Characterization of hemp fiber fire reaction

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RESEARCH ARTICLE

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Characterization of hemp fiber fire reaction

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Abstract
Integrating bio-resources in materials allows to reduce the environmental impact of the building industry. This study deals with fire-retardant treatments, alternative to boric acid and ammonium salts solutions, applied to hemp fibers for thermal insulation application. The aim is to limit the energy, sanitary and environmental impacts of the treatment, while optimizing the technical performances. A laboratory protocol evaluates the flame-retardant effect of the developed treatments. Treated fibers, including commercial treatments, are subjected to characterization tests: direct ignition small flame, thermal analysis (thermogravimetric analysis [TGA], differential thermal analysis [DTA]), pyrolysis-combustion flow calorimeter and cone calorimeter. Some of the methods have been adapted to be applied to fibrous materials. The obtained results orientate the formulation of a treatment and highlight the complementarity of the analysis methods. Coupling the results, the ConeTools's predictive model leads to the estimation of the reaction to fire class according to the Euroclass. Euroclass C appears accessible with a tailored treatment.

KEYWORDS
bio-sourced fibers, cone calorimeter, fire-retardant treatments, hemp fibers, thermal analysis

1 | INTRODUCTION

The use of bio-based materials such as plant fibers as part of the formulation of materials is highly exploited by various industry sectors. Indeed, in the case of building materials, the use of agricultural resources represents an attractive alternative for several reasons[1]:

- plants sequester CO_2 during their growth, providing a resource with a positive carbon balance;
- agri-resources present high intrinsic technical performances: thermal and acoustic insulation, water regulation, good mechanical properties;
- their valorization contributes to the development of territories and favors the development of activities in short circuits.

Hemp is a local (France) and annually renewable plant resource. The entire plant can be valorized and integrating hemp into the cultivation rotation has agricultural advantages.[2] Hemp fibers are already used to produce good thermal insulation, with high hygroscopic and acoustic properties.[3–5] However, these organic bio-based materials have a potentially unfavorable reaction to fire. On that topic, interesting work about hemp fiber flammability used as raw insulation is proposed by

Freivalde et al.,[6] used in fabrics is proposed by Misnon and Islam[7,8] and used as filler in polymer composites is proposed by Zhao et al.[9] The study of the flammability of cotton fabrics proposed by Zhang et al.[10] focuses on alkaline treatment, potentially usable for hemp fiber.

This article presents a part of the ADEME project "INNOFIB". The objective of this project is to develop an innovative industrial process of functionalization of hemp fiber using a dry process. The two main challenges of this project are:

- the refining of the hemp fiber to obtain improved thermal performances
- the treatment of the fibers to improve their fire and fungal behavior in case of exposure to high relative humidity.

The fire-retardant treatment solution must limit the health and environmental impacts,[11–13] while optimizing the technical performances of treated products. An alternative to boron and ammonium salts is sought.

To do this, a laboratory protocol is set up to evaluate the flame-retardant effect of various treatments. This protocol includes a step of fiber treatment by humidification, followed by a drying. The treated hemp fibers are then subjected to various characterization tests: direct ignition tests, small flame tests, thermal analysis (DTA-TGA), pyrolysis-combustion flow calorimeter and cone calorimeter. Some of the test methods had to be adapted for bulk fibers insulation.

Obtained results are used to identify correlations between measured parameters and to make easier the identification of an efficient treatment. An evaluation of the reaction to fire according to the Euroclass[14,15] is then discussed.

2 | MATERIALS AND METHODS

2.1 | Tested fibers, flame retardant treatments and application

Hemp fibers are produced by CAVAC Biomatériaux (St Gemme La Plaine – France) through a dry mechanical refining process. The hemp variety is *Futura 75*. The chemical composition is reported to be 55%–90% of cellulose, 10%–20% of hemicelluloses, 5% of lignin and 1%–3% of pectin, since this was in a previous study.[16] Some reference insulating materials are also integrated in the study: recycled cotton fibers, wood fibers, cellulose wadding and sheep's wool.

The different fibers are characterized by their fiber lengths and average diameters (Table 1, Figure 1). Hemp fibers have an average length of 10 mm and an average diameter of 101 μm. The hemp fibers used in this study have a bulk density of 50 kg/m^3.

Different fire-retardant treatments, mono or bi-compound, formulated from mineral and metallic compounds (including phosphate-based compounds, alkali metal hydroxide, metal hydroxide, various hydrated salts and hydrated silicates and others mineral salts) are tested. In total, 15 fire retardant treatments are evaluated for different percentages of treatment ranging between 5 and 20 wt%. 20 wt% is the maximum amount of FR treatment remaining economically realistic and also not degrading the density of the final product. Some fire treatments are mixed to formulate bi- compound treatment. In the case of bi-compound the compatibility of the components was checked to avoid any chemical precipitation. As example alkaline solution cannot be associated with phosphate-based treatment. Alkaline treatments based on alkali metal hydroxide are already used on naturals fibers[9,17] but in such case carbonation may occur and alter the durability of the treatment. Concerning details of the treatments, information are not available due to confidentiality reasons.

Commercial treatments used as a basis for comparison are also integrated into the study. Boric acid and ammonium phosphate are used as reference.

Flame retardant additives are placed in water solution or suspension and are applied on fibers by humidification. A method was created to treat the fibers at laboratory scale. One hundred gram of fibers are placed in a blender (conforming to EN 196[18]) equipped with a paddle. The treatment solution is added slowly while the paddle rotates (118 rpm). When all the solution is added,

TABLE 1 Fibers characterization

	Average df (μm)	df Standard error (μm)	Fiber length μm (%)				
			>150	150–100	100–50	50–20	<20
Recycled cotton	15	5	0.0	0	6.4	21.2	72.4
Sheep's wool	33	18	0.0	21.2	44.8	30.2	3.9
Wood fiber	31	10	0.0	0.0	0.0	0.0	100.0
Short technical hemp fiber	101	124	0.0	0.0	0.0	0.0	100.0

FIGURE 1 Left: hemp fibers and right: sheep's wool

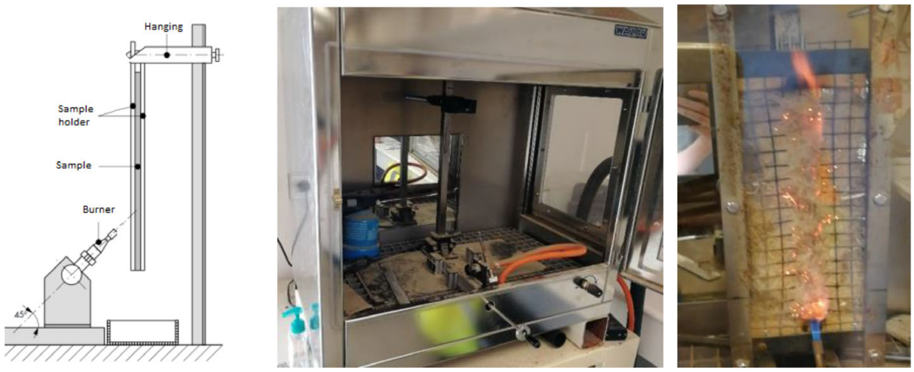

FIGURE 2 Left and middle: small flame tests installation and right: test in progress

the mixer is stopped to scrape the bottom of the bowl. The mixing is restarted for 2 min at 118 rpm. The fibers are then placed in an oven (70°C). This protocol was validated by analyzing the dispersion of a pigment solution applied on hemp fibers. This validation shows that the treatment is perfectly homogeneous when 100 g of solution are applied on 100 g of fibers, during 3 min of mixing. All the treatments are applied according to this method.

2.2 | Small flame tests

The standardized small flame test determines the flammability of the vertical sample by direct incidence of a flame (NF EN 11925-2,[19]) (Figure 2). For each treated fiber batch, three tests are conducted. They are systematically filmed, in order to better distinguish the conditions of ignition, propagation and extinction of the flame. After each exposure, the flame height is measured. The remanence of a smoldering fire is also observed.

2.3 | Thermal analysis

Thermal analysis is performed on a Pyris-Diamond DTA-TGA. Fibers are previously micronized to 0.5 mm with a laboratory knife mill (Retzsch ZM 200). Then, a 10–20 mg sample of micronized fibers are heated from 25 to 900°C, at a rate of 10°C/min, under nitrogen. The flow rate of nitrogen administered during the process is 100 ml/min. During the heating, the sample's mass and its heat exchange are monitored (Figure 3). The test leads to the evaluation of the pyrolysis temperature, the rate of char, the endothermic and exothermic reactions related to the sample chemical composition.

As an example, Figure 3 shows the DTA-TGA curves of several untreated and treated fibers.

2.4 | Pyrolysis-combustion flow calorimetry

Flammability of fibers is assessed using pyrolysis-combustion flow calorimetry according to method A (anaerobic pyrolysis). A small amount of fibers (around 3 mg) is heated up to 750°C under nitrogen flow at a rate of 1 k/s. Gases released are sent to a combustor at high temperature (900°C) in presence of an excess of oxygen. In these conditions, combustion is believed to be complete. Heat release rate is calculated using Huggetts' relation (oxygen depletion method)[20] and drawn versus pyrolysis temperature.

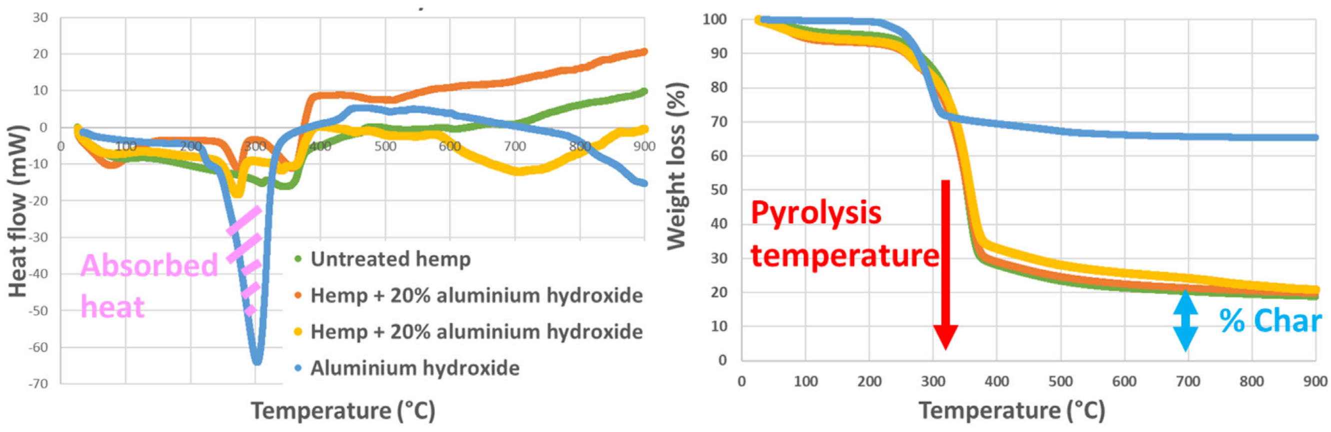

FIGURE 3 Graphs showing results of the DTA-TGA

2.5 | Cone calorimeter

The cone calorimeter test is used to quantify the flammability of a material. It is carried out on an FTT apparatus according to the ISO5660 standard.[21]

Preliminary tests were carried out on treated and untreated bulk hemp fibers, under different conditions in order to adapt this analysis method to fibrous material (sample implementation method, density of the sample, heat flux). A specific procedure was then defined.

The fibers are implemented by scattering in a sample holder of dimensions 100 mm × 100 mm × 77 mm. Density reached for the hemp fibers range between 38 and 80 kg/m^3. The sample is horizontally exposed to a heat flux of 35 kW/m^2 generated by the radiant cone. The distance between the upper surface of samples and the cone heater is 25 mm, as requested in the standard. There is no grid on the surface of the sample holder. The released flammable gases are ignited by a spark generated by an electric arc. The air flow rate is fixed at 24 L/s. The heat flow rate versus time is measured by the oxygen depletion method. According to Huggett's principle[20]: 1 kg of oxygen consumed by the combustion corresponds to an energy release of 13.1 MJ. Additional tests were also performed at other irradiances (25, 50, and 75 kW/m^2) and/or with varying amounts of fiber or sample thickness (i.e., thickness of fibber's bed). The test leads to the evaluation of the time to ignition (TTI), the peak heat rate (pHRR) and total energy released (THR) (Figure 4 right). The ratio of THR and loss mass corresponds to the effective heat of combustion (EHC). The measurement error is 1 kJ/g on the THR, about 20 W/g on the pHRR for low values as obtained with hemp fibers.

It is observed from cone calorimeter data that smoke release is negligible or very small. The reason is that the heat release rate is still limited. The smoke production depends not only on the nature of the material but also on its burning kinetics.[22] Most often the pHRR of our fibers is lower than 150 kW/m^2 and the period for which the HRR is higher than 60 kW/m^2 is very short.

2.6 | Euroclass classification by ConeTools software

The Euroclass according to the Single Burning Item (SBI) test[14] was evaluated using the ConeTools software.[23–25] It predicts the ranking from a cone calorimeter test via a phenomenological model. The classification is based on:

- the THR600, the energy released after 600 s, in MJ;
- the FIGRA0.4, the fire growth rate, in W/s, the FIGRA value is considered when the THR exceeds 0.4 MJ.

The classes range from A1 (non-combustible materials) to F (materials with the highest hazard in case of fire). ConeTools allows to discriminate the following classes A2/B, C, D and E/F.

3 | RESULTS AND ANALYSIS

The obtained results highlight the complementarity of the used analytical methods.

3.1 | Obtained results

The small flame test on the untreated fiber sample shows a quite instantaneous ignition and a flame propagation until the top of the sample. A smoldering effect is observed. The tested treatments do not notably increase the time to ignition (few seconds) but reduce the flame propagation, and in some case, the smoldering effect disappears.

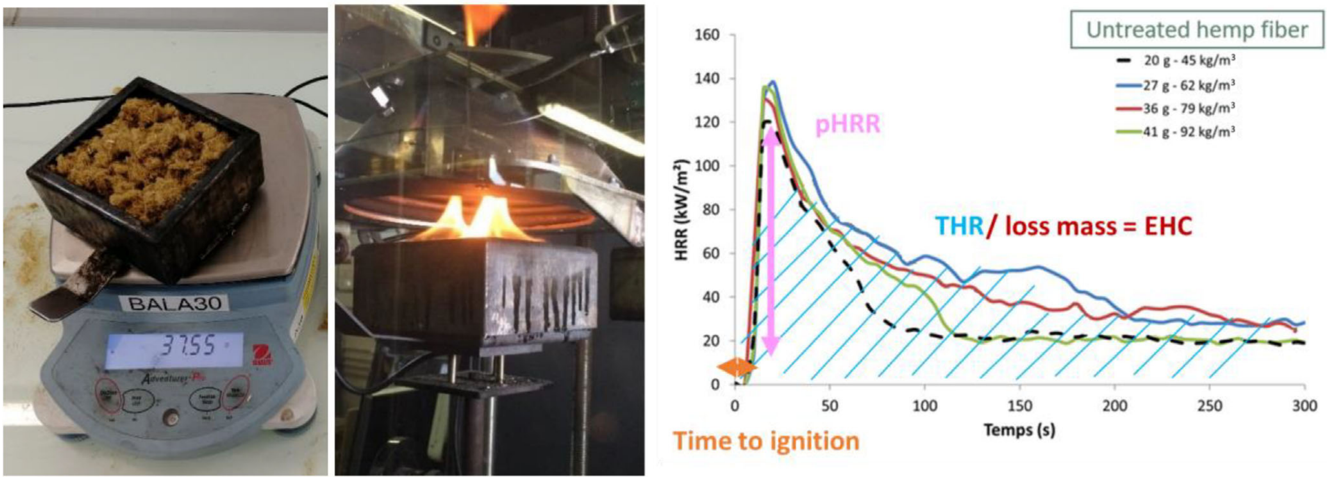

FIGURE 4 Left: sample before combustion; middle: test in progress; right: graphs showing results of the cone calorimeter

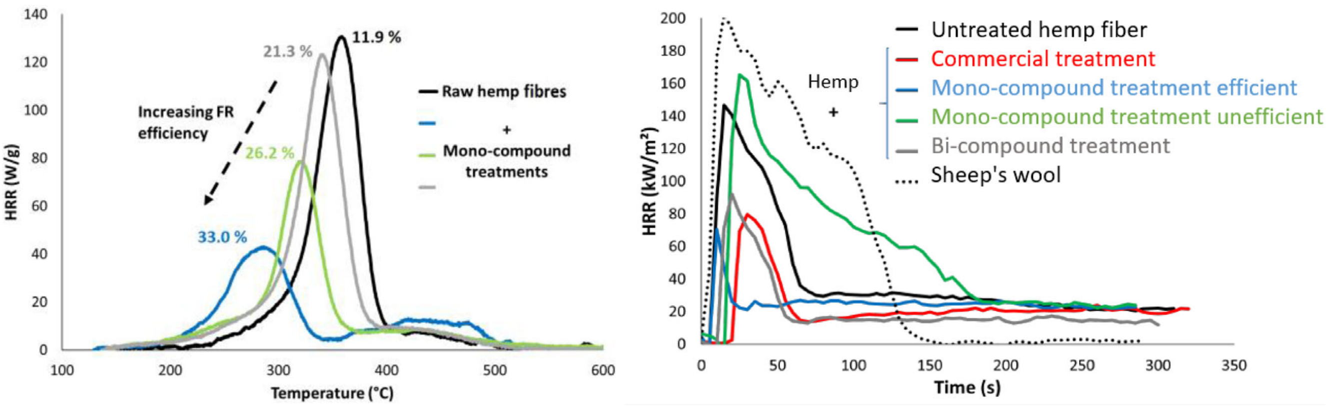

FIGURE 5 Left: Heat rate curves and char content (%) for some hemp fibers in PCFC; right: comparison of heat release rate curves obtained with cone calorimetry (35 kW/m²) for a selection of samples

During the thermal analysis, for all analyzed samples, the main degradation occurs between 100 and 400°C (depending of the treatment) and is followed by a decreasing plateau (Figure 3 right). These results correspond to those found in the literature.[26] Indeed, thermogravimetric analyses show that cellulose decomposes between 315 and 400°C with a maximum degradation rate at 355°C. The decomposition of hemicelluloses takes place between 220 and 315°C with a maximum degradation rate at 270°C. Lignin are the compounds that start to degrade gradually over a wide temperature range from 100 to 900°C, making them the most stable compounds in the fiber.

The pyrolysis temperature is affected by the composition of the treatment. The char is about 18% for untreated hemp fiber, and can reach 30% with some treatments. Complementary the DTG reveals the endothermic or exothermic reactions occurring at different temperature between 150 and 600°C.

Pyrolysis-combustion flow calorimetry (PCFC) data confirm that hemp fibers decompose in one apparent peak (because the decomposition steps of the various components overlap) centred at 360°C (Figure 5). Its intensity is approximately 130 W/g and total heat release is in the range 7–8 kJ/g. All these values fit well with the literature,[27] The values in this reference are slightly lower than in the present study. Figure 5 also shows some curves for fibers flame retarded with different treatments, based on mineral salts. The peak temperature and peak intensity appear correlated, for all types of treatment. When the peak intensity is lower, the measured peak temperature is lower. Moreover, the pHRR decreases when the char content increases, showing that FR promotes strongly the charring (reducing in turn the total heat release). It is noteworthy that these tendencies are also well correlated to cone calorimeter data: low THR and pHRR in PCFC also correspond to low HRR in cone calorimeter. pHRR appears as a direct indicator of the treatment efficiency.

The heat of complete combustion Δh calculated for the fibers tested in PCFC (complete combustion is ensured in PCFC) was compared to their effective heat of combustion in cone calorimeter. The combustion efficiency, calculated as the ratio between EHC and Δh is in all cases around 1 ± 0.2. This result confirms that the heat of complete combustion measured in PCFC can be used instead of the effective heat of combustion in our model discussed below.

In cone calorimeter, ignition is followed by the peak heat rate (pHRR) which varies, according to the types of fibers or FR treatment (Figure 4 right, Figure 5), between 40 and 200 kW/m^2 (for an incident heat flux of 35 kW/m^2). The heat release rate then decreases rapidly, and stabilizes at a value of about 15–30 kW/m^2, which is maintained even after extinction (due to thermo-oxidation of the fibers char). Previous calibration tests have shown that the pHRR varies little with bulk density in the studied range (< 100 kg/m^3) (see Figure 4 right). This small influence of fiber's bulk density makes easier the comparison of the results.

For the hemp fiber, samples treated with effective and ineffective treatments are compared Figure 5. The commercial treatment (ammonium phosphate) leads to an expected pHRR decrease and to a sensible delay of the time to ignition. Such delay is not observed for the other tested treatments. Effects on pHRR are observed. Quite same pHRR are obtained with some efficient mono compound treatments, the reference commercial treatment and a bi-compound treatment. The potential synergy of the components of bi- compound treatments has never been quoted with the selected formulations. As a comparison of bio-sourced fibers, the curve obtained for sheepwool is characterized by higher pHRR without plateau.

The observed thin thermal behavior may appear surprising with such thick samples (>7 cm). This means that bulk fibers are good thermal insulator. There is no significant heat transfer: only the upper fraction of the fiber receives the incident radiative flux and pyrolyzes. Beyond this upper fraction, pyrolysis does not take place or is progressively induced, the heat diffuses slowly.

3.2 | Complementarity with small flame, TGA and cone calorimeter test

The search for an inter-analysis relationship also leads to the identification of a link between (Figure 6):

- the energy released at the cone calorimeter and the pyrolysis temperature (identified with TGA): the higher the pyrolysis temperature, the higher the total energies released (THR) during combustion; the reason is that many FR treatments (especially phosphorus-based ones) lead to a decrease in energy released while reducing the thermal stability of fibers;
- the energy released at the cone calorimeter and the flame height (at the small flame test): the more energy released during combustion, the more the flame spreads over the sample.

Some samples do not adhere to these correlations, evidencing that the modes-of-action of the corresponding FR are different. They are not systematically the same samples for both graphs.

It has been shown[28] that the pHRR of thermally thin and geometrically thin materials (fabrics) can be predicted from:

- the initial sample mass
- the incident radiant flux
- the effective combustion energy.

Such model assumes that the entire sample mass is heated homogeneously and thus contributes to the pHRR.

For this study, this model was adapted to consider the fact that only a fraction of the fibers is pyrolyzed at the time of pHRR, in the case of fiber beds (Figure 7). The adapted model (model in the process of being published) predicts the pHRR with acceptable accuracy: the estimated average error is 17%. Moreover, this model performed well in the presence of other classes of insulating materials (i.e., thermally thin but geometrically thick) such as foams, agro-concretes, particle boards or woods. The relevance of the model shows that the pHRR of bulk fibers depends mainly on their heat of combustion. In fact, there is an acceptable correlation between the heat of combustion and the pHRR. For example, sheep's wool has both the highest pHRR (about 200 kW/m^2) and the highest combustion energy (about 16 kJ/g).

The effective heat of combustion (EHC) of fire-retarded hemp fibers varies between 3.5 and 9.5 kJ/g, depending on the treatment. One way to reduce the effective heat of combustion is to form char. Indeed, lignocellulosic fibers have an EHC close to 10 kJ/g, which corresponds to the heat of combustion of the released gases. The char that is, the polyaromatic residue formed during pyrolysis, has a structure rich in C and H (its formula can be roughly simplified to C_5H_2). Its "potential" heat of combustion is higher than 30 kJ/g. By increasing the char rate, a flame retardant will therefore deplete the gas phase from C and H elements, and thus reduce the EHC of the gas phase. This strategy has been applied in the case of hemp fibers. Treatments promoting char are limiting the EHC and should also lead, according to the correlations observed in Figure 6, to the reduction of the thermal stability (i.e., pyrolysis temperature).

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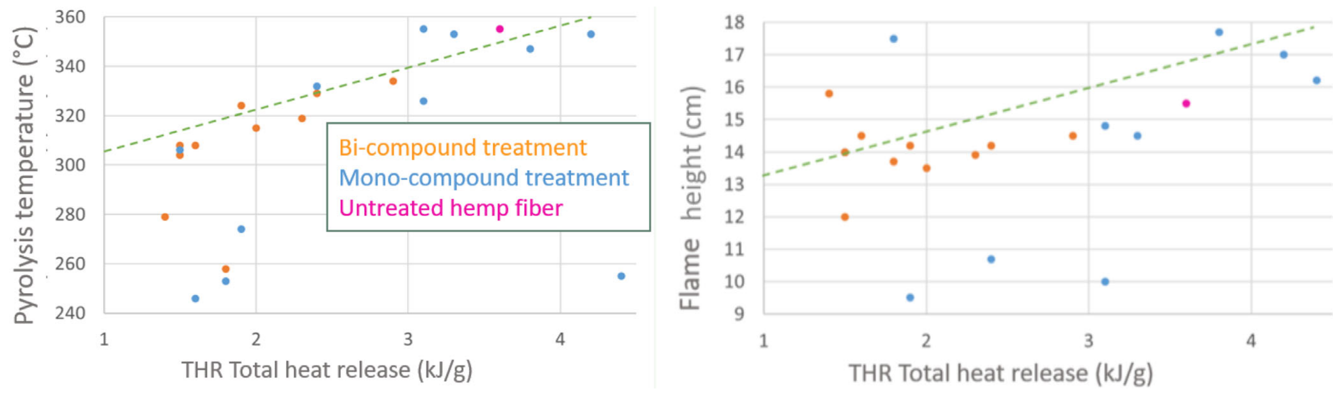

FIGURE 6 Graphs showing the correlations between TGA, small flame and cone calorimeter

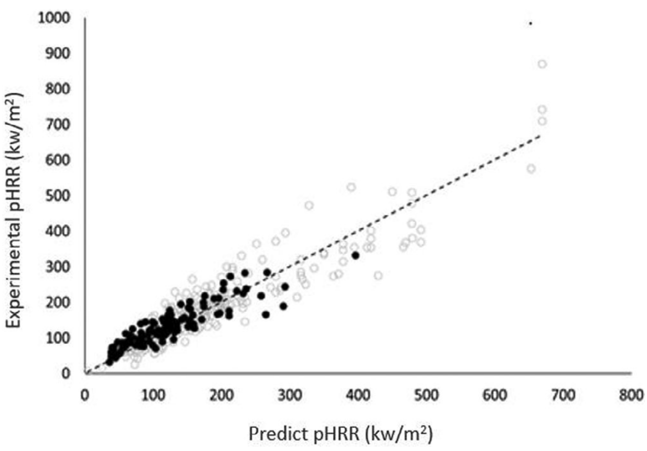

FIGURE 7 Comparison of predicted and experimental peak heat rate, black marks correspond to the present study and white marks to other thermally thin tested samples (polymer fabrics, polymer fibers)

3.3 | SBI prediction of formulations

SBI predictions using Conetools software show that untreated fibers (hemp but also other reference fibers) are rated E or F according to the Euroclass, mainly due to a high FIGRA value (>750 W/s) (Figure 8). FIGRA is indeed the most severe criterion. According to the second considered criterion (the energy released after 600 s, called THR600), all fibers are classified C or better.

Treated fibers have performances ranging from E/F to C. The C rating is obtained for fibers with a pHRR, lower than 70–80 kW/m^2 in cone calorimeter. However, some fibers with a pHRR of this order but a very low ignition time (<5 s) can be classified D or E/F.

It should be noted that these tests, unlike the SBI test, were conducted without a holding grid in front of the fibers. These predictive classifications are therefore more penalizing than when tested with a grid.

4 | CONCLUSION

The high flammability of insulating materials composed of bulk hemp fibers requires a fire treatment to prevent the fire propagation, in the event of a fire. Due to their low thermal conductivity and low density (close to 50 kg/m^3), their behavior is thermally thin. Only the upper fraction of the fiber receives the incident radiative flux and pyrolyzes. This is why a very short ignition time and thus a high propensity to spread fire is observed.

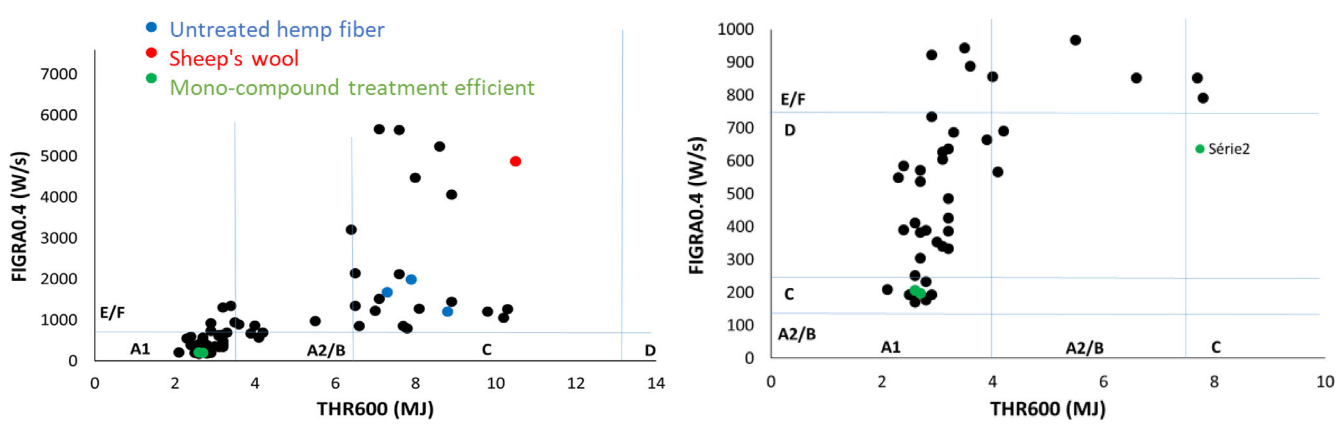

FIGURE 8 FIGRA0.4 and THR600 prediction from ConeTools (black points correspond to all other FR treatments)

Analyzing the cone calorimeter test results, it appears that treatments promoting the char seems to be the most effective but do not notably increase the time to ignition. Interestingly, the results are quite not influenced by a change in the sample density.

Coupling TGA and cone calorimeter results reveals that the higher the pyrolysis temperature, the higher the total energies released (THR) during combustion probably because the FR treatments promote char and reduce thermal stability in the same time. In parallel, the energy released at the cone calorimeter and the flame height (at the small flame test) are correlated. Consequently, selecting a treatment characterized by a low pyrolysis temperature is appropriate.

The coupling of the results also allows to propose a better understanding of the fire behavior. Peak of HRR in cone calorimeter can be predicted assuming that the effective heat of combustion is close to the heat of complete combustion usually measured by pyrolysis-combustion flow calorimetry. The estimated pHRR value (corresponding to the combustion of the top thin layer) is very close to the experimental value. HRR data from cone calorimeter have also been used to operate the ConeTools software. Then, the ConeTools's predictive model is of full interest to the estimation of the reaction to fire class according to the Euroclass.

The understanding of the changes in the heat transfer and thermal decomposition modes due to treatments has allowed the identification of efficient alternative formulations. The selected fireproof treatments allow to limit the heat of combustion, which conditions the SBI classification, by reducing the pHRR in cone calorimeter below 50 kW/m^2 (for a radiative flux of 35 kW/m^2). This would correspond to a Euroclass C, according to the ConeTools software, during a penalizing test without holding grid.

Reaching the Euroclass C appears as a key for insulation materials based on hemp fibers to the building market acceptance.

AUTHOR CONTRIBUTIONS

The main experiments were conducted by Lily Deborde and Loic Dumazert. This article–original draft was written by Lily Deborde. Christophe Lanos, Rodolphe Sonnier contributed to writing–reviewing, editing the manuscript and to the data analysis. Valentin Colson gave the helpful suggestions in revision of our manuscript.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Christophe Lanos is Professor at University of Rennes. Her research topics focus on building materials and biobased materials.

Valentin Colson, PhD, is the R&D manager of CAVAC Biomatériaux, a company specialised in the transformation of hemp and the manufacture of insulation product made from natural fibres.

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