CONE CALORIMETER TESTS ON FR TREATED NORWAY SPRUCE
Comparison of Different Fire Retardant Products for Timber Structures

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Abstract
Fire retardants are effective in reducing different reaction to fire parameters of wood such as the ignitability, the heat release, the burning rate and the flame spread. This paper discusses the different mechanisms of fire retardant products as pressure impregnated wood, non-intumescence surface coatings and intumescence coatings on Norway spruce (Picea abies).The tests were performed by using the cone calorimeter test. The comparison of the investigated products will describe the mechanisms of action to reduce combustion by using the heat release rate of 25 kW/m² and 50 kW/m² and the standard IS0 834 test curve. As result information on the ignition time, the heat release rate, the mass loss and the temperature profile over the material thickness will be presented in this paper.

Keywords: fire retardant treatment, intumescent coatings, Norway spruce, heat release rate, cone calorimeter test, oxygen consumption

INTRODUCTION

For centuries, wood has been used in construction both structurally and as a decorative material. Due to its natural combustibility, timber burns if exposed to severe fire conditions. However, wood products can be used safely by improving their fire performance which includes chemical, biochemical and physical modification. At present, the level of knowledge of wood products with improved fire performance is not high enough for their extensive utilization. The main problems are not clear defined technical requirements in building standards and minor existing investigation regarding the long term behaviour under different environmental conditions. Even though some wood products with improved fire performance show excellent fire properties and reach in the European classification system class B, examples of products with hardly any benefit compared to ordinary wood also exist, which typically fall into class D (Hakkarainen et al. 2005). Another problem is the selection of an unsuitable product for a certain application. But the fire retardant treatment, if correctly specified, provides added value to the wood based substrates and extends the market potential of the world's most natural building material.
The purpose of this project is to assist the wood construction with selection criteria for FRT and fire retardant coating products for wood by systematically assembling data on the flammability and other fire related properties of these materials. The ultimate use of such a data base assembly is input for a method for accurately predicting the real fire performance and flammability characteristics of products from bench-scale tests. The cone calorimeter test as used in this investigation is widely used to evaluate the flammability characteristics of materials.

1 MATERIALS

The materials used in this study are listed in Tab. 1. Untreated and treated test specimens made of defect free Norway spruce was cut into 100 mm by 100 mm squares and generally at 30 mm thickness. With exception the samples of FRT-wood (C2-Series) were only in 20 mm thickness available by the supplied product. For each test series three replications were performed. Each test series were cut out of the same three boards s. Fig. 1. The underlying wood was selected in that way to have twin samples and the influence on the natural wood properties is minimized. The samples were prepared for testing perpendicular to the grain orientation. The series of impregnated wood is not fully comparable with the other series as the material was supplied by the providing company. The grain orientation and the material itself (density) do not correspond to the other test samples. Commercial fire retardant products were chosen instead of model formulations so that the effects of single chemicals and other additives are included in the fire performance results. All selected fire retardant products have a classification certificate according the EN 13501 of class B. The compounded formulations were provided by different manufacturers. The coatings were applied by spraying the required amount of 350 g/m² resp. 300 g/m² on the surface. For the FR impregnation a concentration of 20,9 % and an amount of 91,2 kg/m³ was brought into the wood.

Tab. 1 Fire retardant treated Norway spruce - test series

Test series	**Test Replications**	**Fire retardant product**	**colour**	**applied quantity [g/m²]**	**Sample thickness [mm]**
A1	3	intumescent coating	transparent	350	30
A2	3	intumescent coating	transparent	350	30
B1	3	intumescent coating	white	350	30
B2	3	intumescent coating	white	350	30
C1	3	fire retardant solution	transparent	300	30
C2	3	fire retardant treatment	transparent	91,2 kg/m³	20
D (REF)	3	Natural Wood - reference	-	0	30

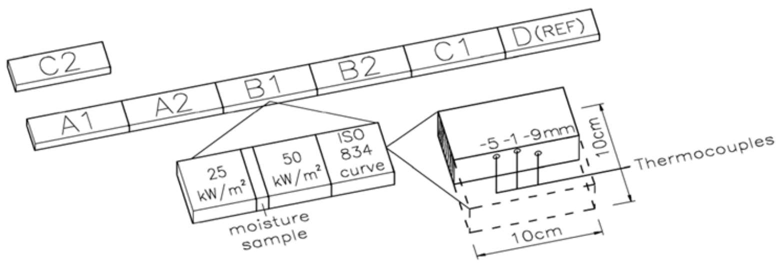

Fig. 1 Schematic representation of the sample preparation procedure

After the coating resp. the impregnation the samples were conditioned at laboratory conditions at 65 % RH and 20 °C for at least for weeks prior to testing to meet equilibrium moisture content (EMC). Before testing the moisture content was determined according to ISO 3130:1975 and the density was determined according to ISO 3131:1975. For the determination of the temperature profile within the sample thermocouples (Type-K) were placed on different depth measured from the exposed surface. The thermocouples were at the depth of -1 mm, -5 mm and – 9 mm. For the cone test the specimen were placed in an aluminium foil with a lip 5 mm above the top surface of the sample.

2 METHODS

The data reported here were obtained using the adapted Conical Heater from FTT (Fire Testing Technology) with respect of the requirements on the Cone Calorimeter Test as described in accordance to the guidance in ISO 5660-1 on choosing a heat flux for cone calorimeter experimentation. The Cone Calorimeter and its function have been previously

described by Babrauskas (1982) and Babrauskas and Parker (1987). Briefly, it is a bench-scale test for determining the rate of heat release based on the principle of oxygen consumption. The energy release rate is computed from the measurements of mass flow rate and oxygen depletion in the gas flow through the exhaust stack. The cone calorimeter brings quantitative analysis to materials flammability research by investigating parameters such as heat release rate (HRR), time to ignition (t_{ig}), total heat release (THR) and mass loss rate (MLR). The HRR measurements can be further interpreted by looking at average HRR, peak HRR and time to peak HRR. Cone calorimeter test results can be used as prediction for the results in the SBI test according to EN 13501-1 (Kristoffersen et al, 2003).
In this study the tests were performed in the horizontal orientation, with the conical radiant electric heater located above the specimen and the retainer frame over the test specimen. The electric spark igniter has not been used in these investigations. The time measured for ignition is the time until the auto-ignition is observed. Heat flux levels of 25 kW/m^2 and 50 kW/m^2 and the standard IS0 834 test curve are used to test the wood products. The real standard IS0 834 test curve was not possible to regulate with the available equipment and therefore a simplified regulation by using target temperatures was used. The IS0 834 test curve was stepwise actuated as shown in Fig.2 for generating the time-temperature curve. For all tests the duration was 600 s.

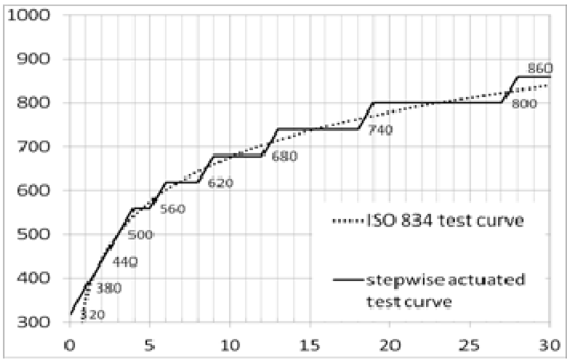

Fig.2 Regulation steps for the standard IS0 834 test curve

A “Specimen shield” is used to prevent radiation exposure to the specimen before the start of the test (t = 0). In the closed position, it completely covers the opening in the heater base plate. The specimen shield is manually opened via a mechanical lever. The start of the test, t = 0 is defined by the moment the specimen shield is opened exposing the specimen to the radiant heat flux. The time to ignition is measured from the start of the test. A load cell was used to continuously measure changes in sample mass, while products of decomposition, (i.e., CO, CO2 and total unburned hydrocarbons) were monitored by appropriate gas analysers. The accuracy of the oxygen measurement is ± 420 ppm. The measurements were logged every second. Heat release calculations were based on the oxygen consumption principle, which states that for complete combustion of a wide range of fuels, 13.1 (± 5 %) kJ of energy is produced for every 1 g of oxygen consumed by the fire (Hugget, 1980). The heat release was calculated according to the ISO 5660 standard as

$$\dot{q}(t) = (\Delta h_c / r_0)(1{,}10)C\sqrt{\frac{\Delta P}{T_e}} \cdot \frac{X^0_{O_2} - X_{O_2}}{1{,}105 - 1{,}5X_{O_2}} \tag{1}$$

and the value of the rate of heat release per unit area is

$$\dot{q}'' = \dot{q} / A_s \tag{2}$$

Nomenclature:

$\dot{q}$ heat rate (kW)

t time (s)

Δh_c net heat of combustion ($kJkg^{-1}$)

r_0 stoichiometric oxygen/fuel mass ratio

C orifice meter calibration constant

ΔP orifice meter pressure differential (Pa)

T_e temperature for orifice meter (K)

$X^0_{O_2}$ oxygen content input

X_{O_2} oxygen content in the exhaust gas

$\dot{q}''$ heat release rate per unit area (kW/m²)

A_s specimen exposed area (m²)

$\frac{\Delta h_c}{r_0}$ is the expression that rates the heat of combustion release per unit mass of oxygen which Hugget (1980) has shown to be sensibly constant with 13.1x 10³ kJ kg^{-1}. The oxygen content $X^0_{O_2}$ is calculated with 0.2095 (± 0.0001). The HRR curves values were calculated from the data recorded on the computer. The results are reported in kW/m² of the exposed surface area. The areas beneath the HRR curves were integrated to give cumulative heat release as total heat release THR in MJ/m² for the test duration of 600 s.

3 RESULTS AND DISCUSSION

The cone calorimeter test results are shown in Tab. 2 as averages values of the three replication tests. Please note that most of the tests performed did not auto-ignite during the 600 s testing.

Tab. 2 Results of the cone calorimeter tests

				Irradiance: 25 kW/m²					Irradiance: 50 kW/m²					Standard time-temperature curve acc. ISO 834				
Test series	Test	Density [kg/m³]	Moisture content [%]	t_{ig} [s]	HRR q''_{180} [kW/m²]	HRR q''_{300} [kW/m²]	HRR q''_{600} [kW/m²]	THR Q''_{600} [MJ/m²]	t_{ig} [s]	HRR q''_{180} [kW/m²]	HRR q''_{300} [kW/m²]	HRR q''_{600} [kW/m²]	THR Q''_{600} [MJ/m²]	t_{ig} [s]	HRR q''_{180} [kW/m²]	HRR q''_{300} [kW/m²]	HRR q''_{600} [kW/m²]	THR Q''_{600} [MJ/m²]
A1	A	563	12,1	-	4,55	4,58	4,72	2,67	-	4,80	10,05	15,96	5,54	-	4,29	10,80	22,54	6,94
	B	572	10,8	-	4,48	9,34	9,35	4,19	-	9,12	13,56	28,34	8,68	-	13,34	22,97	35,62	11,90
	C	511	11,9	-	17,55	22,87	28,19	12,40	-	8,97	13,56	23,31	8,50	-	4,41	4,49	13,16	3,21
A2	A	472	11,6	-	0,05	4,59	4,50	1,73	-	4,74	0,09	8,45	2,77	-	4,23	4,36	4,65	3,14
	B	465	11,2	-	0,05	4,33	4,52	1,89	429	4,81	10,09	56,98	11,30	-	4,47	4,44	9,15	2,93
	C	570	12,7	-	4,50	9,27	9,17	3,80	-	4,52	9,00	15,91	5,68	-	4,38	8,93	22,07	6,00
B1	A	484	11,2	-	9,09	13,60	27,70	8,45	-	13,73	14,96	38,55	12,00	-	4,34	8,71	13,52	4,47
	B	508	10,6	-	9,07	8,99	4,66	5,51	-	8,82	13,84	28,90	10,10	-	4,47	4,58	4,72	2,22
	C	529	12,2	-	8,45	12,92	17,31	7,26	-	9,58	19,34	45,61	11,30	-	10,10	13,50	31,08	8,27
B2	B	486	10,9	-	8,81	8,58	4,46	5,20	-	4,65	9,76	17,55	5,94	-	9,00	9,12	27,64	7,39
	C	579	12,2	-	15,04	16,02	9,23	7,54	-	17,11	13,14	15,74	8,81	-	4,41	8,82	18,12	5,59
	G	473	10,6	-	9,53	8,00	11,97	5,24		8,31	8,79	15,65	6,41	-	6,92	13,53	22,92	6,59
C1	A	508	12,0	-	4,86	10,52	31,62	8,70	71	72,63	74,71	82,43	42,40	-	4,13	8,67	40,47	8,11
	B	517	10,8	-	9,36	20,14	41,52	11,60	62	76,76	82,47	83,97	45,40	-	13,40	18,41	45,69	12,20
	C	548	11,6	-	5,04	15,33	33,74	9,58	-	40,68	50,28	52,54	24,20	-	0,00	4,76	31,51	4,24
C2	A	468	13,1	-	4,45	8,79	9,09	4,32	-	18,99	29,03	46,85	15,90	-	4,33	8,86	18,69	4,93
	B	510	13,0	-	8,81	8,96	4,73	4,92	-	19,20	24,89	42,15	14,40	-	19,00	38,01	62,65	20,80
	C	555	13,8	-	17,51	27,14	35,81	13,70	-	23,67	33,89	49,91	19,40	-	17,61	32,14	66,65	19,80
REF	A	490	10,8	-	11,26	23,18	59,64	15,50	86	72,57	64,50	75,86	38,90	-	8,78	13,40	74,38	13,90
	B	454	11,2	-	22,90	39,70	87,08	24,20	48	105,45	108,40	120,61	62,30	-	9,02	13,96	62,92	12,30
	C	560	11,6	-	6,32	13,62	45,65	9,50	64	108,61	112,68	132,05	63,00	580	4,51	9,57	120,88	8,72

The results of the mass loss rate in Fig. 3 show that at the heat flux of 25 kW/m² and for the standard ISO 834 curve the rates are very similar. However the mass loss rate at 50 kw/m² show higher differences between the tested products. The intumescence coatings in between show quite similar results and the non-intumescence coating and the FR impregnation are in the range of the untreated reference sample regarding the mass loss rate. Note: The mass loss rate at $t_0 = 0$ is not fully correctly given in the diagrams Fig. 3 due to the calculation method used.

The results of the heat release rate in Fig. 4 show that at the heat flux of 25 kW/m² and for the standard ISO 834 curve the rates are very similar. At the heat flux of 50 kW/m² the product C1 show some similarities to the reference but at lower level and the other products have considerable lower heat release rates. By consider the suggestion from Kristoffersen et al (2003) for the prediction of the classification of FRT wood products after running the cone calorimeter test at heat exposure levels of 50 kW/m² for the most of the tested products the class B can be predicted which should be expected as all products have a positive testing certificate as class B according to EN 13501-1. But for the product C1 the results of two samples show HRR > 80 kW/m². This testing results lead to the question whether the product does not fulfil the class B, or the suggested limiting values according to Kristoffersen et al (2003) are not always valid and have to be determined by further tests.

The results of the temperature measurements at the heat flux of 50 kW/m² in Fig. 5 show that at the depth of -1 mm the intumescent coatings show a very clear insulation effect. The temperature at -5 mm and at -9 mm depth rise some questions as the untreated reference series show similar temperatures as the intumescence coated products but the non-intumescence coated and the impregnated product show quite higher temperatures at this depth.

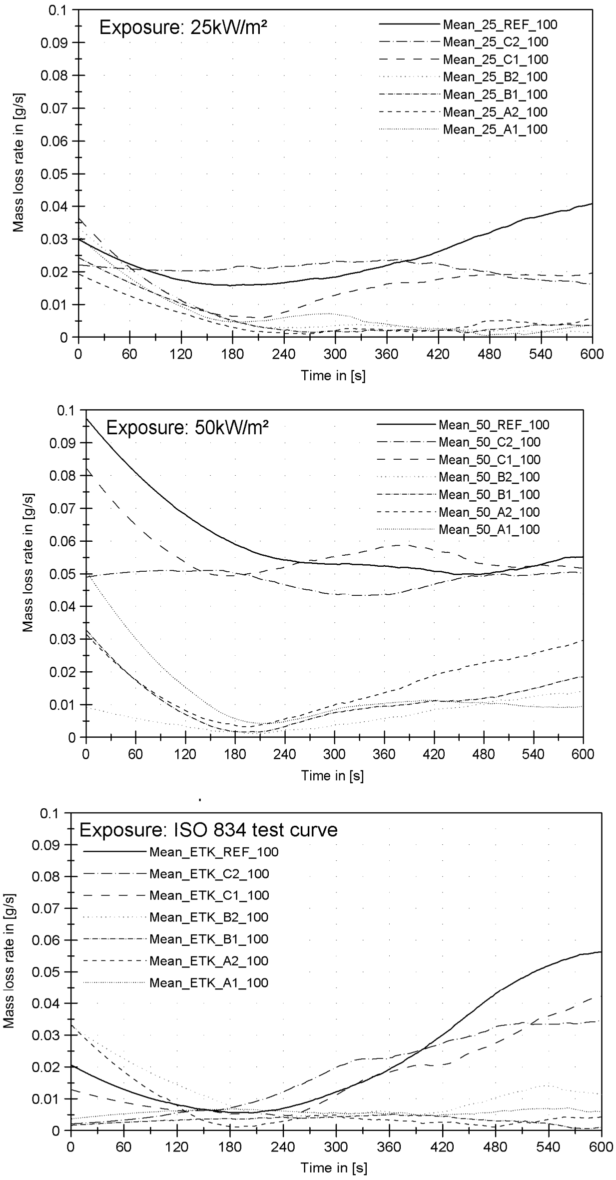

Fig. 3 Mass loss rate curves (mean value of 3 replications) during the cone test with the irradiance of 25 kW/m², 50 kW/m² and the standard ISO 834 curve for the test duration of 600 s for the different fire retardant products and the untreated reference series

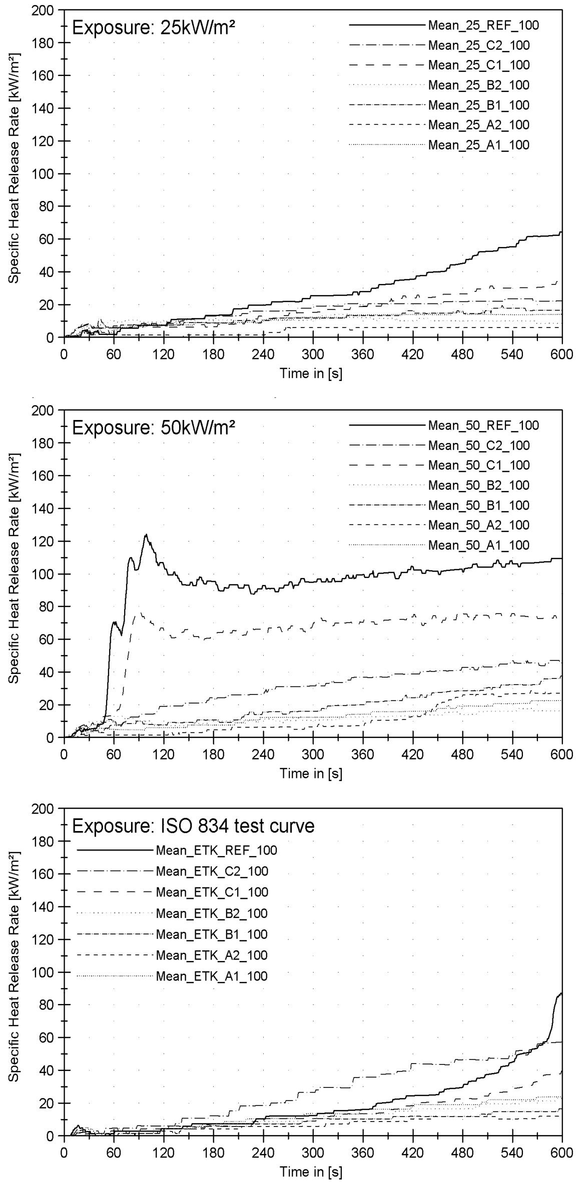

Fig. 4 Rate of heat release curves (mean value of 3 replications) during the cone test with the irradiance of 25 kW/m², 50 kW/m² and the standard ISO curve for the test duration of 600 s for the different fire retardant products and the untreated reference series

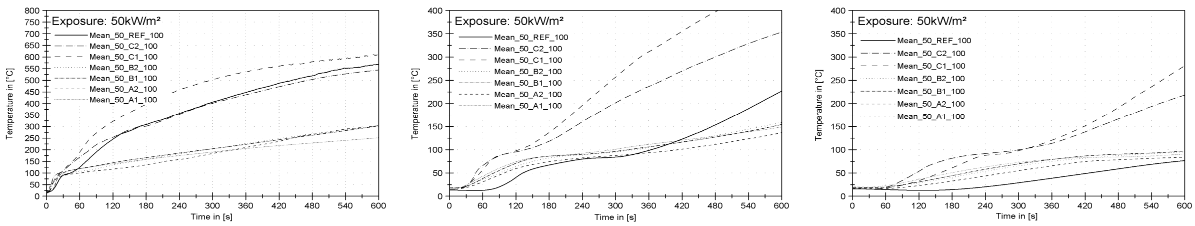

Fig. 5 Temperature profiles (mean value of 3 replications) in the sample at the depth of -1 mm, -5 mm and -9 mm during the cone test with the irradiance of 50 kW/m²

4 SUMMARY AND ACKNOWLEDGMENT

In this paper we have shown the HRR data on various fire retardant treatments on Norway spruce tested in the cone calorimeter. Intumescence coatings on wood reduce significant the HRR and the temperature in the substrate. Almost no difference between transparent and coloured intumescence coatings in the behaviour under heat load could be determined. The non-intumescence coating and the FR impregnation show quite similar results within the cone calorimeter test as the untreated reference sample. Hence the question arises if those products are useful where fire resistance is required for the protection of structural elements. Further the investigations show that the data generated with the cone calorimeter can be used to estimate the fire reaction behaviour according to Euro classes but are limited applicable on intumescent coatings. The Standard ISO 834 curve for 600 s show that the results could be useful for generating information on the behaviour of fire retardant treated wood for simulation purposes as this curve describes a natural fire.

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REFERENCES

Babrauskas V., Development of the Cone Calorimeter A Bench-Scale Heat Release Rate Apparatus Based on Oxygen Consumption (NBSIR 82-2611), (1982).

Babrauskas V. and Parker W.J., Ignability Measurements with the Cone Calorimeter, Fire and Materilas11, 31-43 (1987).

Hakkarainen T., Mikkola E., Östman B., Tsantaridis L., Brumer H., Piispanen P., InnoFireWood: Innovative ecoefficient high fire performance wood products for demanding applications. State of the art, Project Report: Inno Fire Wood, March 2005.

Huggett C., Estimation of Rate of Heat Release by Means of Oxygen Consumption Measurements. Fire and Materials 4, 61-65 (1980).

Kristoffersen B., Steen Hansen A., Hakkarainen T., Östman B., Johansson P., Pauner M., Grexa O. & Hovde P. J. Using the Cone Calorimeter for screening and control testing of fire retarded wood products. Report Nordtest project 1526-01. Trondheim: Norwegian Fire Research Laboratory, 2003. 63 p. + app. 18 p. (NBL A03119.)

ISO 3130:1975, Wood -- Determination of moisture content for physical and mechanical tests

ISO 3131:1975, Wood -- Determination of density for physical and mechanical tests.

ISO 5660:1993, Fire Tests; Reaction to Fire; Part 1: Rate of Heat Release from Building Products (Cone Calorimeter Method).

EN 13501-1:2002, Fire classification of construction products and building elements – Part 1: Classification using test data from reaction to fire tests. Brussels: European Committee for Standardization