Charring rate of intumescent fire protective coated Norway spruce (*Picea abies* L.)

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ABSTRACT: The objective of this study was to determine the combustion properties of Norway spruce (*Picea abies* L.) wood specimens coated with intumescent fire protective coatings of four commercial products in comparison to the uncoated reference material. Two of the products were transparent coatings and two coloured non transparent coatings. The tests were performed with an adapted cone calorimeter by using the heat flux of 25 kW/m², 50 kW/m² and the ISO 834 test curve. The test duration was 30 min. Results indicate that the intumescent coatings reduce significant the charring rate at all irradiance levels in comparison to the uncoated reference material. The temperature profile for intumescent fire protective coated test samples show for all products quite similar results with an insulating effect.

KEYWORDS: Cone calorimeter, fire retardant wood, intumescent coatings, temperature profile, charring rate

1 INTRODUCTION

Due to the composition of wood, which mainly consists of cellulose, lignin and hemicellulose, it burns if exposed to severe fire conditions. As wood is used for structural building elements it will be essential to make wood flame retardant. Basic studies of intumescent fire protective coated wood are necessary to determine the combustion properties of protected structural timber elements. It is well known that materials made of wood can be treated with compounds containing nitrogen, phosphorus, halogens, and baron such as ammonium phosphate and other to improve flame retardance and accelerate the formation of a carbonized layer on the materials [1]. Wood coatings for structural timber elements more often are designed to retard ignition and rate of burn rather than to provide the fire-resistive barrier which is more typical of steel coatings. In the recent years more and more intumescent fire protective coatings for wood are available on the market. For most of the products information on the increased reaction to fire class are available but quite no information regarding the performance of the fire-resistive barrier as required for structural calculations are available. Intumescent coatings offer a passive protection against heat and flame spread by increasing their thickness by 50 to 200 fold under the influence of heat. They build up a multi cellular structure of low thermal conductivity that delivers an efficient heat barrier. In addition endothermic chemical reactions take some heat away from the protected material [2], [3].

Charred wood is bounded by the transition between the pyrolysis layer, the zone where thermal degradation of wood and char formation are actually occurring and the char layer, a zone of cracked charcoal that has no relevant strength or stiffness properties. Charring depth is the distance between the outer surface of the original member and the position of the char-line. The definition of the base of the char layer is widely accepted to be between 280 and 300°C, with 300°C defined in EN 1995-1-2 [4] . The one-dimensional charring rate values for softwoods given in EC5 are 0.65 mm/min and are independent of the density for densities above 290 kg/m³ [4].

The speed at which charring depth advances in the material is called the charring rate and is an essential parameter for fire resistance of wooden structures because it allows the determination of the size of the residual section of wood. Charring rate of wood is dependent of numerous factors, such as wood species, density, moisture, permeability, composition, or direction of burning (along or across the grain).[5]

This study was aimed to determine some combustion properties as the charring rate, the mass loss and the temperature development on the surface of Norway spruce wood samples coated with intumescent fire protective coatings. For the tests the Cone Calorimeter test according to ISO 5660 [10] was used.

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2 MATERIALS AND METHODS

2.1 MATERIAL

One uncoated reference series and four coated test series made of defect free Norway spruce (*Picea abies* L.) were cut into 100 by 100 mm squares with 30 mm thickness. For each test series three replications were performed (s. Table 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 in the tangential direction.

Commercial intumescent fire retardant coatings were chosen instead of model formulations so that the effects of single chemicals and other additives are included in the fire performance results. The products were selected in the way that all fulfil the reaction to fire class B according to the European standard EN 13501-1[7] and have the same required amount to coat. The formulations were provided by different manufacturers. The coatings were spread-coated by applying the required amount of 350 g/m² as required by the technical data sheets on the surface.

After the coating the samples were conditioned at laboratory conditions at 65 % RH and 20 °C for at least four weeks prior to testing to meet equilibrium moisture content (EMC). Before testing the moisture content and the density were determined according to the ISO 3130 [8] and the ISO 3131 [9] standard. For the cone calorimeter test the specimen were placed in an aluminium foil with a lip 2 mm above the top surface of the sample. To determine the protection efficiency of the intumescent coatings thermocouples (Type K) were placed at -1 mm, measured from the surface, to log the temperature during the test. As in the European standard EN 13501-2 [10] the protection time of fire barriers (encapsulation criteria "K" of timber with non combustible materials e.g. gypsum plasterboards) is defined at which time the mean temperature of the protected surface raises over 250°C on the protected surface, this criterion was used for the definition of the protective time.

Table 1: *Overview of the test series*

Test series*	Coating	Test duration (min)		
		25 kW/m²	50 kW/m²	ISO 834 test curve
A1	transp.	30	30	30
A2	transp.	30	30	30
B1	white	30	30	30
B2	white	30	30	30
Ref.	none	30	**15**	30

*For each test series three samples were tested

2.2 METHODS

The tests were performed 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 [10] on choosing a heat flux for cone calorimeter experimentation. 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). But in this study only the mass loss rate and time to ignitions t_{ig} will be presented. The focus in this study is the determination of the thickness of the char layer for intumescent coated wood under different heat fluxes. The results will be used as predictions for wood under real fire scenarios.

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, because the intumescent foam should not be damaged. Heat flux levels of 25 kW/m² and 50 kW/m² and the standard IS0 834 test curve [11]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 stepwise adjusted regulation by using target temperatures was used s. Figure ***1***.

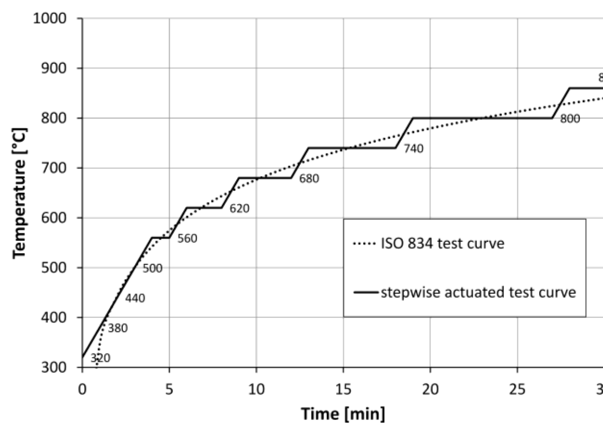

Figure 1: *Temperature regulation steps for the irradiation level according to the IS0 834 standard test curve [11]*

The duration of the tests was 1800 s in general with exception of the reference material at 50 kW/m² for which series the test has to be stopped at 900 s due to the limited material thickness of 30 mm.

The charring was determined by measuring the remained wood to the char-line on three positions in the sample cut in the middle *Figure 2* and then the difference of the mean value to the original material thickness of 30 mm was used as the charring depth. The charring rate was calculated by dividing the charring depth to the test duration.

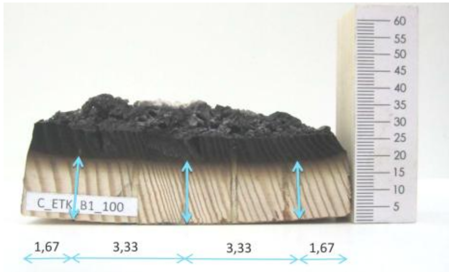

Figure 2: *Measuring positions for the determination of wood char*

3 RESULTS AND DISCUSSION

Moisture content and density: The density and moisture content was determined prior to test by using for both the same sample according to [8] and [9]. The mean value of the density were determined by 515 ± 43 kg/m² and the mean moisture content was 11,4 ± 0,6 %.

Charring rate: The testing results (s. Figure 3 and Table 2) show a reduced charring for all samples coated with intumescent fire protective coatings. By calculating the charring rate the results below have to be carefully used, because the charring rate was calculated on the base of the 30 min resp. 15 min test duration. In real the start of charring for wood coated with intumescent fire protective coatings start after some protective time, which has to be determined in further investigations.

The charring rates at 25 kW/m² irradiance show that the intumescent coating has a high insulation effect and the charring is reduced from 89% (A2 and B2) to 94% (A1) in comparison to the uncoated reference material s. Figure 3.

The charring rates at 50 kW/m² irradiance are reduced from 34% (B1) to 57% (B2) in comparison to the untreated reference material with the charring rate of 0.84 mm/min, which was only possible to test 15 min because of the limited material thickness of 30 mm. Therefore the comparison for this series at 50 kW/m² irradiance to the reference series is limited applicable.

The samples tested under the irradiance level acc. to the ISO 834 test curve show that the untreated reference material with the charring rate of 0.63 mm/min are in good agreement to the charring rate of 0.65 mm/min for softwoods in the EC5. The testing results of the coated samples show reduced charring rates from 36% (B1) up to 54% (A1) for the test duration of 30 min (s. Table 2 andTable 2: Charring rate and time to ignition for intumescent fire protective coated and uncoated Norway spruce under the irradiance level at 25 kW/m², 50 kW/m² and the ISO 834 test curve tested in the Cone calorimeter Figure 3).

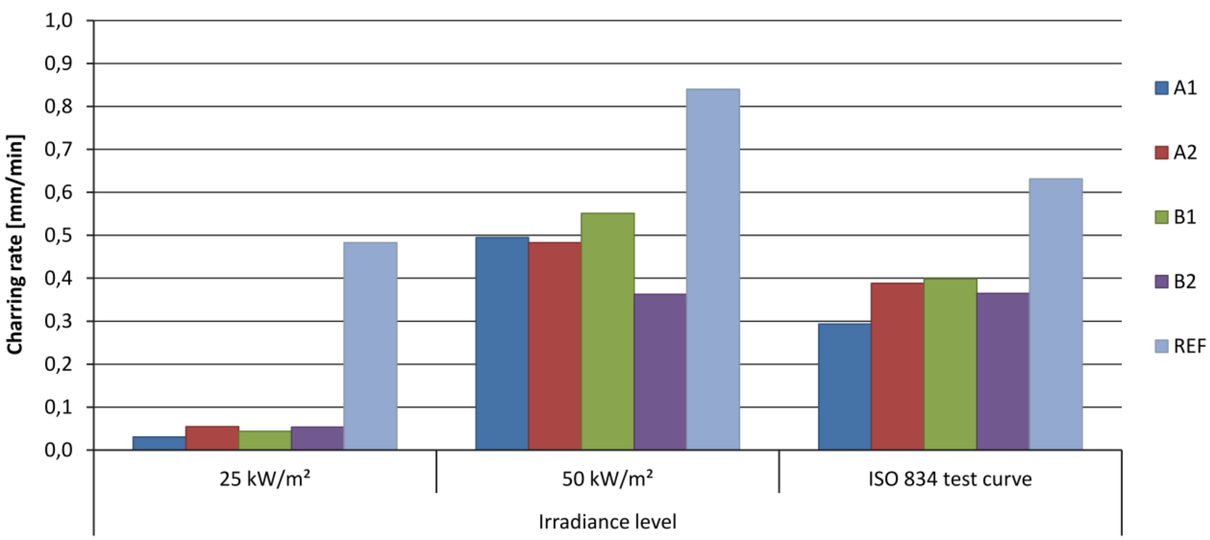

Figure 3: Charring rates of intumescent coated Norway spruce and uncoated reference material at different irradiance intensities tested with the cone calorimeter

Time to ignition: None of the test series did ignite under the irradiance level at 25 kW/m² (s. Table 2). Because the spark igniter was not used, this could be the reason that also the uncoated test series did not ignite under this irradiance level.

As shown in the results in Table 2 it is not clear whether the ignition is restrained due to the intumescent coating or there are also other reasons because in both series with the irradiance level at 50 kW/m² and the ISO 834 test curve the coated material did ignite in general very late and in some cases no ignition occurred e.g. test series B2 at 50 kW/m².

Table 2: Charring rate and time to ignition for intumescent fire protective coated and uncoated Norway spruce under the irradiance level at 25 kW/m², 50 kW/m² and the ISO 834 test curve tested in the Cone calorimeter

		Irradiance level at 25 kW/m²			**Irradiance level at 50 kW/m²**			**Irradiance level acc. ISO 834 test curve**		
Test series	Test sample	Time to ignition t_{ig} [s]	Char thickness [mm]	Charring rate [mm/min]	Time to ignition t_{ig} [s]	Char thickness [mm]	Charring rate [mm/min]	Time to ignition t_{ig} [s]	Char thickness [mm]	Charring rate [mm/min]
A1	A	-	0,86	0,03	1755	14,29	0,48	1741	9,01	0,30
	B	-	1,05	0,04	1253	15,80	0,53	1753	9,33	0,31
	C	-	0,86	0,03	1580	14,46	0,48	1713	8,06	0,27
	Mean value:			**0,03**			**0,50**			**0,29**
	s:			0,004			0,028			0,022
A2	A	-	2,46	0,08	-	14,49	0,48	1382	12,52	0,42
	B	-	1,52	0,05	429	15,25	0,51	1232	12,26	0,41
	C	-	0,94	0,03	1329	13,72	0,46	1203	10,17	0,34
	Mean value:			**0,05**			**0,48**			**0,39**
	s:			0,026			0,025			0,043
B1	A	-	2,16	0,07	-	17,64	0,59	1389	11,67	0,39
	B	-	0,94	0,03	1066	16,86	0,56	1201	12,74	0,42
	C	-	0,81	0,03	1279	15,10	0,50	1452	11,48	0,38
	Mean value:			**0,04**			**0,55**			**0,40**
	s:			0,025			0,043			0,023
B2	B	-	1,32	0,04	-	11,50	0,38	1065	11,88	0,40
	C	-	2,42	0,08	-	11,74	0,39	1703	10,92	0,36
	G	-	1,08	0,04	-	9,41	0,31	1636	10,04	0,33
	Mean value:			**0,05**			**0,36**			**0,36**
	s:			0,024			0,043			0,031
Ref.	A	-	15,12	0,50	86	12,91	0,86*	1232	18,01	0,60
	B	-	15,56	0,52	48	13,34	0,89*	1204	20,50	0,68
	C	-	12,81	0,43	64	11,57	0,77*	580	18,32	0,61
	Mean value:			**0,48**			**0,84**			**0,63**
	s:			0,049			0,062			0,045

* test duration 15 min

Temperature at -1 mm: In Figure 4 to Figure 6 the mean values of three experiments from the temperature at the position of -1 mm, measured from the surface of the specimens, are shown. In general all intumescent coatings show a quite similar thermal insulation effect to protect the wood from heat. The temperature of 250°C at -1 mm was used as reference for the determination of the protective time of the intumescent coatings.

For the irradiance level of 25 kW/m² the reference series reaches the 250°C after about 400 s and the coated series A1, A2 and B2 behave quite similar and protect the wood for about 1200 s and the temperature of the series B2 does not reach the 250°C border during the test duration.

While the temperature in the reference series after about 120 s under the irradiance level of 50 kW/m² the 250°C border exceed, the coated samples go above it after about 420 to 600 s.

Under the irradiation level of the ISO 834 test curve the temperature in the reference series exceed the 250°C border after about 480 s and the intumescent coated series in between 780-900 s.

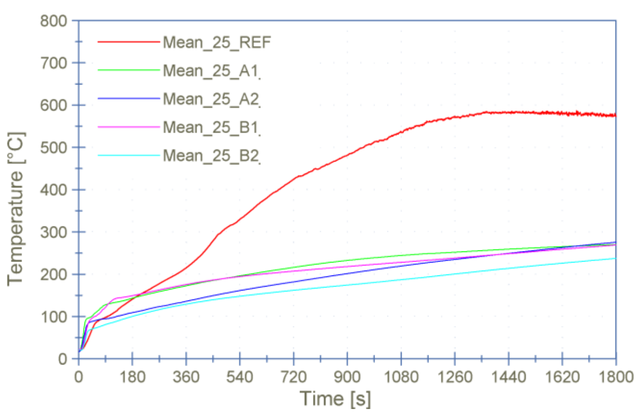

Figure 4: Temperature measured at -1 mm from the coated surface at the irradiation level at 25 kW/m²

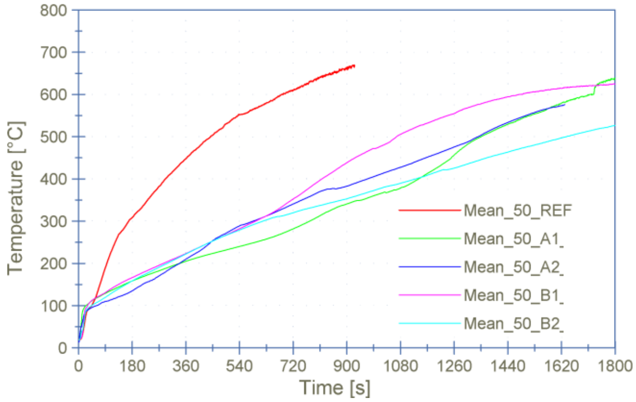

Figure 5: Temperature measured at -1 mm from the coated surface at the irradiation level at 50 kW/m²

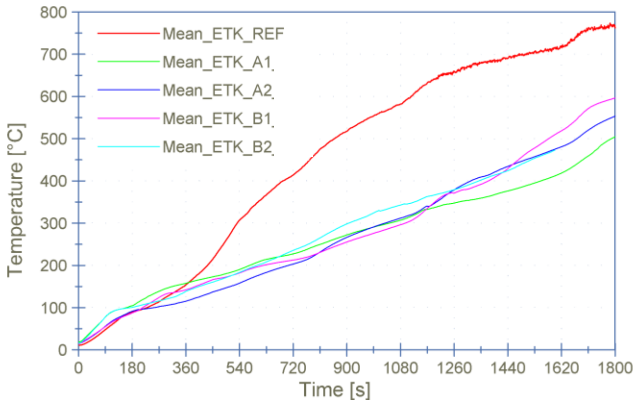

Figure 6: Temperature measured at -1 mm from the surface at the irradiation intensity of the ISO 834 test curve

Mass loss rates: The mass loss rate of the test series are demonstrated in Figure 7 to Figure 9. Within the results of the mass loss rate there can be general stated a significant protection effect of intumescent fire protective coatings.
The mass loss rate of the irradiance level at 25 kW/m² show that all intumescent coatings protect the samples in a similar way during the testing time and very little material is degraded as indicate also the char depth measurements (s. Table 2) in comparison to the unprotected reference material.
The mass loss rate of the irradiance level at 50 kW/m² in Figure 8 show that the coated samples converge after 900 s to the reference material. Also the results of the mass loss rate for the irradiance level acc. the ISO 834 test curve (s. Figure 9) show that after about 1500 s the mass loss rate of the intumescent coated samples converge to the mass loss rate of the reference material.

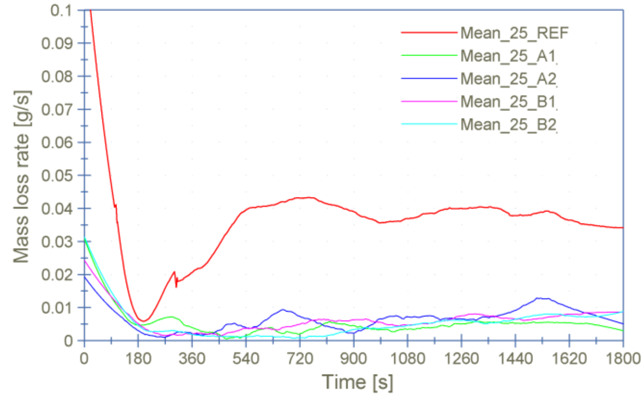

Figure 7: Mass loss rate at the irradiance level at 25 kW/m²

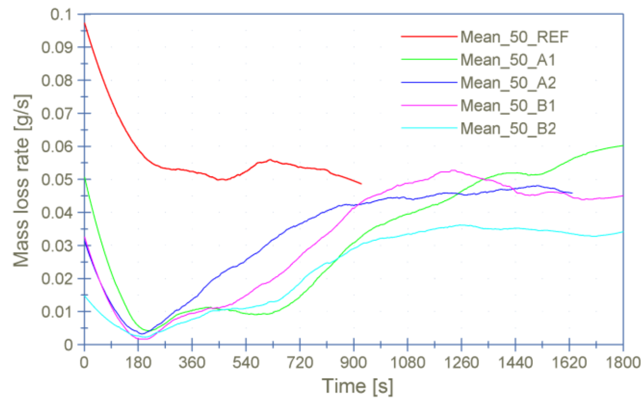

Figure 8: Mass loss rate at the irradiance level at 50 kW/m²

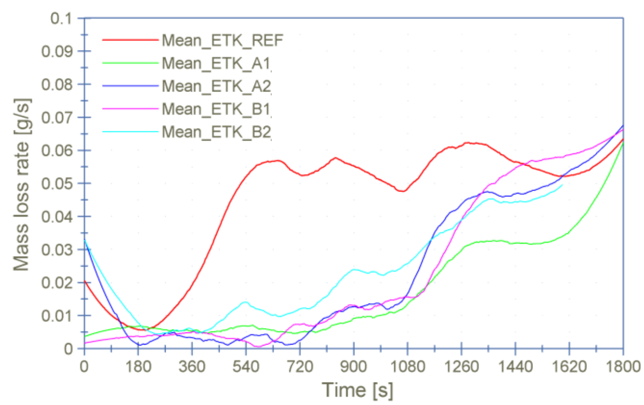

Figure 9: Mass loss rate at the irradiance level acc. the ISO 834 test curve

4 CONCLUSION

Several samples with intumescent fire retardant coating have undergone tests in order to investigate their combustion behaviour. From the tests executed so far it can be concluded that:

- The four investigated intumescent fire protective coatings showed similar fire protection properties in comparison to uncoated reference material.

- The charring rate of the wood coated with intumescent fire protective coatings is reduced at about 35-55% in comparison to uncoated wood for the testing time of 30 min and the heat flux of 50 kW/m² and the ISO 834 test curve.
- The charring rate of coated samples show a reduction of about 90% in comparison to the reference material for the heat flux of 25 kW/m²
- The temperature measurements show an protective effect from the intumescent coating in the range of 5-15 minutes dependent on the irradiance level.
- The mass loss rate of the coated sample converge after about 15 min to the reference material by expose at 50 kW/m² and after 25 min by the irradiance level acc. to the ISO 834 test curve.

By using intumescent fire protective coatings some protection time regarding the charring/decomposition of wood could be demonstrated. Further investigation should focus on the start of char and the charring rates at different time windows and under real fire scenarios for generating data, which could be used for model calculation of the protection time.

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