the sample until self-extinction; EHC = effective heat of combustion; peak HRR = maximum heat release rate corresponding to the second maximum of record; TOC = total oxygen consumed during combustion; av = average value; fr = sample coated with FR; MARHE = a maximum value of ARHE (average rate of heat emission) ARHE = $\frac{\int \mathrm{HRR} * t * \mathrm{d}t}{t1}$, with an integral in the nominator from 0 to t_1.

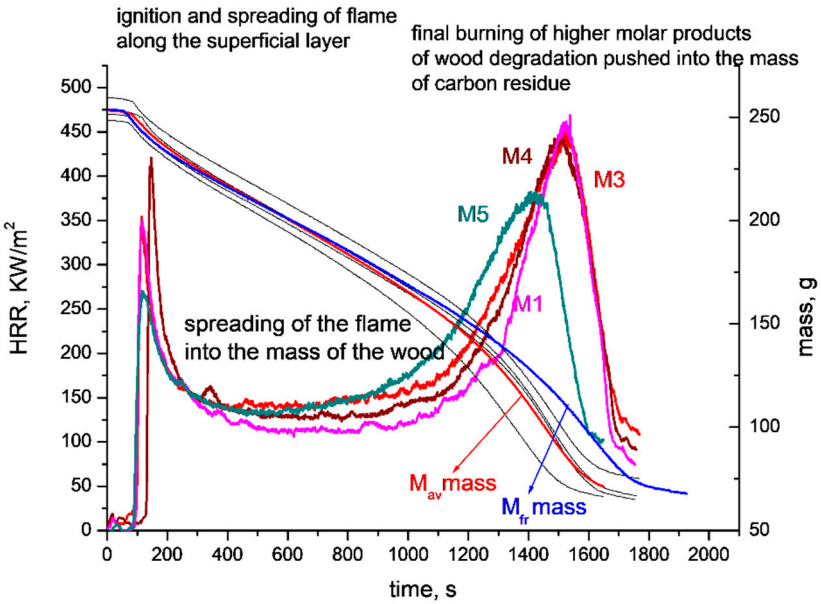

Figure 4. Merbau sample in four parallel runs of heat release rate (HRR) and mass with time. Cone radiancy is 35 kW/m^2; M_{av}mass is the average mass change of samples M_1, M_3, M_4, and M_5.

The most important parameters for similar experiments with spruce samples are in the Table 5.

As it may be seen from Figures 5 and 6, the peak HRR (the second maximum) of treated Cumaru is significantly lower than that of the untreated one; lower peak HRRs for treated samples were also observed for Kempas, Merbau, and Ipe, while Garapa gave a more intense peak of HRR. The difference is the highest for Cumaru.

All essential parameters read from the cone calorimetry experiments for both untreated and treated samples are presented in Table 4.

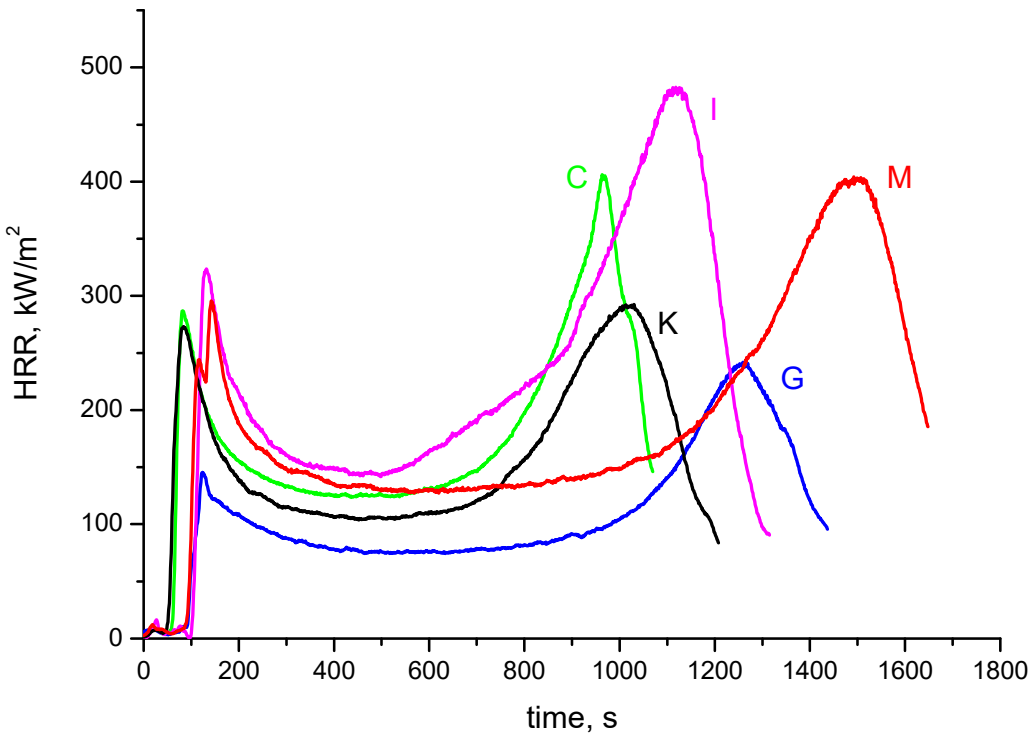

Figure 5. The average HRR–time runs for untreated samples at 35 kW/m^2; C—Cumaru, G—Garapa, I—Ipe, K—Kempas, M—Merbau.

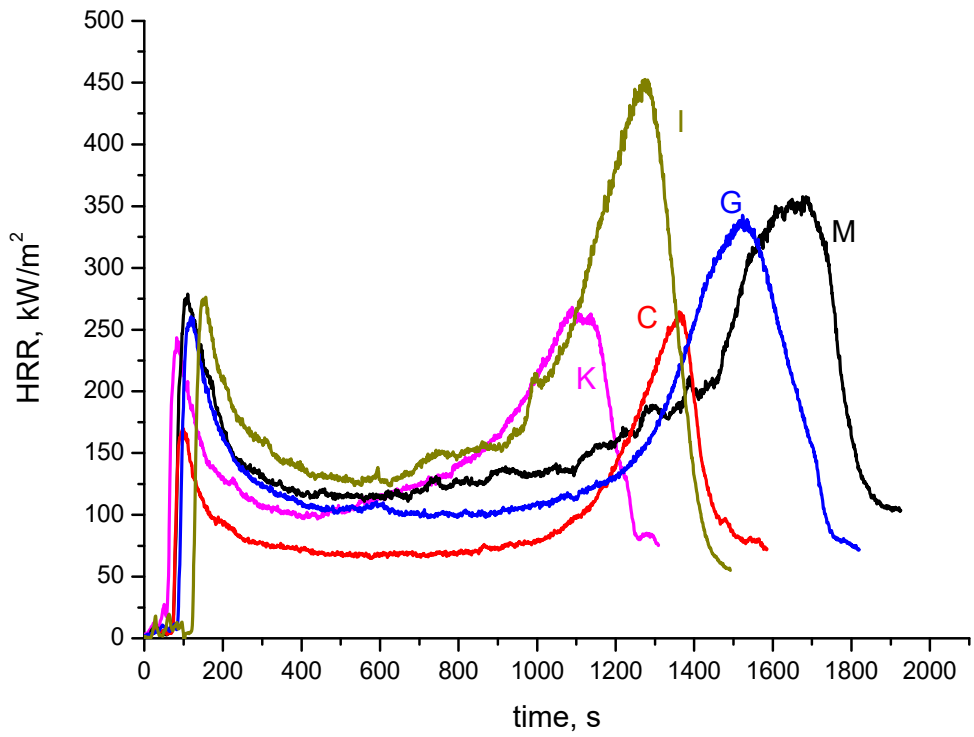

Figure 6. HRR of the samples coated with FR; C—Cumaru, G—Garapa, I—Ipe, K—Kempas, M—Merbau. Cone radiancy at 35 kW/m^2.

Table 5. Some parameters from the cone calorimeter after the burning of selected samples of spruce fir differing in initial density (dimensions of samples at 9 × 9 × 1 cm) with a cone radiancy of 35 kW/m^2.

Sample	Some Parameters from Cone Calorimeter after the Burning of Selected Samples						
	Density (kg/m^3)	Initial Mass (g)	Time to Ignition (s)	EHC (MJ/kg)	TOC (g)	Peak HRR (kW/m^2)	Carbon Residue (%)
Spruce Fir	561	55.02	47	15.2	41.6	227.0	17.2
	553	53.17	51	14.3	37.2	232.7	17.9
	445	43.64	47	14.4	31.7	234.9	15.1
	458	44.45	41	14.4	32.5	218.5	–
	385	37.37	31	15.3	30.1	224.2	12.6
	392	38.06	36	15.3	29.9	241.0	13.9

The ignition time t_{ig} (s) can be expressed as follows, as given in Equation (1):

$$t_{ig} = \pi k \rho c \left(\frac{T_{ig} - T_0}{2\delta l} \right)^2 \quad (1)$$

where t_{ig} is ignition time in seconds, π is Pi, k is thermal conductivity in W/m K, ρ is density in kg/m^3, c is specific heat in J/kg K, T_{ig} is the surface temperature of the material when ignited (in K), T_0 is the ambient temperature (in K), δ is emissivity, and I is radiation intensity (in kW/m^2).

From this, it follows that the density and thus the initial mass at the identical dimensions of the sample is the decisive factor determining the time to ignition (Figure 7). On samples of the same quality (spruce), this is quite evident, while for tropical trees differing in structure and the amount of other additional compounds, the picture is rather complex. K_{av}, C_{av}, and I_{av} follow the straight line X approximately, but G_{av} and M_{av} decline. Figure 7 also shows the changes of the time to ignition when the samples were treated with the FR. The significant reduction of time to ignition occurs with samples Merbau and Garapa, while Cumaru, Kempas, and Ipe are affected only a little.

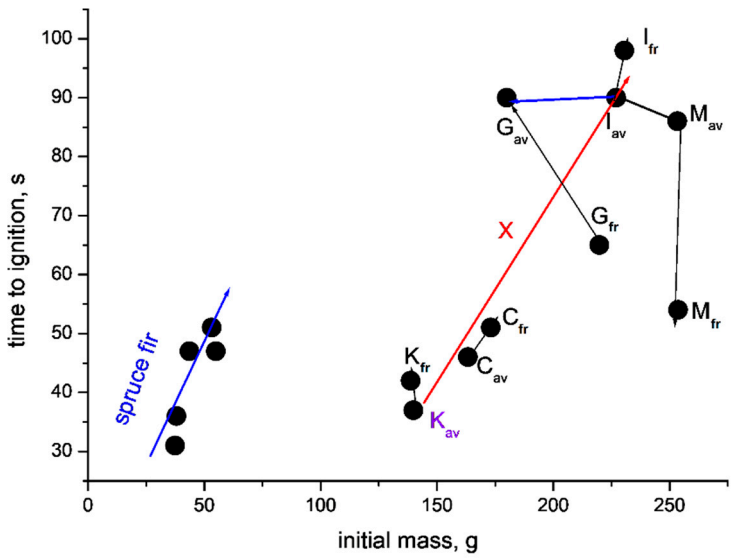

Figure 7. Time to ignition in dependence on the initial mass of the untreated and samples treated with FR.

4. Discussion

The main component of the fire retardant (FR) is secondary ferrous phosphate, which may provide antagonistic effects in retardation of burning as it comes to ignitability. This may represent a particular interest. Ferrous and subsequently ferric ions will promote the degradation of cellulose in a wood to

lower molar mass products, while phosphate moiety may initiate crosslinking, and later on the increase of carbon residue, as it has a pronounced effect on the trapping of higher molar mass products of the degradation. This appears to be the case for Garapa wood (Table 2) where the prodegradation effect of ferrous ions seems to be suppressed. This may be documented by the higher peak HRR. We assumed that the carbonaceous residue is denser and keeps more degradation products, while in all other cases it is quite opposite. The reduction of peak HRR is the most pronounced in the case of Cumaru and Merbau. In the case of Kempas and Ipe, the effect is rather weak. The overview of the effect on the modified samples concerning the HRR and the time to ignition may be seen in Figure 8. The MARHE parameter may be considered as a scale of the samples' flammability (Figure 9). Except for Garapa, all other samples have a MARHE of lower flame retardancy (lower flammability) than untreated ones.

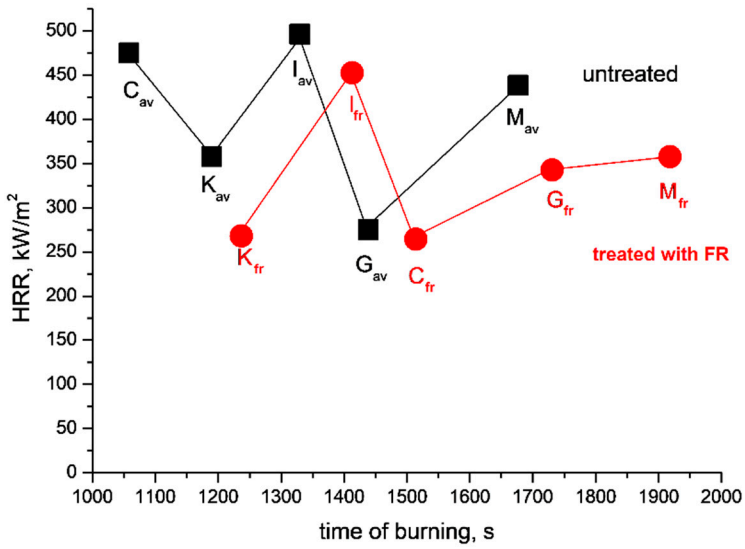

Figure 8. Changes of HRR with time of burning for tropical woods untreated and treated by FR (red line).

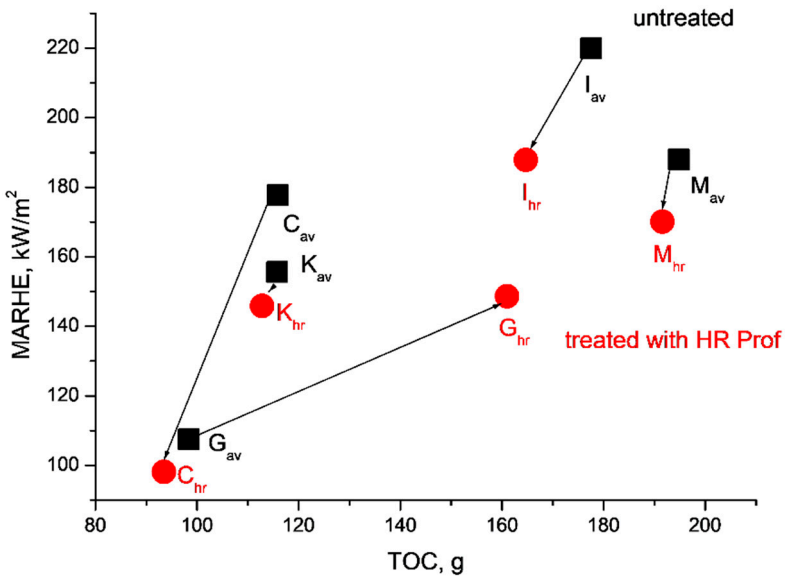

Figure 9. The correlation between MARHE and total oxygen consumed for untreated and treated samples of tropical woods (Table 2). The cone radiancy is 35 kW/m^2.

Figure 10 shows the universal relation between the effective heat of combustion (EHC) and the total oxygen consumed (TOC) divided by the mass lost during burning. The straight line received in both cases

of tropical woods as well as spruce fir has the following shape: EHC = 1.008 + 15.46 × TOC/mass lost, where the slope around 15.5 kJ/g is higher than the parameter 13.1 kJ/g of consumed oxygen implemented in the cone calorimeter. The difference around 20% to 30% appears to be due to the formation of carbon residue in the case of woods. It may be of interest that the above correlation is valid also for spruce samples that are thinner than tropical woods.

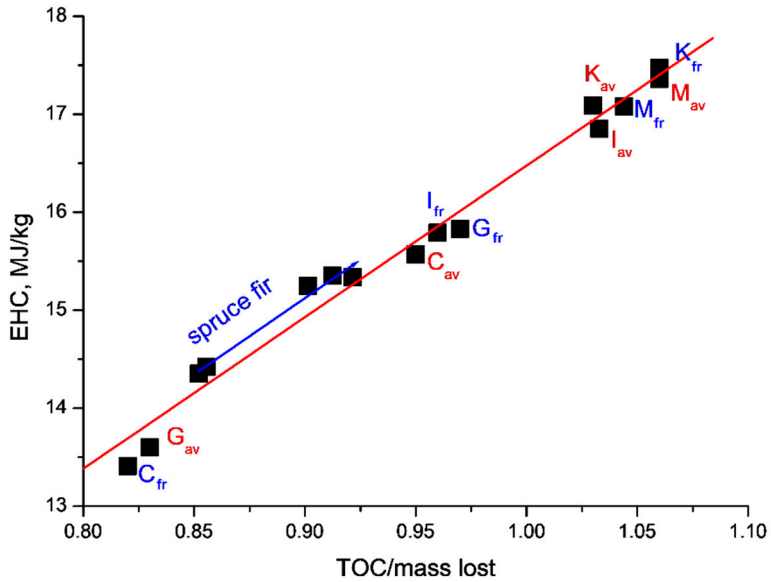

Figure 10. Universal correlation between effective heat consumption (EHC) and total oxygen consumed related to unity of mass lost during burning.

In Figure 11, we verified the assumption that the total smoke released should be inversely proportional to the carbonaceous residue formed. In the case of Cumaru and Garapa, after the modification with FR it appears to be true. The unmodified Garapa gave a relatively high carbon residue, while the total release of smoke is low; after modification, carbon residue is significantly reduced, and the total smoke increases. Unmodified Cumaru gave a higher smoke release and a lower amount of carbon residue, but in the case of modified samples it is vice versa. For other tropical woods, this is not unambiguous as the effect is rather low (Figure 11).

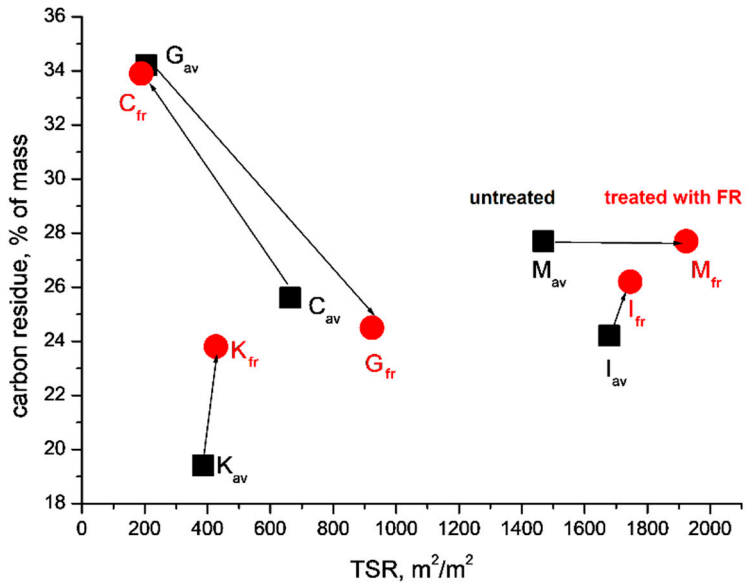

Figure 11. Correlation between carbon residue after sample extinction and total smoke release.

5. Conclusions

There is an unambiguous relation between the EHC and TOC related to mass lost during burning. The slope is, however, higher (about 15 kJ/g of consumed oxygen) than that implemented into the cone calorimeter (13.1 kJ/g of consumed oxygen). The data received from the spruce fall into this graph as well. This appears to be due to the compensation by carbon residue resting at the end of burning. This may contribute to a better understanding of the results obtained for the sample, as its residue leaves when burning. In the case of Cumaru and Garapa, there exists an inverse relation between the amount of smoke and carbon residue.

As it was also reported in [1,11,12], the time to ignition is affected by the initial mass of the sample. This is valid for the spruce and the Cumaru, Ipe, and Kempas, both treated and untreated with retardant, while the Garapa and Merbau were found to decline.

Cumaru and Garapa treated with the fire retardant show a significantly longer time of burning. The difference with the others was not so distinct. With the exception of Garapa, most of the samples performed a lower peak HRR. A similar effect may be found with a MARHE parameter that is lower (which indicates lower flammability) for treated samples, except again for Garapa wood.

Treated Garapa and Merbau also have a significantly lower time to ignition than untreated ones. This may be a demonstration of the antagonistic effect of the fire retarding agent—ferrous ions shorten the time to ignition, while phosphate moiety playing a role in the formation of carbon residue prolongs the overall time of sample burning.

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