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FUNDAMENTAL THERMOPHYSICAL PROPERTIES OF MATERIALS DERIVED FROM THE CONE CALORIMETER MEASUREMENTS

Ondrej Grexa

State Forest Products Research Institute, Bratislava, Slovakia

Marc Janssens

American Forest & Paper Association, Washington D. C., USA

Robert White & Mark Dietenberger

Forest Products Laboratory, Madison, USA

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INTRODUCTION

This paper presents a partial progress report of an ongoing international research project. The three year project is conducted under the auspices of the U. S.- Slovak Science and Technology, with the title "Room/Corner Test and Reaction to Fire of Wood and other Building Materials". The project consists of three major parts; bench scale test measurements according to ISO 5660 (Cone calorimeter), full-scale testing according to ISO 9705 (room/corner test), and mathematical modeling of the room/corner test based on the bench scale measurements. In this paper, only the results of bench scale tests obtained during the first year and partly in the second year of the project are presented and analyzed. The results of the first year of project are reported in [1-3]. The Cone calorimeter measurements were done at the State Forest Products Research Institute (SFPRI), Bratislava, Slovakia, the full-scale tests were done at the Forest Products Laboratory (FPL), Madison, USA. From the bench scale measurements, the material properties were derived in a form suitable for input into a mathematical model of the room/corner test.

The Cone calorimeter has become one of the most spread and used apparatuses for the reaction to fire characteristics measurement of materials during last few years. The Cone calorimeter method is the ISO standard (ISO 5660-1) [4] and it was adopted in many

countries. The main parameter measured in the Cone calorimeter is the heat release rate (RHR) based on the oxygen consumption principle. According to this principle heat released during burning of material is proportional to the amount of oxygen needed for burning. For most solid materials this relationship equals 13.1 MJ of heat released per 1 kg of oxygen consumed [5]. Besides the heat release rate some other parameters are measured in the Cone calorimeter, which are effective heat of combustion, total heat release, mass loss rate, specific extinction area of smoke, time to ignition, rate of production and yields of various gases (usually CO and CO₂).

MATERIALS TESTED

Tested materials and their characteristics are given in table 1.

Table 1. Characteristics of Tested Materials

Material Name	Character.	Origin	Thickness [mm]	Overall Density [kg/m ³]
Douglas Fir plywood	5 layers	ASTM round robin	11.5	541
Douglas Fir plywood	3 layers	FPL material bank	12.0	513
Oak Veneer plywood	5 layers	FORINTEK	13.0	479
Douglas Fir plywood FRT	5 layers	ASTM round robin	11.8	558
Southern Pine plywood FRT	5 layers	FORINTEK	11.5	599
Polyurethane foam FRT	rigid	ASTM round robin	23.0	29
Gypsum Board type X	paper covered	Local supply FPL	16.5	755
Beech Wood	lumber	Local supply SFPRI	15.0	749
Spruce Wood (Picea excelsa)	lumber	Local supply SFPRI	15.0	468
Oriented Strand Board		FPL material bank	11.0	643
Preswood hard board		FPL material bank	6.0	1026
Redwood lumber	flooring	FPL material bank	19.0	421

EXPERIMENTAL

The materials were conditioned to equilibrium at 55 % RH and 23 °C prior to testing. The dimensions of the samples were 100 x 100 mm. The thickness of the test

specimens was the actual thickness of the materials. The edge frame was used to minimize the side and edge effects. The specimen was placed on low density ceramic fiber blanket, backed by the high density ceramic fiber board. The materials were tested in the horizontal orientation at external irradiance levels of 20, 25, 30, 35, 40, 50, 65 kW/m². For some materials additional tests were conducted at lower irradiance levels, or at irradiance levels in between, Tests were terminated when the average value of mass loss rate over a period of one minute dropped below 150 g/m², as specified in the ISO 5660-1 standard [4].

RESULTS AND DISCUSSION

The aim of this work was to measure and derive thermophysical properties of materials needed for mathematical modeling of the room/corner test (table 2). From the measurements of time to ignition at various irradiance levels, \dot{q}_e'' , the following properties were derived; total heat transfer coefficient, h_{ig} ; ignition temperature, T_{ig} , thermal inertia, $k\rho c$; and critical heat flux for ignition, \dot{q}_{cr}'' . The procedure used for this purpose was originally developed by Janssens [6] and is briefly summarised as follows. It is assumed that the test specimens behave as a semi-infinite solid The boundary condition at the exposed surface when ignition occurs is given by equation (1):

$$\varepsilon \dot{q}_{cr}'' = h_c (T_{ig} - T_a) + \varepsilon \sigma (T_{ig}^4 - T_a^4) \equiv h_{ig} (T_{ig} - T_a) \quad (1)$$

The time to ignition is correlated with external irradiance via (2):

$$\dot{q}_e'' = \dot{q}_{cr}'' \left\{ 1 + 0.73 \left[\frac{k\rho c}{h_{ig}^2 t_{ig}} \right]^{0.547} \right\} \quad (2)$$

First the critical irradiance level \dot{q}_{cr}'' is found as the intercept of the regression line according to equation (2) with the abscissa, Average values of t_{ig} from two or more replicates at each flux level were used for the calculation of the regression line. The ignition temperature, T_{ig} , is then calculated from equation (1) using the Newton-Raphson iteration method. Subsequently, h_{ig} is also calculated from equation (1). Finally, the $k\rho c$ parameter is found from the slope of the regression line in equation (2)

To apply this procedure the convective heat transfer coefficient h_c is needed The value suggested by Janssens for vertical orientation in the Cone calorimeter was used in this study; $h_c = 13.5$ W/m²K. A value of 0.88 was chosen for the emmissivity, ε , as recommended by Janssens for wood products [6]

The heat of gasification, L was derived as suggest by Quintiere [7] Peak values of the heat release rate (RHR) were plotted against external irradiance. The heat of gasification, L , was calculated from the slope of a linear fit through the data, The slope is equal to

EHC/L. EHC is the heat of combustion, which is obtained as the ratio of heat release rate and mass loss rate, both measured in the Cone calorimeter. An average value of EHC was used to determine L from the slope of the regression line.

UNTREATED WOOD PRODUCTS

Figure 1 shows the resulting correlation of ignition time versus external irradiance for Douglas fir plywood (ASTM round robin). The correlation of peak heat release rate versus external irradiance is in Figure 2 for the same material. Both graphs illustrate a linear dependency, confirming the validity of the procedures used to derive effective material properties. The regression analyses for the remaining untreated wood products show a similar trend

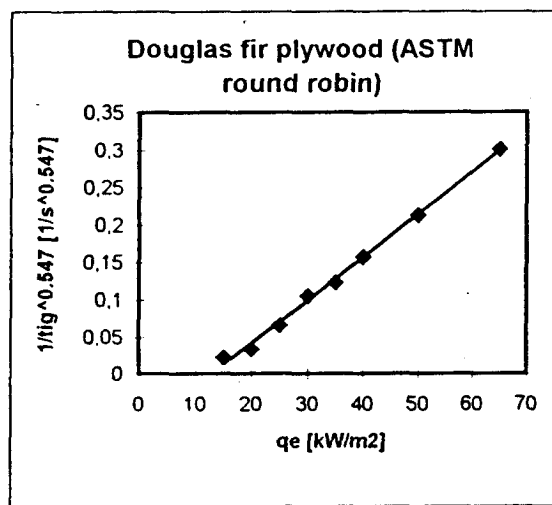


Figure 1: Ignition time correlation for Douglas fir plywood (ASTM round robin).

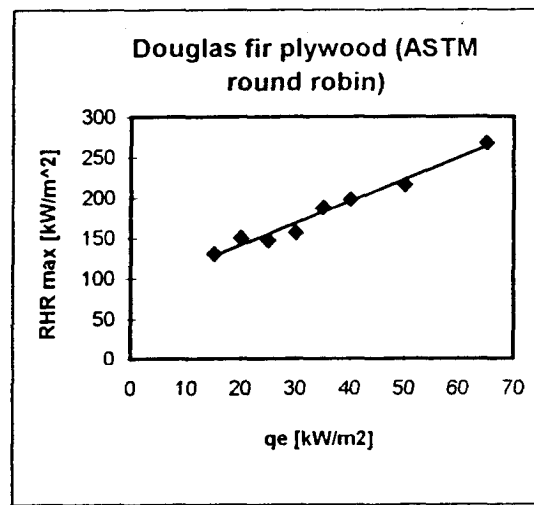


Figure 2: Heat release rate correlation for Douglas fir plywood (ASTM round robin)

FIRE RETARDANT TREATED WOOD PRODUCTS

One of the main assumptions in the procedure suggested by Janssens [6], is that the materials are inert prior to ignition. This means that the chemical degradation is considered negligible prior to ignition. This is a reasonable assumption for untreated wood products. However, chemically treated wood products behave in a significantly different way. The processes occurring at FRT wood depend on the type of fire retardant used, the method of treatment and the uptake by the material. If an efficient fire retardant is used and the uptake is high, sustained ignition may not occur under realistic exposure levels, despite high mass loss.

Two fire retardant treated wood products were tested in the Cone calorimeter. Douglas fir plywood (ASTM round robin) and FRT plywood from FORINTEK. Douglas fir is difficult to impregnate due to its anatomic structure. Distribution of fire retardant is not homogeneous, resulting in a higher concentration near the surface. The non-uniformity

of the distribution is even more pronounced for plywood, because it consists of thin veneers that are separated by a layer of water-resistant (in this case) adhesive. Furthermore many fire retardants increase the hygroscopicity of wood, resulting in an increased water content over the untreated material conditioned in the same atmosphere.

A major problem with evaluation of experimental ignition data for treated wood products was the uncertainty of the time to sustained ignition. Visually determined ignition times showed significant scatter. The use of a criterion based on heat release rate greatly eliminated subjectivity. After some trial and error, 30 kW/m^2 was chosen as suitable limit. Correlations of the ignition times based on this criterion for the FRT Douglas fir plywood is shown in Figure 3. After eliminating ambiguity in the recording of ignition times, the heat release rate data behaved in similar fashion as for untreated wood products. Peak heat release rate turned out to be a reasonable linear function of external irradiance, and no difficulties were encountered in obtaining values for L.

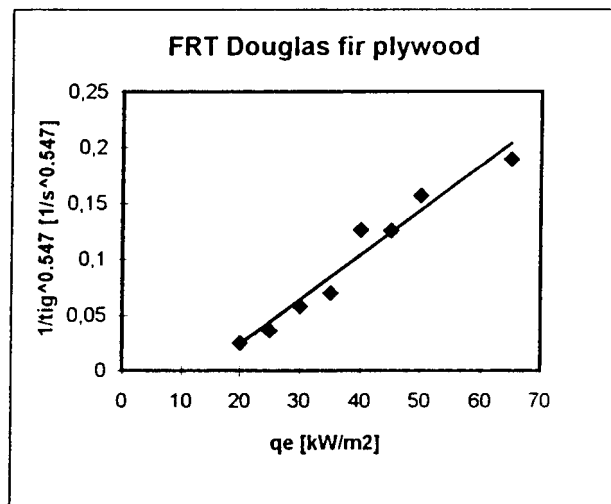


Figure 3: Ignition time correlation for FRT Douglas fir plywood

OTHER MATERIALS

Two non-wood materials were tested; rigid FRT polyurethane foam and paper faced gypsum board (type X). The experimentally observed minimum irradiance level, below which no sustained burning occurred, was approximately 20 kW/m^2 . The critical irradiance level derived from the experimental correlation was 12.6 kW/m^2 . The difference between the minimum and critical irradiance for this type of material was explained earlier by Janssens [8]. At low irradiance levels, fuel volatiles are exhausted prior to the time that the lower flammability limit is reached in the gas phase. At higher irradiance levels, the minimum mass flux of volatiles to create flammable mixture is generated before fuel exhaustion.

The ignition times for the polyurethane foam were very short (between 2 - 12 seconds) and were subject to significant error. The experimentally observed minimum irradiance level, below which no burning occurred was approximately 22 kW/m^2 . The ignition time decreased with increasing external irradiance in the range of 22 to 50 kW/m^2 . At higher irradiance levels the measured ignition time was around 2 seconds. This is the practical lower limit that can be measured in the Cone calorimeter. The ignition parameters were derived from the data measured at external irradiance levels in the range of 22 to 50 kW/m^2 .

TOTAL HEAT RELEASE

The total heat release is important parameter which characterizes the total available energy in the material in a possible fire situation. It can be found as the area under the heat release rate curve, measured in the Cone calorimeter. The burning time and consequently the burnout area of a material in the room/corner test can be calculated based on the total heat release [7]. The correlation of total heat release as a function of external irradiance for Douglas fir plywood is shown in Figure 4. The total heat release slightly increased with increasing external irradiance. Similar correlations were found also for other untreated wood products. On the other hand effective heat of combustion did not change with changing external irradiance (Figure 5).

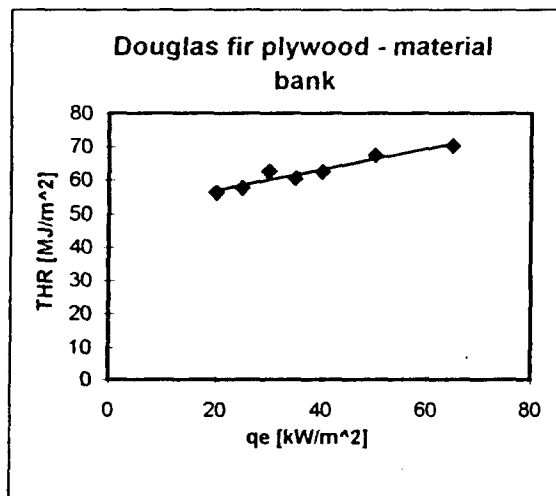


Figure 4: Total heat release vs. external irradiance for Douglas fir plywood (FPL material bank).

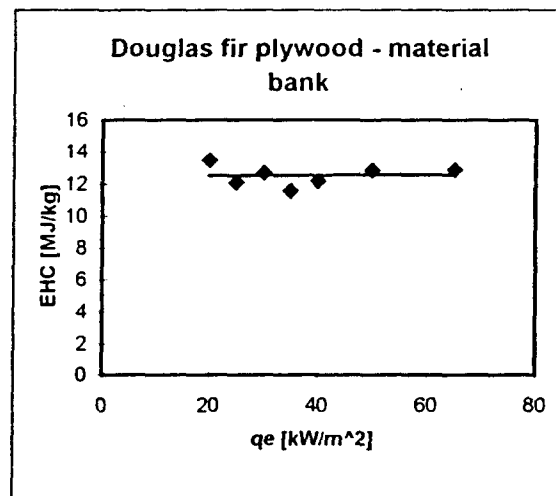


Figure 5: Effective heat of combustion vs. external irradiance for Douglas fir plywood (FPL material bank)

The change of total heat release with the increasing external irradiance was stronger for the fire retardant treated wood products (Figure 6). However effective heat of combustion did not show systematic change as a function of external irradiance (Figure 7).

The only exception from the tested materials was FRT rigid polyurethane foam. The polyurethane foam was treated with fire retardants. One of the effects of the fire retardants was the significant change of total heat release as the function of external irradiance (Figure 8). The effective heat of combustion changed with the change of external irradiance as well (Figure 9)

The increase of total heat release with increasing external irradiance for wood products was due to the fact, that at higher irradiances more mass was consumed by burning, but the potential energy of the volatiles remained the same. At the tested polyurethane foam however the increase of the total heat release was caused also by the increase of the "flammability" of volatiles

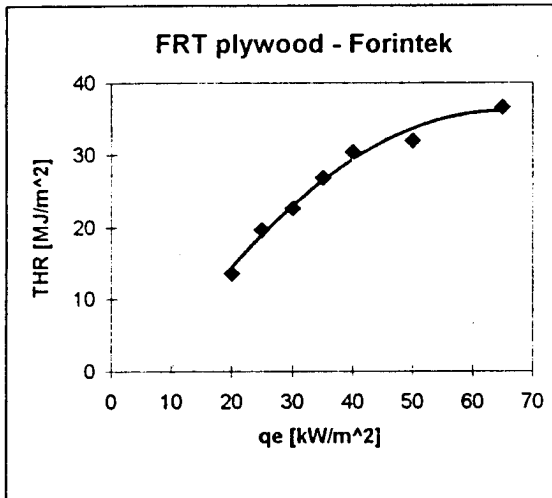


Figure 6: Total heat release vs. external irradiance for FRT plywood Forintek.

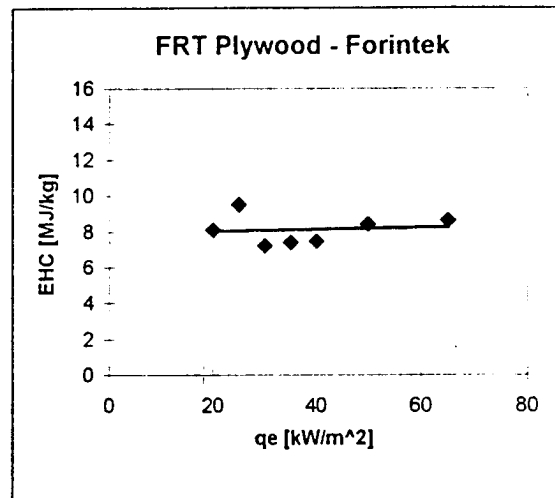


Figure 7: Effective heat of combustion vs. external irradiance for FRT plywood Forintek

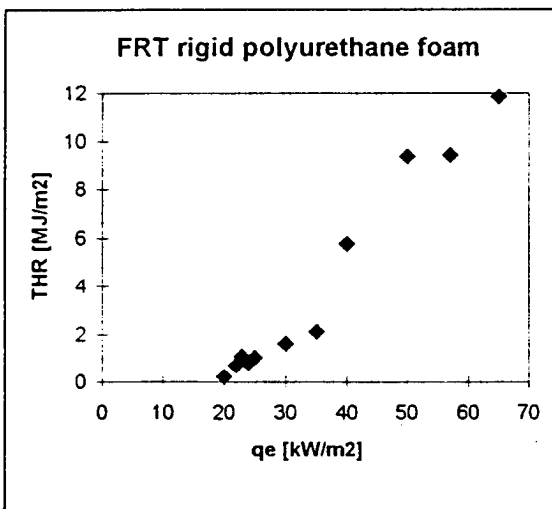


Figure 8: Total heat release vs. external irradiance for FRT rigid polyurethane foam.

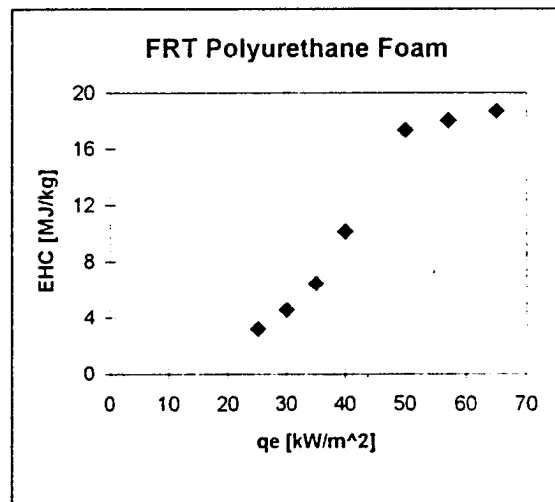


Figure 9: Effective heat of combustion vs. external irradiance for FRT rigid polyurethane foam.

CONCLUSIONS

The effective thermophysical properties were derived for the twelve materials from the Cone calorimeter measurements. For the fire retardant treated wood products the ignition time was determined based on a heat release rate threshold of 30 kW/m². For the wood materials slight increase of the total heat release with the increasing external irradiance was observed. The average effective heat of combustion remained constant in the whole range of irradiance levels for untreated and fire retardant treated wood materials. The FRT rigid polyurethane foam, however showed strong increase of both total heat release and effective heat of combustion with increasing external irradiance.

Table 2: Resulting thermophysical properties of materials.

Material	q_{cr}'' [kW.m ⁻²]	T_{ig} [K]	k_{pc} [kJ ² .m ⁻⁴ .K ⁻² .s ⁻¹]	h_{ig} [W.m ⁻² .K ⁻¹]	EHC [MJ.kg ⁻¹]	L [MJ.kg ⁻¹]	THR [MJ.kg ⁻¹]
D.fir plywood r.robin	13.6	628	0.236	35.6	11.8	4.3	57.3
D. fir plywood mat. bank	15.2	642	0.194	37.0	12.7	5.9	61.0
Oak veneer plywood	8.9	553	0.465	30.1	11.8	4.2	58.8
D. fir plywood FRT r.robin	14.0	635	0.453	36.1	9.3	5.9	30.4
FRT plywood from Forintek	17.3	676	0.449	39.8	6.7	9.3	24.1
Gypsum board type X	12.6	614	0.457	34.5	9.5	7.5	3.3
Polyurethane foam FRT	8.4	545	0.042	29.5	10.1	6.8	5.9
Beech wood	13.7	631	0.504	35.8	11.7	4.3	111.4
Spruce wood	15.0	648	0.208	38.1	12.4	6.1	81.2
Oriented strand board	14.3	638	0.210	36.5	12.2	3.8	83.2
Preswood	16.5	666	0.277	38.9	13.5	4.1	88.7
Redwood	16.0	661	0.184	38.4	13.5	5.5	102.7

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NOMENCLATURE

EHC	Effective heat of combustion [MJ/kg]
h_c	Convection coefficient [W/m^2K]
h_{ig}	Total heat transfer coefficient at ignition [W/m^2K]
k_{pc}	Thermal inertia [kJ^2/m^4K^2s]
L	Heat of gasification [MJ/kg]
\dot{q}_{cr}''	Critical irradiance for ignition [kW/m^2]
\dot{q}_e''	External heat flux [kW/m^2]
RHR	Heat release rate [kW/m^2]
T_a	Ambient temperature [K]
T_{ig}	Surface temperature at ignition [K]
t_{ig}	Ignition time [s]
THR	Total heat release [MJ/m^2]

Greek

ε	Surface emmissivity
σ	Boltzman constant ($5.67 \times 10^{-11} kW/m^2K^4$)

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