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FIRE BEHAVIOR OF BIO-BASED EARTH PRODUCTS FOR SUSTAINABLE BUILDINGS

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Abstract
Construction is one of the most polluting industrial sectors. For this reason, developing sustainable building materials is a world-wide interest. Earth as a building material is thus increasingly studied because of its low environmental impact and its ability to regulate indoor moisture and to improve occupant's comfort. Moreover, earth is a well-known non-combustible material. Recent studies deal with unfired earth bricks with plant aggregates incorporated to the earth matrix in order to lighten and improve various properties of the composite materials. However, these vegetal additions, of combustible nature, raise questions about the fire-behavior of the bio-based material. To our knowledge, the fire-behavior of this kind of materials has been little investigated in the literature. The present paper constitutes a preliminary study about the fire reaction of earth bricks containing 0%, 3% and 6% weight content of barley straw or hemp shiv. A pyrolysis combustion flow calorimetry (PCFC) was done to predict the fire-behavior of the plant aggregates. Other tests were performed on the composites to determine their flammability, their thermal insulation and their mechanical strength after high temperature exposure. The following conclusions were reached. The PCFC test has shown a peak of heat release rate around 350°C for the plant aggregates, which corresponds to the degradation of the cellulose. The ignition-extinction test has proved that the material is still non-flammable even with vegetal additions. Thermal conductivity of the composites decreased when the experienced temperature increased (until 800°C) due to the higher porosity. The higher the plant aggregates content, the more fire resistant was the material, meaning that the rise of temperature was delayed. Concerning mechanical performance, a strength drop was observed for composites around 400°C before a slight increase until 800°C thanks to the firing of the earth.

Keywords:
Bio-based building material; Earth bricks; Fire resistance; Barley straw; Hemp shiv

1 INTRODUCTION

Sustainable construction is a major concern. In the building sector, a growing interest has been observed during the last decades within ecological materials, namely renewable, energy efficient or healthy.

Earth-based material is therefore part of a sustainable solution. This material, increasingly studied, is abundantly available, recyclable and requires little energy for the transformation process [Minke, 2006]. It is moreover able to regulate indoor moisture and presents a good thermal inertia. Some properties such as thermal insulation or mechanical strength can however be improved by adding some plant fibers or aggregates in the earth matrix [Laborel-Préneron, 2016].

Developing a « new » material requires to characterize its basic properties. The verification of the safety and comfort of the building's user is also necessary. Some hazards may indeed occur on a building that can lead to reconsider these two criteria. It is the case, for instance, in a fire situation. The fire risk is high in a dwelling with various factors triggering such as electricity networks, chimney fires or lightning strike. Consequences can be severe, notably in case of collapse, if the resistance to fire of the materials was not adapted.

The material is suitable for building construction if it prevents fire from spreading and if the stability of the construction is ensuring enough time to potentially evacuate the occupants. Fire safety regulations vary from country to country, but they are generally based on international standards such as ASTM E119 [ASTM, 2000] or ISO 834 [ISO, 2014]. In Europe, the standard Eurocode 6 - EN 1996-1-2 [CEN, 2005] focuses on the fire design of masonry structures. For testing fire resistance, the European standards EN1363-1, EN 1364-1 and EN 1365-1 [CEN, 1999a, 1999b, 2012] are used.

These regulations request that the tests to assess the load-bearing capacity are realized at actual size (on walls for example). Still, this preliminary study is realized at small scale to estimate the fire behavior of this type of material, very poorly studied in the literature. The earth, as a mineral material, is a non-combustible material. However, adding straw or other vegetal particles, which combust readily, can raise some issues. According to the German Standard DIN 4102-1 [German Standard, 1977], even with some straw content, as long as the earth-based material is higher than 1700 kg.m^{-3}, the material is still considered as non-combustible. The present study focusing on adding high vegetal aggregates content in the sample, it is thus important to evaluate the fire behavior of such materials, which bulk densities are lower than 1700 kg.m^{-3}.

Few studies exist on the fire behavior of bio-based earth materials. Buson et al. showed a good fire resistance of walls made of compressed earth blocks of soil-cement and cellulose pulp derived from the recycling of cement sacks [Buson, 2013]. The characterization of straw and earth to fire was also realized by [Apte, 2008]. The effect of high heat sources on two straw bales rendered by earth or lime of 45 and 43 mm thick respectively was studied. When the material was subjected to a heat of 30 kW.m^{-2} during 30 minutes, it resulted in little damage to the render layer and no combustion of the straw bale. However, when subjected to 50 kW.m^{-2} during 40 minutes, the combustion of the straw was initiated 24 hours after the end of the exposure regime. The render has played the role of a barrier to oxygen transfer into the straw.

Fire behavior studies of bio-composites often focus on insulated materials, mostly composed of organic matter, such as rice husk with alginate for example [Palumbo, 2015, 2017]. It also deals with polymer composites with natural fibers. The effect of a fire retardant on a bio-composite of polybutylene succinate with natural fibers was studied in [Dorez, 2013]. The fibers decreased the thermal stability but the increase in mass residue led to the creation of a char barrier. Fire behavior of another type of bio-based material, made of cellulose derivatives and starch matrix and sisal fibers was investigated by [Alvarez, 2004]. It seemed that the addition of fibers did not influence a lot the fire resistance of the material.

In this research, various tests were realized in order to evaluate the fire reaction and fire resistance of an earth-based material containing 0%, 3% or 6% by mass content of straw or hemp shiv. First, small-scale flammability tests were carried out on the plant aggregates only. Other thermal degradation tests were realized on the bio-composites to evaluate their flammability, their residual thermal insulation and compressive strength after a high temperature exposure. These last two tests are part of the fire resistance characterization, composed of three criteria (stability, insulation, sealing) while the two firsts define the fire reaction, indicating the way the material behaves as a combustible.

2 MATERIAL AND METHODS

2.1 Materials and manufacturing

Fines from limestone aggregate washing process (FWAS) were used for this investigation. These fines have a high proportion of limestone (around 60%) and only around 20% of clay. Barley straw and hemp shiv were also tested in the earth matrix in different proportions: 3% and 6% by dry mass. The manufacturing water content was determined with the Proctor test. Formulations of the different types of samples are recapitulated in Tab. 1.

To manufacture the specimens, earth and plant aggregate fractions were poured into a blender and mixed by hand. Water was then added and the materials were mixed mechanically in the blender until a homogeneous mix was obtained. The raw materials were mixed the day before molding.

The prismatic samples of 15 x 15 x 5 cm^3 were manufactured by static compression. They were then dried at 40°C for 24 hours followed by an increase in temperature until 100°C at a rate of 0.1°C.min^{-1}. This temperature was finally kept constant until the weight became constant (weight variation less than 0.1% between two weighings 24 hours apart).

For purposes of the different tests, the samples were cut with a circular saw into different sizes:

- 7.5 x 7.5 x 5 cm^3 for ignition time and extinguishability and thermal conductivity tests;
- 5 cm cubes intended for compressive strength test;
- 15 x 10 x 5 cm^3 for the fire resistance test.

Tab. 1: Mixture proportions and density of the samples

Ref.	FWAS	S3	S6	H3	H6
Aggregate	-	Straw	Straw	Hemp	Hemp
Aggregate content (%)	0	3	6	3	6
Water content (%)	14	19	21	17	20
Dry density (kg.m^{-3})	1988	1520	1195	1553	1190

2.2 Pyrolysis Combustion Flow Calorimetry

Small-scale flammability was characterized with a Pyrolysis Combustion Flow Calorimeter (PCFC) from Fire Testing Technology. Some fire reaction properties were predicted thanks to this device. In that experiment, thermal analysis was combined with oxygen consumption calorimetry. The temperature range was between 100°C and 750°C and was increased at a rate of 1°C.min^{-1}. Products from the anaerobic thermal degradation completed in a nitrogen atmosphere were mixed with a 20 cm^3.min^{-1} stream of oxygen prior to entering the combustion furnace at 900 °C. Heat release rate (HRR) was then determined from the oxygen consumption. The measurement was conducted on two specimens of each type of plant particle which mass was around 5 mg (balance at one-hundredth milligram).

2.3 Ignition time and extinguishability

This fire reaction test, based on the Spanish standard UNE 23-725 [AENOR, 1990] and its French counterpart NF P92-505 [AFNOR, 1995], highlights the flammability of the material. The test protocol involves placing the sample below a radiator that act as a source of heat of around 500 W. The radiator has to be removed if the sample undergoes an ignition and replaced when the flame is extinguished.

The parameters determined are the first ignition time, the number of ignitions and the average value of combustion extent during the first 5 min of assay. In this case, only one test was performed on each formulation.

2.4 Thermal conductivity

This test provides information about the insulation aspect of the material fire resistance. Specimens were heated up at various temperatures until 800°C following the temperature curves of Fig. 1. Their residual thermal conductivity was measured with a thermal properties analyzer Quickline-TM 30 Anter Corporation, which uses a dynamic method of measurement. Thermal conductivity values were measured the day after their exposure to high temperatures. The size of the specimens tested in that study was 7.5 x 7.5 x 5 cm^3.

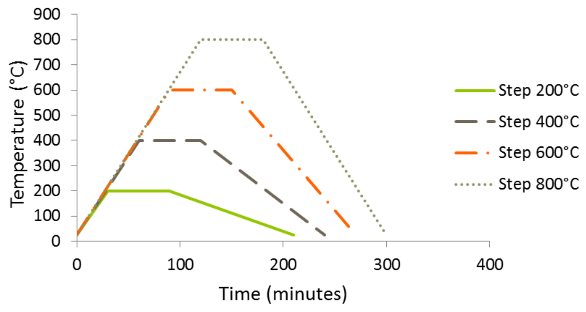

Fig. 1: Temperature curves

2.5 Compressive strength

Measuring compressive strength allows determining the fire resistance of a material and more specifically its stability. Compressive strength of 5 cm cubic specimens was measured after high temperature exposure. The four temperature steps are illustrated in Fig. 1. This test was carried out by means of a hydraulic press Wyhehan Farrance Ltd. 5000 kg the day after exposure. The load was applied at a constant deflecting rate of 3 $mm.min^{-1}$. Two to three specimens for each formulation were tested.

2.6 Temperature of the unexposed face test

This test is part of the fire resistance characterization and identifies the capacity of a material to prevent heat spreading. One face of a 15 x 10 x 5 cm^3 sample was exposed to high temperatures. This face was directly submitted to an increase in temperature: 2 hours of increase until 800°C then 2 hours stable at this temperature. The other face, unexposed, is stuck out of 1.3 cm in a room at 25°C. The temperature was measured by a thermocouple inside the oven and 2 other thermocouples were in direct contact with the middle of the unexposed face. Lateral sides of the sample outside the oven were thermally insulated with mineral wool (Fig. 2). The heat flow is thus considered one-dimensional.

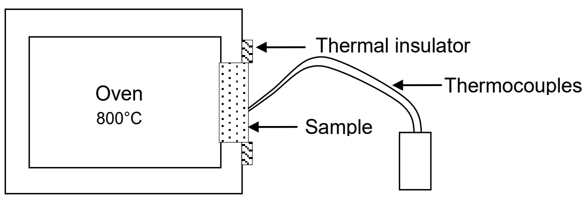

Fig. 2: Temperature of the unexposed face

3 RESULTS AND DISCUSSION

3.1 Pyrolysis Combustion Flow Calorimetry

Two tests for each type of plant aggregate were realized: barley straw (Straw1 and Straw2) and hemp shiv (Hemp1 and Hemp2). The Heat Release Rate (HRR) is plotted as a function of the temperature in Fig. 3. A single peak of heat release is observed around 350°C for each vegetal particle type. This corresponds to the thermal decomposition of the cellulose [Alvarez, 2004]. A significant gap can be noticed between the two measurements of the hemp shiv which does not allow concluding about a precise value. The result of a PCFC test on barley straw published by Palumbo et al. is also plotted [Palumbo, 2015]. It can be observed an important difference between the two straws: the straw studied in Palumbo et al. presents a lower HRR. The results could be influenced by the chemical composition of the particle type or the moisture content.

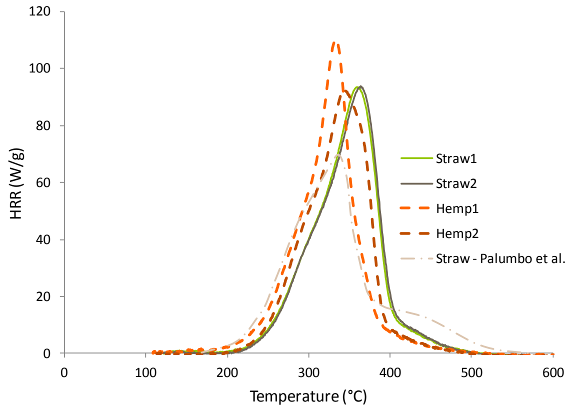

Fig. 3: Heat Release Rate as a function of temperature for the plant aggregates

Other parameters can be deduced from this test. They are recapitulated in Tab. 2.

- The total Heat Release (HR), corresponding to the integral of the Heat Release Rate;
- The temperature of start combustion (T_0), which is considered from a HRR of 10 $W.g^{-1}$;
- The temperature corresponding to a maximal HRR (T_{max});
- The Peak of Heat Release Rate (PHRR);
- The mass loss fraction during the combustion (M_{loss})

Results are very similar between straw and hemp, with a total heat release very close to each other. However, the temperature of the degradation of the particle is slightly lower for hemp than straw and the peak of HRR is slightly higher for hemp but this is due to the scattered results of the hemp particles. The mass loss fractions of the two particle types are very similar.

Tab. 2: PCFC results for the plant aggregates

Raw material	HR (MJ/kg)	T_0 (°C)	T_{max} (°C)	PHRR (W/g)	M_{loss} (-)
Straw	8.3	255	362	93.8	0.83
Hemp	8.2	244	339	101.3	0.81

3.2 Ignition time and extinguishability

During the test, no flame was observed, even on samples containing plant aggregates, which is highly positive. The whole protocol described in the standard was thus not adapted to this type of material. However some observations could be made. The smoldering of straw and hemp shiv on the surface of the specimen occurred quickly at the beginning of the test, emitting smoke. The higher was the plant aggregate content, the higher was the smoke release. After removing the sample from the heat source, its surface was black (Fig. 4), but after cooling it became slightly red, as fired bricks are. The cooling of bio-sourced samples occurred faster than earth alone samples.

Fig. 4: S6 sample after ignition time test

3.3 Thermal conductivity

Vegetal matter is decomposed by high temperatures and the microstructure of the material is modified. Thermal conductivity is impacted by such transformation. After a fire, it is thus interesting to know the evolution of the material properties. From 400°C and higher, the cohesion of S6 samples is declined, when the straw is burnt. All the other formulations were kept cohesive.

Thermal conductivity for each formulation is displayed in function of the temperature experienced from 25°C to 800°C in Fig. 5 and the evolution of the sample mass in function of the temperature is shown in Fig. 6.

Thermal conductivity is decreased when the experienced temperature is increased. For the first stage, it can be explained by the evaporation of the water adsorbed by the material which had not sufficient time to re-adsorb much water. Between 200°C and 400°C, the decrease in thermal conductivity seems to be due to the decomposition of the vegetal matter which occurs around 350°C, generating an increase in porosity and thus a lower density. However, this decrease is not much important in comparison with the earth alone sample. This can be explained by the kinetics of the degradation process, which might be longer than the time of the stage temperature. Between stages 400°C and 600°C, the sample mass is quite stable, however thermal conductivity is still decreasing. It should be due to the deshydroxylation of the clay which is the loss of the constituting water OH and the transformation in metakaolin. An important decrease in thermal conductivity takes place between 600°C and 800°C, which can be linked to the decarbonation of the material.

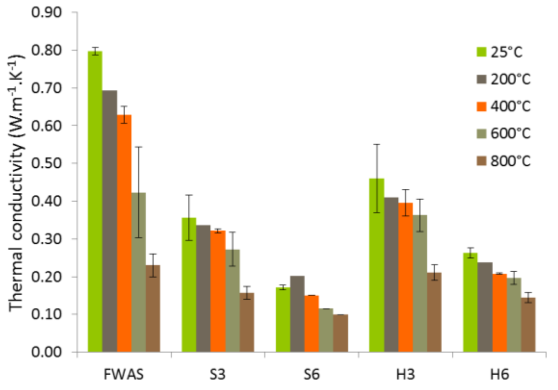

Fig. 5: Thermal conductivity according to the experienced temperature

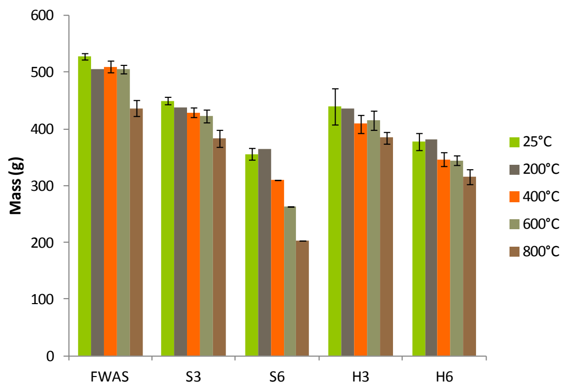

Fig. 6: Sample mass according to the temperature experienced

3.4 Compressive strength

After a fire, the resistance of a building should be maintained. It is thus important to verify if the physical-chemical changes of the material lead to a high decrease in strength.

The results are presented in Fig. 7. Between 25°C and 200°C, no real difference in strength is observed, standard deviations being quite important. From 400°C and higher, the effect of the temperature on compressive strength is different whether it is earth alone or earth with plant aggregates. For earth alone samples (FWAS), the compressive strength is increased at 400°C and 600°C (6.8 MPa and 14.6 MPa respectively) but is slightly decreased at 800°C (10.8 MPa). However, standard deviations are important. The increase of the resistance could be due to the transformation of kaolinite into metakaolinite between 460°C and 600°C, 550°C according to [Cultrone, 2001].

For samples containing vegetal matter, no significant difference is observed between the samples subjected to room temperature and samples which experienced a temperature of 200°C. Standard deviations are also quite important. At 400°C, the resistance decreased significantly due to the degradation of the vegetal matter around 350°C as shown by the PCFC results. This drop in strength increases with the amount of vegetal particles. Between 400°C and 800°C, the compressive strength progressively increases keeping however a strength value lower than the initial one. This increase may be linked to the firing of the earth, engendered by the loss of the constituting water and its transformation into metakaolin.

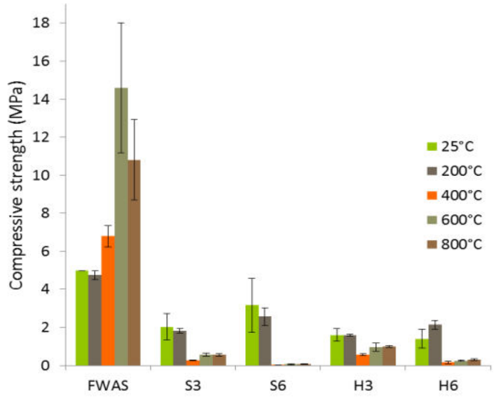

Fig. 7: Compressive strength according to the experienced temperature

3.5 Temperature of the unexposed face test

Fire resistance can be characterized with an insulation criteria such as the one determined thanks to the test presented hereafter. A material is considered to protect against fire when a fire-like increase of the temperature induces a temperature increase of the unexposed face lower than 180°C measured with either thermocouple.

Temperatures of each face of the samples in function of the time are presented in Fig. 8. The temperature programmed for the earth alone sample is slightly different from the others, the temperature step of 800°C lasting only 30 minutes instead of 2 hours for the other formulations.

It can be observed that the higher is the vegetal aggregate content, the better the materials seems to insulate from high temperatures. This result is in good agreement with the thermal conductivities previously obtained (Fig. 5). There is a delay and a decrease of the temperature rise with the addition of 6% of plant aggregates.

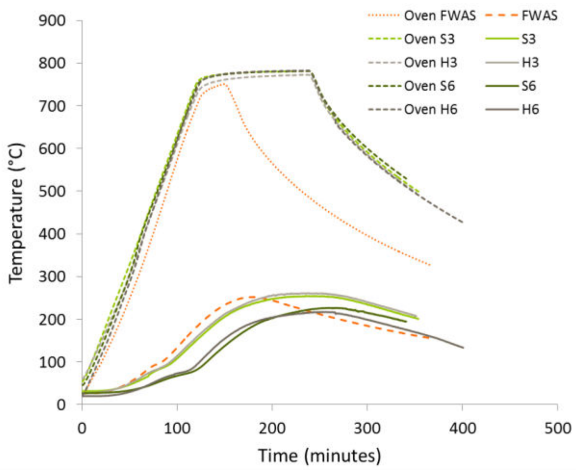

Fig. 8: Exposed and unexposed face temperatures in function of time

The temperatures of the unexposed face measured in the worst case scenario are shown in Tab. 3. The increase in temperature ΔT is calculated thanks to the initial temperature T_0 and the maximal temperature reached T_{max}. The time in minutes that a sample could maintain an increase in temperature lower than 180°C is also displayed (Time ΔT < 180°C).

For the different sample formulations, the rise in temperature is always higher than the required value of 180°C (between 197°C for H6 samples to 235°C for H3 samples). However, this required value is relative to a standard heating curve presented in the ISO 834-1 [ISO, 2014] which prescribe a temperature of 739°C at 15 minutes and 1049°C at 120 minutes. The heat curves of the current test are then less aggressive than the standardized ones. The conclusions about the increase in temperature drawn must then be taken carefully. The samples containing 6% of plant aggregates are the best thermal insulators, with temperature rises of 197°C and 200°C, which is quite close to the standard EN 1363-1 [CEN, 1999a]. The samples with plant aggregates maintained the unexposed face temperature lower than 180°C for longer time: higher than 3 hours for S6 and H6 samples against around 2h30 for S3 and H3 samples and 2h10 for a FWAS sample.

Tab. 3: Unexposed face temperatures

	T_0 (°C)	T_{max} (°C)	ΔT (°C)	Time (min) ΔT< 180°C
FWAS	26.4	252.3	225.9	132
S3	32.0	255	223.0	154
H3	25.8	261.2	235.4	145
S6	27.1	226.8	199.7	206
H6	20.3	217.3	197.0	191

However, this increase in temperature is influenced by the material thickness, which is around 5 cm in this case of study. Whether it is a material used as a partition wall or a load-bearing wall, the thickness would be higher, meaning that the temperature rise would be lower. Other tests are necessary to evaluate that rise in the case of a thicker material.

4 CONCLUSION AND OUTLOOK

The effect of straw and hemp shiv additions on the fire reaction and resistance of an earth matrix has been analyzed.

These results show the tendency of the bio-based material to not ignite itself. Although the PCFC test has shown a peak of heat release rate around 350°C for both plant aggregates, corresponding to the degradation of the cellulose, ignition-extinction test has proved that the composite material is still non-flammable. The smoldering is present only on the plant particles, making the material more porous and so a better thermal insulator. However, compressive strength of earth containing plant aggregates is decreased when the material is submitted to high temperatures.

Tests were realized during a short period, the experiments realized shall be repeated to reduce the spread of the results. It also should be interesting to extend the tests to a larger scale, at least to the brick size.

5 ACKNOWLEDGMENTS

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