TESTING OF NATURAL INSULATION MATERIALS USING A CONICAL CALORIMETER

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Abstract: This paper examines three types of natural insulation materials, such as fiberboard, hemp and straw, from the point of view of fire safety. Cellulose-based materials allow a wide range of applications when used for insulation and weatherproofing of buildings, in particular floors, roofs, ceilings, attics, sound barriers, etc. The use of these materials is increasing in ecological constructions as well as for weatherproofing wood-based structures. In terms of fire safety requirements, the question is: Which insulating material is the safest in terms of fire propagation? The article focuses on natural products used as external insulation systems which are covered by a facade plaster. Each type of insulation is briefly described in terms of its composition, use, and production process. We describe the process of preparation of samples as well as the testing and measurement procedures. Three tests were carried out for each type of material. For a more objective evaluation, results were averaged. The results of the cone calorimeter were used to obtain data for comparison. The aim is to clarify the behavior of the natural insulating material with regard to the heat release rate, ignition time, burning duration, and maximum heat release rate. These are the essential parameters for comparison. The values were compared to determine the safest material from the point of view of fire safety.

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Introduction

Thermal insulation is the perfect protection of both load-bearing structures and residential space. External facades should save energy, provide good acoustic protection for people and have aesthetic value. This way, the structures are gradually coated with combustible materials. Building fires can cause huge economical damage to the property of several million euros, but more significantly, many people can die as a consequence. These facts make it necessary for us to study fires and insulation materials for safety as well as for economic reasons. Many people prefer traditional and natural materials, which will gradually replace chemically modified materials. The aim is to minimize negative impacts on both people and the environment. Insulating materials are no exception. The popularity of such materials is increasing especially in ecological constructions. The article deals with the current natural insulation materials, their fire properties, mechanical properties and trends of their use. Cellulose-based materials allow a wide range of applications in the insulation and weatherproofing of buildings, especially floors, roofs, attics, etc. We describe three types of natural insulating materials such as hemp, straw and fiberboard, in more detail. Basic fire-technical characteristics include ignition time, end of combustion, heat release rate, weight loss, and combustion heat. This data is obtained using the cone calorimeter. These methods will provide us with enough values for optimal decision-making and ranking natural insulation material from the point of view of their fire safety.

Test Material

There is a wide range of synthetic or natural thermal insulation materials to choose from at present. Natural materials are mainly composed of hemicellulose, cellulose and lignin. We evaluated the natural products that are used as exterior insulation. Insulation materials in the form of hard boards were used for the test. Three types of thermal insulations, which are often used in ecological constructions, are described below.

Wood-fiber Insulation

Fiberboard is an insulation material made from wood fibers. It is mainly used for structural sheathing insulation of a building. Fiber insulation boards are usually made out of wood fibers (85 %), which are obtained from sawdust and chips of trees, mainly coniferous (Tepore 1 2019).

Hemp Insulation

It comes from a fast-growing annual plant called *Cannabis sativa*. It is highly vapor-permeable (diffusely-open), and provides high protection against cold in winter and overheating in summer (Tepore 2 2019).

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Straw Insulation

The natural raw material used for the production of straw insulation boards are lignocellulosic fibers of annual plants represented by cereal grain crops. Using lignocellulose fibers obtained from annual plants with the addition of cellulosic fibers improves its compactness, which is beneficial for the physical and mechanical properties of the boards (Vesteco 2019).

Sample Preparation

Samples were prepared before measurement. The samples had to be adjusted to the specified dimensions. Three pieces of samples of 100 x 100 mm were made from each type of material. This dimension follows the ISO 5600-1-2002 standard, taking into account the size of the holder. The thickness depends on the manufacturer. The number of pieces corresponds to the number of measurements per material, so that we can average the results for a more objective assessment. Each sample was labelled by a number and weighed. Then the samples were placed into the climate chamber. The climatic chamber Memmert Peltier HPP 260 provided homogenous humidity for all samples. The chamber was set to a constant moisture of 60 % and 23 °C. The samples were kept in the chamber for about 8 days and then they were weighed on the 3th, 5th, and 8th day. If the weight of the samples stabilized, testing could begin.

Method

The necessary data to evaluate the behavior of the material in the burning conditions were obtained using suitable test equipment. The heat release rate was measured using the cone calorimeter method (Kučera 2010). This is an important readout which can determine the behavior of the material in the event of a fire. The range of parameters measured by the calorimeter is very wide and diverse, too. The reason for this is to determine, as precisely as possible, how the product (material) behaves under heat load conditions and how it can contribute to the development of a fire (Kaiser 2010).

A cone calorimeter is a measuring instrument which can determine heat release rate (HRR - Heat Release Rate) using model materials on the basis of oxygen consumption and the measurement of carbon monoxide and carbon dioxide concentration under thermal load during small-scale tests. About 13.1 MJ of energy is released per one kilogram of oxygen consumed (Krajčovičová 2009). This device monitors the creation of heat during the burning process, weight loss rate, and time to ignition. The name is derived from its cone-shaped heating element, which is the source of radiant heat. One of the other reasons we use this calorimeter is that it shows exactly how the products behave under heat load conditions and how the material can contribute to the development of a fire. Consequently, we monitored the following:

- Maximum heat release rate HRR – the basic parameter used to compare or detect a certain material's behavior, as heat release rate generally affects the development of fire (ISO 5660-1: 2002). HRR is one of the basic characteristics of fire which should be taken into account when estimating fire hazard, since it has an important impact on the development of fire in a building (STU 2014).
- Average heat release rate after 60, 180, 360 seconds of test
- Ignition of the sample – the beginning is determined when thermal degradation occurs.
- The end of burning, when the material is burning flamelessly and the sample is only smoldering.
- Total burning length – from ignition up to the end of burning.
- Weight loss

The above mentioned parameters are a simple way to compare the results. The maximum heat release rate can be used to classify the material into groups of materials with similar reactions to the heat flow. The average speeds then allow a similar classification, while during the first 180 seconds of the test the behavior of the material is most interesting and most intense in terms of the values measured (Václav 2015).

Measurement Procedure

- Samples were taken out from the climatic chamber and placed in a polypropylene bag, in which the samples were transferred to a laboratory with a conical calorimeter
- Samples were re-weighed using a digital analytical balance.

- The horizontally placed sample was exposed to a heat of a radiator set to 35 kW/m^2 - 665°C . This was adjusted by means of the respective metal gauge from the edge of the tube assembly, which created a gap of 25 mm from the surface of the sample.
- The aluminum foil of $150 \times 150 \text{ mm}$ was placed into the holder.
- The holder, together with the aluminum foil, was placed on the scales of the cone calorimeter and they were tared.
- The foiled sample was mounted into the holder, and non-flammable plates of approximately 10 mm were placed underneath the sample, so that the sample touched the top of the cover. The weight of the sample was re-checked to ensure it corresponded with the pre-weighing parameters.
- The humidity and ambient temperature of the environment was checked and the values recorded into the computer.
- The data included the name of the sample, its size and thickness, the humidity of the environment, ambient temperature, the material the mat is made from.
- The sample was then placed on the scales. The program was launched, we waited for 1 minute, initiated the spark for ignition, removed the cover of the radiator and waited until the sample ignited. If flame burning occurred, we pressed start.
- The weight loss was recorded in one-second intervals by the computer.
- The burning process was monitored visually until flameless burning occurred.
- Finally, we continued recording the data for 60 more seconds.

The test equipment allowed us to obtain all information about the tested materials. Based on the parameters obtained (e.g. ignition time, weight loss, heat release rate), we were able to separately analyze the selected types of natural insulating materials. The choice of specific parameters was derived from the fact that every single parameter can reliably characterize the behavior of the material under thermal load conditions.

Measurement Results

We determined the flammability rate - the ability of building materials to ignite, burn and smolder and thus contribute to the development of fire (Vacula, 2007) - using a cone calorimeter and measuring the ignition time, flameless burning time, total test time, peak heat release rate, maximum weight loss, total heat released, HRR in 60, 180 and 360 second, total weight loss. The first tested sample was the hemp insulation sample. The values are shown in Table 1. The heat flow applied to the test material was 35 kW/m^2 , this value corresponds to a temperature of 665°C . The initial sample weight was 28.93g and the dimensions were $950 \times 930 \times 140 \text{ mm}$. The air temperature was 25°C and humidity 30%.

Table 1: Test Results Hemp insulation					
	Hemp insulation		35 kW/m ²		
	Times of the test	parameter	unit	value	
		ignition time	s	9	
		flameless burning time	s	527	
		end of the test	s	675	
	Peak values	parameter	unit	value	time (s)
		HRR	kW.m ⁻²	195.41	27
		Wight loss	g.s ⁻¹ .m ⁻²	0.17	17
	Test Results	parameter	unit	value	
		total heat release rate	MJ.m ⁻²	35.58	
		average HRR after 60 s	kW.m ⁻²	127.04	
		average HRR after 180 s	kW.m ⁻²	92.86	
		average HRR after 360 s	kW.m ⁻²	75.34	
average weight loss rate		g.s ⁻¹ .m ⁻²	5.44		
	weight loss	%	11.94		
Source: Author (2020)					

Graphic results are shown in Fig. 1. In the first part, the curve reaches negative values. This means that within 10 seconds from the start of the measurement, the sample consumed 21.55 kW/m^2 of heat from the radiator. This amount was needed for the initiation. During heat consumption, gas and vapor were released (Durica 2010). Their correct concentration caused the sample to ignite in the 9th second, and flame burning occurred. Flame burning means a flare-up, when flammable surface products, which were

created by wood thermolysis, oxidize in the gaseous phase near the surface of the wood and are relatively exothermic, and the reaction heat is only partly removed from the system (Kupilik 2006). After the specimen ignites, the curve achieves a rapid increase in heat release values. Within 18 seconds from ignition, a sharp increase in HRR is observed, which is reflected in the perpendicularity of the curve. The maximum HRR is 195.41 kW/m^2 in the 27th second. The rapid increase ends at its peak and gradually decreases. The highest average HRR of 127.04 kW/m^2 was reached within the first 60 seconds. The burning slightly decreases following the maximum HRR. In the 527th second, flame-free burning was observed, as well as smoldering (Balog 1993). Smoldering is the burning of material without any visible light effects which are caused by temperature rise and formation of smoke (Herecova 2012). At 545 seconds, we observed a slight increase in the curve due to the appearance of a small flame. The test lasted 675 s, during which 11.94 % of the sample burned away.

Figure 1: HRR – Hemp insulation

Source: Author (2020)

After burning away, the sample remained in its entirety, increased its volume, and consequently formed into a lump.

The second material tested was the straw insulation. The measured data are given in Table 2. The heat flow the test material was exposed to was 35 kW/m^2 ; this value corresponds to a temperature of 665°C . The initial sample weight was 99.61g and the dimensions were 103x101x36 mm. Air temperature was 25°C and humidity 30 %.

Table 2: Measurement results

	Straw insulation		35kW/m ²	
	parameter	unit	value	
Times of the test	ignition time	s	12	
	flameless burning time	s	1620	
	end of the test	s	1740	
Peak values	parameter	unit	value	time (s)
	HRR	kW.m ⁻²	129.27	25
	weight loss	g.s ⁻¹ .m ⁻²	0.13	83
Test results	parameter	unit	value	
	total heat release rate	MJ.m ⁻²	75.04	
	average HRR after 60 s	kW.m ⁻²	89.62	
	average HRR after 180 s	kW.m ⁻²	68.54	
	average HRR after 360 s	kW.m ⁻²	56.66	
	average weight loss rate	g.s ⁻¹ .m ⁻²	5.19	
	weight loss	%	41.83	

Source: Author (2020)

Figure 2 shows the HRR of straw insulation. Once again, we can see that the curve starts in negative values. This is the amount of heat consumed 7.55 kW/m^2 , 10 seconds from the beginning of the test. Then, at 12 seconds, a heterogeneous burning occurs. It is interesting that the curve consists of two peaks.

At 25 seconds, it reaches its first peak with a maximum HRR value of 129.27 kW/m^2 . It decreases again and maintains, for approximately 1200 seconds, the HRR ranging from $40\text{--}60 \text{ kW/m}^2$. This fact means that the layers of straw are gradually burning away. In the production process, the sample is joined together (two parts) to obtain the required thickness. At 1400 s, the HRR rises up to 100.98 kW/m^2 ,

which is represented on the graph by the second peak. This HRR is slightly lower (by 30 kW/m²) compared to the first peak. Homogeneous combustion started at 1620 s. The test lasted 1740 s, during which the weight decreased 41.83 % from the original weight.

The results of the third measurement are shown in Table 3. The sample dimensions were 980 x 980 x 40 mm; initial weight of the sample was 103.29 g.

Figure 2: HRR – Straw insulation

Source: Author (2020)

Figure 3: Straw sample – a) before the tes b) after the test c) disintegration of the sample after burning

Source: Author (2020)

Table 3: Measurement results

	Fiberboard insulation		35 kW/m ²	
	parameter	unit	value	
Times of the test	ignition time	s	11	
	flameless burning time	s	2,166	
	end of the test	s	2,240	
Peak values	parameter	unit	value	time (s)
	HRR	kW.m ⁻²	155.93	30
	weight loss	g.s ⁻¹ .m ⁻²	0.12	22
Test results	parameter	unit	value	
	total heat release rate	MJ.m ⁻²	109.73	
	average HRR after 60 s	kW.m ⁻²	119.33	
	average HRR after 180 s	kW.m ⁻²	88.79	
	average HRR after 360 s	kW.m ⁻²	74.55	
	average weight loss rate	g.s ⁻¹ .m ⁻²	4.14	
	weight loss	%	33.64	

Source: Author (2020)

Figure 4: HRR – fiberboard

Source: Author (2020)

Fiberboard is composed of two parts to achieve the required insulation thickness. The two-part composition is projected in the HRR graph fig. 4 in the form of two peaks. The amount of heat consumed was 5.23 kW/m^2 . The first peak is more pointed than the second one. The initiation of the sample occurred in 11 seconds. The increase in HRR is very steep. Its value is 155 kW/m^2 and it is reached in a short time frame of 30 s. The middle part of the curve is kept below 60 kW/m^2 , the sample layer is burning away in the same way. Heterogeneous burning occurred at 2166 s. The test lasted 2240 s. Weight loss from the initial sample weight was 33.64%.

Comparison of Measurements

At first glance, the curves differ significantly. They all have a rapid increase in heat release rate. From the curves in Figure 5 of all samples, it can be seen that the hemp sample (red) absorbed the most heat during the preparation phase. This was reflected by the shift of the curve on the y-axis to negative values -27.55 kW/m^2 . On the other hand, fiberboard's heat absorption was the lowest (Netopílová 2010). The moisture content of the samples was 60 %. During exposure, moisture evaporates

Figure 5: Curve comparison chart

Source: Author (2020)

Figure 6. Average HRR in 60, 180, 360 second

Source: Author (2020)

The density of the hemp sample is different from the other two insulations. Hemp is less compressed and more spaced. It can be concluded, then, that the density of the sample has a major effect on burning. The moisture evaporates more quickly through the pores of the sample, which is not compressed. For such a sample, the initiation phase comes earlier than for a sample with a greater density. The graph (fig. 5) shows a significant difference in the ignition of the samples. The first one to start burning was a hemp sample in 9 s followed by a fiberboard sample in 11 s and finally the straw sample in 12 s. After ignition, a rapid increase in HRR occurred equally in all three samples. The maximum HRR was recorded for hemp samples 195.41 kW/m^2 , over a period of 27 seconds. The lowest HRR was reached for straw samples (green) and was only 129.27 kW/m^2 at 25 seconds. The straw and fiberboard sample is composed of two parts which are glued in the center. First, one layer must burn away and then the second follows. Therefore, there are two peaks appearing on the graph. Hemp insulation does not consist of two layers. The measurement detected only one HRR peak. At 527 seconds, hemp samples started burning heterogeneously, showing no signs of flame burning. The longest duration of the test was 2240 s for fiberboard. The HRR for each sample in 60,180,360 s is shown in Figure 6. We can see that hemp, due

to its structure, composition, and density, contributes most to the development of a fire. In each time interval, it has the highest HRR value. The second sample, with a slightly lower HRR, is the fiberboard sample. Among the three materials, the straw sample has the best results. Its composition does not contribute to the development of fire as much as the other samples.

Conclusion

Each type of insulation showed different behavior patterns during burning. The main objective was to assess, on the basis of the tests performed, the behavior of the selected insulation materials under thermal load conditions using a cone calorimeter. Our intention was to create a clear overview of the burning development of natural insulation materials through the HRR curve. Each type of insulation tested was described based on heat release rate values. Finally, an overview of materials was created and then compared in a single chart. Although all the materials consist of cellulose, lignin, and hemicellulose, their burning behavior varies. The evaluation consisted in finding the differences between the samples. They showed different characteristics in many parameters. This difference stems mainly from differences in structure and composition, although they contain the same substances. The hemp sample had the shortest ignition time out of the three types of insulation. Its weight loss percentage was the lowest – 11.94 % - from these samples. Straw insulation has the lowest heat release rate, so we can thus conclude that out of the three sample types, it contributes to burning the least. Ignition of the sample occurred 3 seconds later than in the hemp sample. Weight loss was 41.83 %. This confirms the greatest resistance of the insulation to thermal degradation. On the other hand, the observed HRR parameter and ignition time showed that hemp has the greatest impact on burning behavior. The behavior of the material or the product in the phase of fire development can therefore be very reliably assessed by monitoring its behavior under radiant heat flow. However, we must emphasize that this is only a relative comparison, since the test conditions and the conditions differ in a real fire. In a real fire, it is justifiable to assume there is an intensive flow of gases (including air), which also has a significant impact on the time of initiation.

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