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Ignition of biobased concretes

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ABSTRACT

Nowadays, the construction field is responsible for a quarter of French emissions of greenhouse gases. In order to minimize this impact, one solution is to promote the use of local and biobased materials with a low carbon impact. Biobased resources can be mixed with a binder to produce light ($150 - 500 \text{ kg.m}^{-3}$), medium ($500 - 1200 \text{ kg.m}^{-3}$), or high density ($1200 - 1800 \text{ kg.m}^{-3}$) biobased concretes. The fire behaviour of these materials is still poorly documented. In this work, combustion microcalorimetry tests were conducted to measure the amount of energy released during the combustion of various bioresources while bomb calorimeter tests allowed quantifying their gross heat of combustion. Cone calorimeter experiments were performed to study the ignition of biobased concretes. Results provide general knowledge and data regarding the fire behaviour of bioconcretes. It was observed that only the lightest ones ignite. It appears that ignition relies on two parameters: the combustion energy density of bioconcrete and the energy required to heat the material to the ignition temperature. One criterion is proposed to predict ignition. It accounts for different endothermic processes, such as the bioresource pyrolysis, the possible decomposition of the binder and the heating phenomenon itself up to the pyrolysis temperature.

Keywords:

Fire behaviour
Biobased concretes
Flammability
Biobased materials
Combustion
Ignition

1. Introduction

In France, the building sector represents 23 % of greenhouse gas emissions and 43 % of the national energy consumption [1]. In 2022, the French government moved from a thermal to an environmental regulation (RE2020), which is more ambitious and demanding for the construction sector, regarding the carbon impact. Consequently, this field requires further efforts to become more sustainable, not only in France, but also in the whole world. Indeed, the impact of construction occurs on a global scale. According to Architecture 2030, an organization established in 2002 in response to the ongoing climate emergency, the construction field generates 42 % of annual global CO₂ emissions [2].

In order to minimize this impact, local biobased materials with low carbon footprint are being considered for construction, as crop straw, hemp shives or rice husks. These biobased resources, as well as others,

can be mixed with a mineral binder to produce biobased concretes, which are used as insulating materials with low environmental impact. Replacing conventional insulating materials by biobased ones allows limiting the current global warming by temporarily sequestering carbon during several decades. The density of plant particles once implemented in lightweight bioconcretes varies between 50 and 200 kg.m^{-3} typically depending on the nature of the particle, its particle size, its compressibility, and the manufacturing technique. As a result, 1 m^3 of biobased concrete stores between 22 kg ($44 \% \times 50 \text{ kg.m}^{-3}$) and 100 kg ($50 \% \times 200 \text{ kg.m}^{-3}$) of carbon (considering that the carbon content in lignocellulosic materials ranged from 44 % to 50 %) [3].

As studied by Amziane and Arnaud, concretes containing plant aggregates have hygrothermal, thermal and acoustic properties that greatly improve the comfort of houses [4]. The porous nature of these concretes is responsible for a very significant acoustic absorption [5].

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Furthermore, their plant aggregates can absorb a considerable amount of moisture, which helps regulate humidity. Finally, regarding thermal behaviour, they are able to reduce heating and cooling needs of buildings to their low thermal conductivity and due to moisture fluxes [6].

The plant aggregates of these biobased concretes come from the stem of plants grown either for their fibres (hemp, flax, etc.) or for their seeds [7]. Due to the structure of the plant stem, such aggregates are usually malleable, elongated and very porous with a low bulk density. The woody core of the stem of the hemp plant (i.e. the hemp shiv) is the most used to produce bioconcretes and the most studied in the literature [8].

Different binders can be used to produce biobased concretes and the choice is essentially made according to the main properties desired. Indeed, the properties of the biobased concrete depend on the material used, the binder to biobased aggregates ratio and the manufacturing process. From a fire performance point of view, gypsum is believed to be the most protective binder when exposed to fire. Indeed, gypsum endothermic decomposition at low temperature (≈ 150 °C) consumes around 560 J.g^{-1} , thus limiting the heating rate of the material [9].

However, there is a lack of knowledge about how these bioconcretes ignite and contribute to heat release in case of a fire [10]. Sonnier et al. studied the fire behaviour of hemp, clay and gypsum-based concretes using a cone calorimeter for a wide range of densities ($180\text{--}1500 \text{ kg.m}^{-3}$) and reported that flammability is mainly controlled by density, whose threshold for ignition occurrence was around 500 kg.m^{-3} . It means that the flammability was only observed for low-density concretes [10]. In addition, Lanos has listed the fire reaction (Euroclass rating) of several biobased materials used in the building industry, including hemp concretes and renders [11]. Menezes et al. investigated the fire performance of wood bio-concretes and observed that the incorporation of an inorganic binder involving wood bio-aggregates inhibits ignition and the variation of volumetric fraction of wood shavings (40 %, 45 % and 50 %) in the bioconcrete mixtures did not significantly change the contribution of the heat release rate (HRR) for these concretes with a density between 1090 and 1353 kg.m^{-3} [12]. Bumanis et al. evaluated the fire behavior of gypsum and phosphogypsum concretes containing hemp shives (densities range from 200 to 400 kg.m^{-3}) and concluded that the role of gypsum content had a significant effect on the fire reaction. Time to ignition (TTI) was increased and the peak of heat release rate (pHRR) was reduced with the increase of gypsum content [13]. Nonetheless, each study focused on one specific material or a limited range of materials.

Nowadays, the Euroclass system classifies the fire reaction of building materials. In Euroclass system, the main ranking is related to the heat release, A being the best classification and F the worst. In addition, two other parameters are considered: the quantity of smoke generated by the product during a fire, which may be s1 (little or no smoke), s2 (visible smoke) or s3 (heavy smoke), and the presence of flaming droplets and particles during the first ten minutes of the fire (d) which can be d0 (none), d1 (a few) or d2 (a lot) [14]. According to our previous paper [10], biobased concretes do not release significant amount of smoke or flaming particles. In order to be classified according to its fire reaction, each category has its own tests and criteria. Non-combustible and limited combustible materials (A) do not contribute significantly to a fire, so, from a fire reaction point of view, they are the best choice for buildings. They can be further classified as A1 or A2, according to their gross heat of combustion (GHC) measured via a calorimetric bomb (EN ISO 1716). When this value is lower than 2 MJ.kg^{-1} , the material is rated A1. When the value is lower than 3 MJ.kg^{-1} , the material can be A2, but it must also be assessed according to the EN 13823 standard, also called single burning item (SBI) test [15]. The experiment is based on a fire scenario of a single burning item located in a corner between walls covered with the lining material to be assessed [16]. This test is mandatory for ranking materials from A2 to D, based on their heat release and fire growth rate index (FIGRA). SBI ranking was successfully predicted for building materials (with a success rate close to 90 %) from cone calorimeter results using phenomenological modelling [17–19]. A

commercial software (called ConeTools) allows performing such simulations.

As construction materials, the fire hazard must be considered as a crucial issue due to the inherent flammability of these biobased materials [20]. To the knowledge of the authors, no research work has been published on general indicators to predict the flammability of biobased concretes, in other words, on the study of the parameters that can allow understanding how these materials would behave in case of a fire. The aim of this article is to identify the relevant parameters to properly predict the ignition of biobased concretes, for a huge variety of different formulations.

2. Materials

The binders used in this study were earth, gypsum, and lime. Earth is a fine natural kaolin (trademark: Blankalite 78 from Soka, France) with 99 % of particles of size lower than $20 \mu\text{m}$. Gypsum was a 0/200 μm natural hemihydrate gypsum plaster (trademark: neige 1 R from Plâtre Vieuxot/Platre.com, France). The lime used was white natural hydrated lime, NHL 3.5 (trademark: Lafarge, France), used to constitute the binder/hemp couple validated by Construire en Chanvre.

Plants aggregates used were hemp shiv, rice husk, wheat straw and sunflower pith. Note that rice straw was added to the rice husk to produce some formulations because rice husk, alone, does not allow low densities to be achieved. Rice straw makes it possible to reduce the density while improving mechanical behaviour. Hemp shiv comes from Poitou Chanvre production. Average shiv length and width are 7.6 and 2.0 mm respectively. Rice husk comes from Balle Concept, produced in the region of Camargue in France. Rice straw was supplied by the Association Bâtir en Balles and also comes from the same region. The Réseau Français de la Construction Paille (RFCP) provided the wheat straw, which came from straw bales cut manually. The sunflower pith, which was separated from the bark, was issued from the SAVASCO project (savasco-poctefa.eu). Fig. 1 shows the different bioresources used for the biobased concrete production.(Fig. 2)

3. Methods

Samples were prepared as described below:

- Bioresources and binders were weighted separately with a $\pm 0.1 \text{ g}$ accuracy balance (which allows for a better than 0.5 % accuracy considering the weighted mass).
- At first, binders were mixed with water in a concrete mixer, and then the aggregates were incorporated.
- The water to binder ratio was adjusted according to the water absorption of plant particles, the binder water demand, and the recipe. It varied from 1 to 1.8 for earth-based samples, from 0.8 to 1.5 for gypsum-based samples and from 1.1 to 1.6 for lime-based samples.
- A first $10 \times 10 \times 10 \text{ cm}^3$ mould was completely filled with the mixture by applying a moderate manual compaction. The mass introduced in this first mould was weighted and noted.
- The other moulds were filled with the same mix and with the same mass in order to obtain repeatable specimen.
- 24 h after manufacturing, moulds were removed so that the samples could dry.
- Finally, when the samples were stabilized in mass in room conditions, they were repacked in cardboard moulds and sent to the laboratory.

Note that some concretes were prepared by mixing two binders (for example gypsum and earth) or two plant aggregates (for example rice husk and rice straw). In a few formulations (especially the densest ones), sand was also added.

For our first study regarding the fire behaviour of light biobased concretes made of hemp clay and/or gypsum [10], the bioconcretes



Rice husk



Wheat straw



Rice straw



Hemp shiv



Sunflower pith

Fig. 1. Bioresources used in the biobased concretes.

were stored in the cone calorimeter room up to mass stabilization where the atmosphere of the room was not controlled given the unforeseen events generated by Covid 19. However, for this work, after preparation, the new samples were stored in a room with a controlled atmosphere at 23 °C and 50 %rh up to constant mass prior to testing.

Regarding their nomenclature, it was decided to name the bioconcretes in the same way as it was done in our previous work [10]. They are called XYZ:

- **X** referring to the nature of the binder (E for earth, G for gypsum, L for lime and GE for a gypsum-earth mix);
- **Y** referring to the nature of the bioresource (H for hemp, R for rice husk, W for wheat straw, S for sunflower pith and RR for a rice husk-rice straw mix);
- **Z** referring to the apparent density of the bioconcrete stabilized at 23 °C and 50 %rh.

Note that, for four formulations, an earth microfilm was applied to the samples, since this is a common practice for mason. Therefore, they were referenced by the suffix “MF”.

For example, EW296 is a bioconcrete made of earth, wheat straw with a density of 296 kg.m⁻³. When the concrete contains a mix of binders, X mentions two letters and the respective weight ratio of binder. For example, G25E75H179 refers to a concrete containing a binder based on 25 wt% of gypsum and 75 wt% of earth and presenting a density of 179 kg.m⁻³. Note that when two bioresources were mixed (rice husk and rice straw), the proportion was always 80 wt% rice husk and 20 wt% rice straw.

Samples density was calculated considering a volume of 10 × 10 × 10 cm³. However, for some samples, the heaviest ones made of earth or lime, shrinkage occurred during curing and led to a slight reduction in volume. This reduction was estimated around 10 %. Nevertheless, the real sample size was not measured for all samples prior to testing.



Fig. 2. Manufacturing of biobased concretes (weighing, mixing in the concrete mixer, after mixing and moulding steps).

Therefore, all the densities reported in this paper must be considered as minimum values. Density was calculated after mass stabilization in the conditions already indicated above.

The flammability of the biobased resources studied was evaluated at microscale through pyrolysis combustion flow calorimetry (PCFC) (Fire testing technology, FAA Micro Calorimeter, East Grinstead, UK) as specified by the method A (anaerobic pyrolysis) and method B (aerobic pyrolysis) of the ASTM D7309 standard. Samples between 2 and 3 mg were heated at $1 \text{ K}\cdot\text{s}^{-1}$ up to $750 \text{ }^\circ\text{C}$ under nitrogen or air. Gases released were sent to a combustor at $900 \text{ }^\circ\text{C}$ in an excess of oxygen to guarantee a complete combustion. In this test, heat release rate (HRR) is calculated using the oxygen depletion method. Indeed, 1 kg of consumed oxygen corresponds to 13.1 MJ of energy released, according to empirical Huggett's relation [21]. For each sample, two tests were performed. Typical standard deviations in PCFC are $1 \text{ kJ}\cdot\text{g}^{-1}$ for the total heat release (THR) and less than 10 % for the peak of heat release rate (pHRR).

Tests in a Parr 6200 oxygen bomb calorimeter, according to the EN ISO 1716 standard, were performed in order to measure the gross heat of combustion (GHC) of the bioresources after being kept in a controlled atmosphere at $23 \text{ }^\circ\text{C}$ and 50 %rh. For this, 0.5 g of each bioresource was burned at constant volume, in an atmosphere of oxygen under pressure (30 bar), inside a calorimetric bomb. The heat of combustion determined under these conditions was calculated from the observed temperature rise, considering the heat loss and the latent heat of vaporization of the water. For each sample, three tests were performed, and the standard deviations were all less than $0.10 \text{ MJ}\cdot\text{kg}^{-1}$ for the GHC.

To perform piloted ignition, cone calorimeter (Fire testing technology, East Grinstead, UK) tests were conducted at $50 \text{ kW}\cdot\text{m}^{-2}$ during 20 min, according to the ISO 5660 standard [22]. The nominal exhaust flow rate was $24 \text{ L}\cdot\text{s}^{-1}$. The procedure consisted of exposing the $10 \times 10 \times 10 \text{ cm}^3$ samples directly on the sample holder without rockwool or

aluminium foil at the backside. A frame was used to ensure that the heat flux was only absorbed by the surface sample and not by the side faces, as it was done in our previous study [10]. The distance between the radiant cone and the upper surface was 25 mm. A spark igniter was used to promote piloted ignition. Even though several specimens have the same initial composition, their density can vary slightly. For this reason, each tested specimen was considered as one datapoint. Nevertheless, standard deviations were assessed on a specific formulation in our previous paper (EH261 i.e. hemp-earth with a density of $261 \text{ kg}\cdot\text{m}^{-3}$) [10]. TTI and pHRR were found to be $17 \pm 6 \text{ s}$ and $91 \pm 7 \text{ kW}\cdot\text{m}^{-2}$, respectively.

Finally, the specific heat capacity of biobased constituents, i.e. binders and bioresources, were measured using a C80 Calvet calorimeter (Setaram Instrumentation, Caluire-et-Cuire, France). This device can reach $300 \text{ }^\circ\text{C}$ and the temperature range chosen depends on the thermal stability of the materials tested. Each measurement consisted of 3 steps: firstly, an isotherm stabilization was performed, during 2 h at $28 \text{ }^\circ\text{C}$, then the sample was heated at $1 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ from $28 \text{ }^\circ\text{C}$ to $300 \text{ }^\circ\text{C}$ for the binders and at $0.2 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$ from $28 \text{ }^\circ\text{C}$ to $100 \text{ }^\circ\text{C}$ for the bioresources. Finally, an uncontrolled cool down step was applied to allow the device returning to room temperature. In addition to the specific heat capacity, the decomposition enthalpy of the binders was also measured for the given temperature range achievable with this equipment.

4. Results

Firstly, the bioresources and their capacity to release energy when burned was studied using PCFC and bomb calorimeter. After that, different formulations of biobased concretes were tested in the cone calorimeter in order to investigate the fire behaviour of these materials. Finally, a model to predict the ignition of the bioconcretes was proposed.

4.1. Bioresources flammability at microscale

The flammability of bioresources was analysed using PCFC (Figs. 3 and 4, Tables 1 and 2). Pyrolysis was performed under anaerobic and aerobic conditions. Anaerobic conditions are generally preferred to simulate flaming combustion in which oxygen is consumed in the flame and does not disturb the decomposition of the condensed phase [23]. Conversely, aerobic pyrolysis of the solid phase occurs when the flame vanishes (and also before ignition) [10]. Moreover, aerobic pyrolysis in PCFC is close to the GHC as measured in a bomb calorimeter since residues are only ashes.

Fig. 3 shows that, in anaerobic conditions, the HRR curves present a main peak for each bioresource, even though a secondary small peak may appear in some cases (e.g. sunflower pith). Significant differences can be found about the energies released during combustion by the plant aggregates. The energy released by the hemp shiv is the highest among

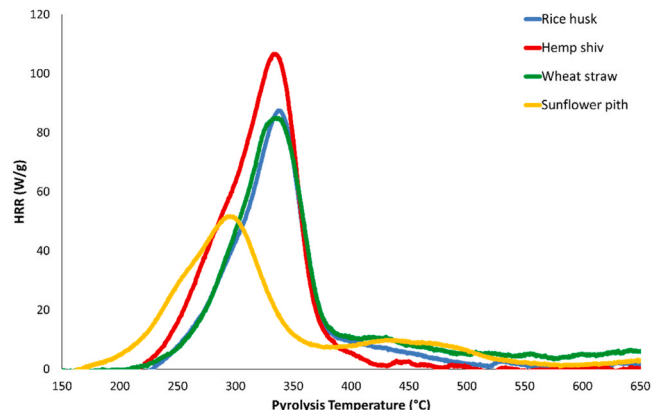


Fig. 3. HRR curves of biobased concretes during anaerobic pyrolysis in PCFC.

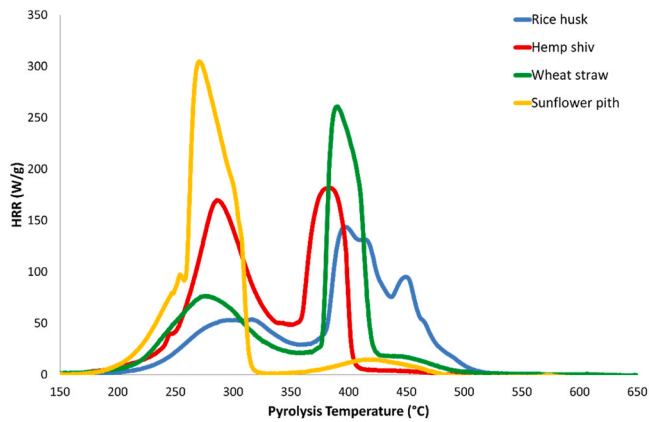


Fig. 4. HRR curves of biobased concretes during aerobic pyrolysis in PCFC.

all the materials evaluated with a THR of 10.4 kJ.g^{-1} , a pHRR of 115 W.g^{-1} at $338 \text{ }^\circ\text{C}$ (i.e. TpHRR) while it leaves the lowest residue content (19 %). The sunflower pith has the lowest value among the bioresources of this study, with a pHRR around 51 W.g^{-1} at low temperature ($293 \text{ }^\circ\text{C}$), a low THR (5.2 kJ.g^{-1}) and a fraction content residue close to 30 %. Arufé et al. [24] showed that the chemical composition of the sunflower pith is considerably different from the other bioresources analysed in this article (rice husk, hemp shiv and wheat straw), which can explain its low heat release when compared to the others. Rice husk and wheat straw exhibit similar properties which are intermediate between hemp shiv and sunflower pith. These properties allow the characterization of the variability of these bioresources, which directly influence the properties of the biobased concretes.

Under aerobic conditions, the bioresource is completely decomposed, consequently, the residue fraction is only constituted by ashes. The ash content is negligible for hemp shiv, rice husk and wheat straw but close to 10 % for sunflower pith, as seen in Table 2. The decomposition pathway is more complex and generally occurs in several steps (cf. Fig. 4). Two main peaks are usually observed. For sunflower pith, only one main peak is observed with a very small secondary peak at higher temperature. For rice husk, decomposition involves more than two pHRR. The first peak results from the decomposition of the main components which occurs earlier in the presence of oxygen and the second one is due to the thermo-oxidation of the char. While no char is formed to store a huge amount of carbon, HRR, THR and heat of complete combustion are much higher under these conditions when compared to anaerobic conditions. For example, in the presence of oxygen, the pHRR for sunflower pith reaches 300 W.g^{-1} , while in the absence of oxygen, its peak barely reaches 50 W.g^{-1} . In terms of THR and heat of complete

Table 1

Main values obtained in the pyrolysis combustion flow calorimetry (PCFC) test under anaerobic conditions for the plant aggregates.

Bioresource	pHRR1 (W.g^{-1})	TpHRR1 ($^\circ\text{C}$)	pHRR2 (W.g^{-1})	TpHRR2 ($^\circ\text{C}$)	THR (kJ.g^{-1})	Residue weight fraction (-)	Heat of complete combustion (kJ.g^{-1})
Hemp shiv	115	338	-	-	10.4	0.19	12.8
Sunflower pith	51	293	10	431	5.2	0.28	7.2
Rice husk	74	346	-	-	7.2	0.29	10.1
Wheat straw	84	339	-	-	7.6	0.25	10.1

Table 2

Main values obtained in the pyrolysis combustion flow calorimetry (PCFC) test under aerobic conditions for the plant aggregates.

Bioresource	pHRR1 (W.g^{-1})	TpHRR1 ($^\circ\text{C}$)	pHRR2 (W.g^{-1})	TpHRR2 ($^\circ\text{C}$)	THR (kJ.g^{-1})	Residue weight fraction (-)	Heat of complete combustion (kJ.g^{-1})
Hemp shiv	170	285	194	380	16.4	0.03	16.9
Sunflower pith	305	271	14	421	12.3	0.13	14.1
Rice husk	60	297	146	392	15.3	0.01	15.5
Wheat straw	106	281	274	420	14.8	0	14.8

combustion, the differences between bioresources are lower than in anaerobic conditions. Nevertheless, sunflower pith still exhibits the lowest values: 12.3 and 14.1 kJ.g^{-1} respectively versus 16.4 and 16.9 kJ.g^{-1} for hemp shiv.

The GHC is a property of fuels and indicates the quantity of energy released by the complete pyrolysis and combustion of a unit of this fuel, assuming that the water vapor is condensed, and the heat recovered. The GHC of all bioresources was measured inside a bomb calorimeter (Table 3). Since the sample weight must not exceed 1 g, it is not possible to test the biobased concrete using this apparatus because bioconcrete is not homogeneous at this scale. Therefore, only bioresources were assessed and the heat of combustion of binder was considered null. Note that the endothermic decomposition of gypsum consumes around 560 J.g^{-1} , but the heat released by water condensation almost compensates this contribution. Indeed, 20 % of water released by gypsum decomposition corresponds to 450 J.g^{-1} considering that the latent heat of water condensation is around 2250 J.g^{-1} . It also appears that considering the GHC in Euroclasses is detrimental to biobased hygroscopic materials and gypsum due to the heat released by water condensation. From our opinion, this value does not measure properly the fire hazard. Indeed, in case of burning, water does not condensate and usually remains as vapour.

Thus, if complete combustion is assumed, the bioresources tested presented GHC between 10 and 17 kJ.g^{-1} , the sunflower pith being the one that releases less heat (9.8 kJ.g^{-1}) and the hemp shiv more heat (16.5 kJ.g^{-1}), as it was also observed for the PCFC. When comparing Table 2 (THR under aerobic conditions) and 3 (GHC), bioresources showed very similar results.

Besides, the GHC allows classifying the biobased concretes regarding their fire reaction according to the Euroclass system. The binders do not release energy when burning, so with the fraction of bioresources in the bioconcretes and their GHC values, it is possible to predict if the bio-based concrete is A1, A2 or another rating, as seen in Fig. 5.

The GHC of the bioconcrete must be lower than 2 kJ.g^{-1} to be rated A1, and between 2 and 3 kJ.g^{-1} to be rated A2 [25]. Fig. 5 reveals the fraction allowed for each bioresource to have a bioconcrete classified as

Table 3

Values for the gross heat of combustion (GHC) measured with the bomb calorimeter.

Bioresource	GHC (kJ.g^{-1})
Hemp shiv	16.5
Sunflower pith	9.8
Rice husk	14.6
Wheat straw	14.8

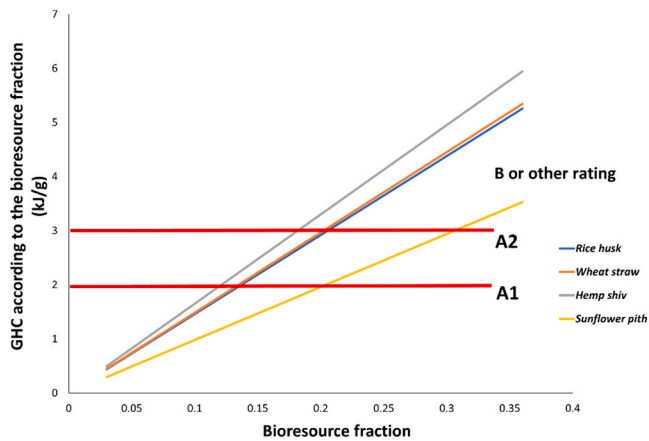


Fig. 5. Gross heat of combustion of bioconcretes according to the bioresource fraction in the Euroclass system.

A1 or A2. Since the hemp shiv releases more energy, its critical quantity is more limited than the other bioresources. For example, to have a A2 bioconcrete with hemp shiv, the maximum hemp fraction would be 0.17, while it is possible to have 0.20 for the rice husk and the wheat straw, and 0.29 for the sunflower pith. When these fractions are exceeded, the best rating which can be expected is B.

4.2. Flammability of biobased concretes at bench scale

For ranking in A2 to D classes, SBI test is mandatory. SBI is known to be well predicted using cone calorimeter and phenomenological modelling (the fire behaviour rankings found in the Appendix A of this article is based on the ConeTools software). When ignition does not occur in cone calorimeter, the predicted performance in SBI is A2 or B (the GHC must then be used to classify the material between these two classes). It is the reason why this article focuses deliberately on the ignition of bioconcretes.

Fig. 6 shows a typical HRR versus time curve measured by cone calorimetry for a flammable bioconcrete (i.e. when ignition occurs). In this example, bioconcrete is made of earth and hemp (weight ratio 1.8 with a density of 254 kg.m^{-3}). The ignition process occurs early, after around 10 s, then a pHRR is quickly reached (around $110\text{--}120 \text{ kW.m}^{-2}$). Then, the HRR drops rapidly, and the pyrolysis moves from anaerobic to aerobic when the flame vanishes (flame out). During this second stage, there is no more flame, but due to the heat from the cone calorimeter, the organic part of the material is slowly oxidized (and decomposition of the binder, as gypsum, can also occur). This is the reason why the HRR

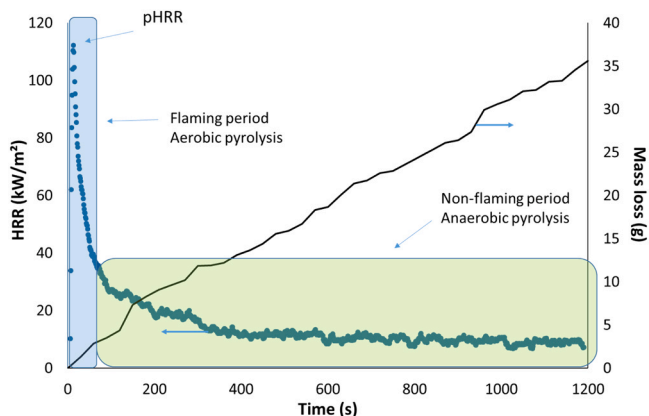


Fig. 6. HRR curves for a sample of EH254 in cone calorimeter tests at 50 kW.m^{-2} .

stabilizes between 20 and 40 kW.m^{-2} , which is a relatively low value.

When the material does not ignite, which is usual for most biobased concretes, especially the densest ones, no peak is observed. Actually, HRR rises to values up to $20\text{--}30 \text{ kW.m}^{-2}$, once again due to the thermo-oxidation of the bioresources. It is noteworthy that all the tests were interrupted after 20 min, even if the HRR was not yet null.

In general, regarding the time to ignition (TTI), it tends to be smaller as the density of the bioconcrete decreases. Indeed, when ignition occurs, it happens more quickly for the lighter bioconcretes, since they present a low conductivity, therefore the temperature increases faster. In addition, it was observed that lime tends to delay the ignition and gypsum tends to make this phenomenon more difficult to occur. Moreover, it was also found that wheat straw fibres tend to straighten at the beginning of the test, providing hot spots which favour ignition.

Mass loss was measured for some samples. Fig. 6 also shows the mass loss of EH254 during the 20 min test. The mass loss rate was constant (around 1.8 g.min^{-1}) throughout the whole test since the flaming period was very short (typically it lasts less than 60 s). When a flame occurs during a test, the inward heat flux into the sample increases, because a heat flux originating from the flame is added to that of the cone. In the present study, as the flame out occurred quickly, the mass loss rate remained almost constant. The mass loss rate depended on the bioconcrete, especially on its density.

After the cone calorimeter tests, the surface of residues tended to turn black and, in some cases, white, as seen in Fig. 7. The black part is the result of the carbonization of the bioresource, which corresponds to the pyrolyzed zone. The thickness of this zone varied over the 20 min of the test. It tends to increase as density decreases. The white colour results from the very high temperatures which oxidized the char, leaving only ashes.

Fig. 8 plots the residual fraction of all bioconcretes (after the 20 min cone calorimeter test at 50 kW.m^{-2}) versus their initial density. A unique trend is revealed, regardless of the constituents of the concrete. In other words, mass loss and mass loss rate do not depend on the binder or biobased resource used, but only on the density of this bioconcrete (therefore, indirectly on the quantity of the bioresource in the bioconcrete). A density threshold around $400\text{--}500 \text{ kg.m}^{-3}$ can be identified. Above this threshold, the residual fraction is higher than 90 % and increases slowly as the density increases. Below this threshold, the mass loss at 20 min decreases more quickly as the density decreases.

Fig. 9 shows the pHRR versus the density for all the biobased concretes. For a density above 600 kg.m^{-3} , no concrete ignited during the cone calorimeter test under a heat flux of 50 kW.m^{-2} . However, below this value, some concretes, which are represented by red points, ignited, but not all. They usually reached a pHRR between 50 and 140 kW.m^{-2} , whereas those that did not ignite reached a pHRR (corresponding only to thermo-oxidation of bioresources) between 20 and 40 kW.m^{-2} . A flash (i.e. flame during a few seconds) was also observed in a single case (for a biobased concrete with a density close to 900 kg.m^{-3}). This phenomenon, which may be related to a surface irregularity, is not considered as ignition, because the flame did not last more than a few seconds. In addition, three samples made with lime as a binder ignited after a relatively long time-to-ignition (around 100 s compared to 10 s) with a pHRR lower than 50 kW.m^{-2} . These results were noticed, but they do not provide enough experimental data to draw clear conclusions regarding this binder. Moreover, four red squares corresponding to samples covered by microfilms (surface-deposited microfilm made of earth and water) ignited, even though it appears that the pHRR are lower for these bioconcretes. As a conclusion, the greatest hazard of ignition was observed for light concretes, and a critical density value around 600 kg.m^{-3} can be proposed as an approximate criterion to assess this hazard (when heat flux was fixed to 50 kW/m^2). This threshold is close to the threshold defined in our first article devoted only to hemp-based bioconcretes (500 kg.m^{-3}) [10].

As discussed above, density appears to be the main parameter controlling the flammability of bioconcretes. The densest concretes (which

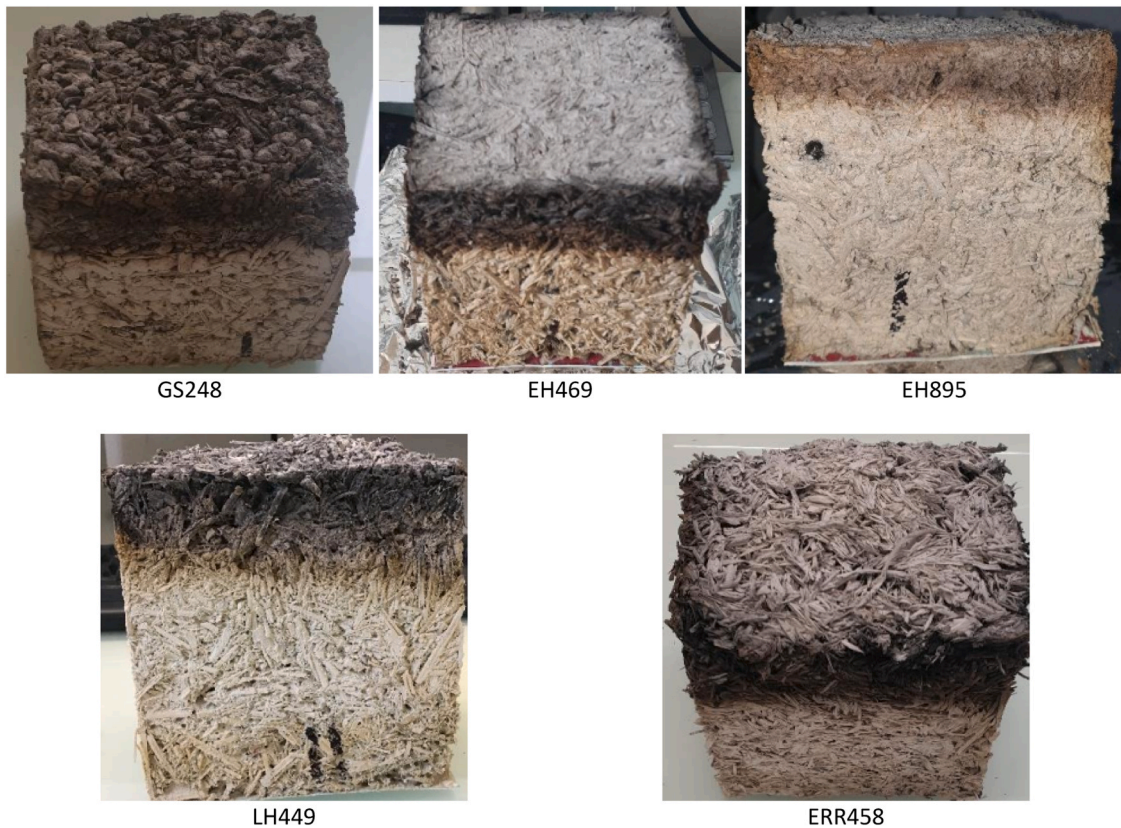


Fig. 7. Biobased concretes samples after cone calorimeter tests at 50 kW.m^{-2} and their pyrolyzed area (dark area of the 10 cm-side cube).

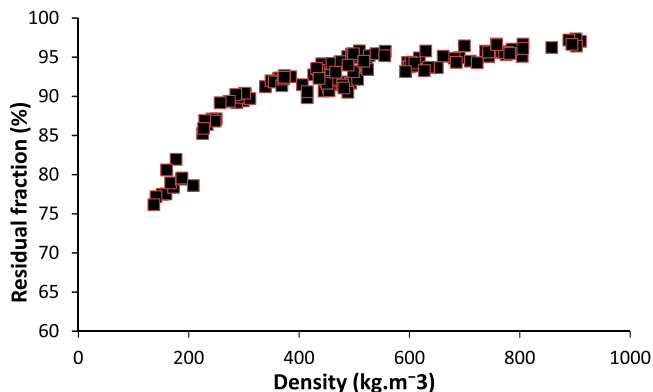


Fig. 8. Residual fraction versus density.

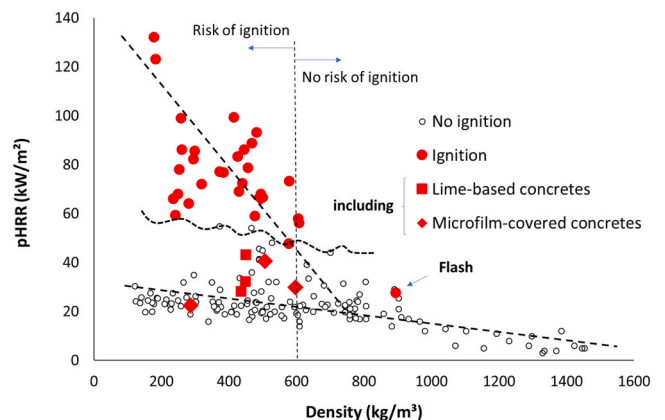


Fig. 9. pHRR versus density in cone calorimeter tests at 50 kW.m^{-2} .

generally contain low amount of bioresources) do not ignite and their mass loss after 20 min is lower. The density depends on the nature of bioresource and the binder/bioresource ratio. Hence, bioconcretes made of rice husk have a quite high density because rice husk has a small aspect ratio, which does not allow low densities to be achieved. Therefore, these concretes tend not to ignite. Nevertheless, density might not be the only relevant parameter. Indeed, many bioconcretes containing sunflower pith do not ignite despite their very low density. This is most probably due to the low heat of combustion of sunflower pith. In the following part, we attempt to propose simple parameters to predict the risk of ignition of bioconcretes.

4.3. Thermo-physical properties of biobased concretes for predicting ignition

Among the relevant parameters influencing the ignition, the heat of

combustion (as discussed below and measured via PCFC for example) is of first importance. However, thermo-physical properties are also to be considered.

Materials can be considered as thermally thin or thermally thick, depending on their thermal behaviour. A thermally thin material is usually defined as a material where the whole volume is at the same temperature (no heat gradient). This definition is relevant for thin materials with a high thermal conductivity. On the contrary, in thermally thick materials, heat diffusion from the surface to the bulk contributes to limit the surface temperature. From another point of view, in thick materials with a very low heat conductivity, only a top volume absorbs the heat from the radiant cone and its temperature increases fast and homogeneously, while the underlying volume does not heat (or very slowly because the heat diffusion is negligible). Such materials can be considered as bilayer materials with a thermally thin top volume

monitoring the whole behaviour (at least at the beginning of the test, when the pHRR occurs).

Sonnier et al. proposed a model predicting the pHRR of such thick but thermally thin materials including many biobased materials [26]. They proved that their model was relevant in the case of many bioresources like bulk fibres used as insulating materials for construction, dense woods, and also lightweight biobased concretes [26].

While biobased concretes (at least those which can ignite, i.e. the lightest ones) are thermally thin, thermal conductivity should not be a relevant parameter to predict the ignition. Indeed, the thermal conductivity was measured for a large series of bioconcretes. Thermal conductivity does not exceed $0.1 \text{ W.m}^{-1}.\text{K}^{-1}$ for the lightest bioconcretes ($< 500 \text{ kg.m}^{-3}$) confirming that heat transfer is limited.

The specific heat capacity of the biobased concretes constituents was measured with a Calvet calorimeter for a given temperature range, as previously described (Table 4). The binders specific heat capacity is in accordance with the literature, the measured values being a little higher since this property tends to increase as the temperature increases.

The bioresources specific heat capacity also appears to be in accordance with the literature. The difference between the literature and the measured values is probably related to the temperature dependence of this property, but also to the intrinsic variability of plant aggregates, which for the same material, can present considerable different chemical composition, therefore different properties, induced by geographical location, soil conditions and even the processing method applied to obtain the bio-aggregates, as discussed by Magniont and Escadeillas [27].

Moreover, the value for the decomposition enthalpy of the binders was also investigated with this device (Table 4). The decomposition enthalpy of gypsum was reported in the literature to be 560 J.g^{-1} at around $190 \text{ }^\circ\text{C}$ [28], which is also in accordance with our results. Concerning lime, this material undergoes the decomposition process with an enthalpy around 1150 J.g^{-1} but at elevated temperature ($> 430 \text{ }^\circ\text{C}$ for calcium hydroxide [29] and $> 600 \text{ }^\circ\text{C}$ for calcium carbonate). Finally, the earth does not present an endothermic decomposition like the other binders, therefore the measured value is very small.

Regarding the bioresource pyrolysis enthalpy, it is important to highlight that it is a difficult measurement to conduct and in the literature, a wide range of values was found. For the present article, the recent study of Jerzak et al. [30] was taken as reference since their work presents, among other materials, the heat required for the pyrolysis of wood, which is lignocellulosic, like our bioresources. The value found for a medium density fibreboard (MDF) was 2016.8 J.g^{-1} .

4.4. Criterion for ignition prediction

Based on the various biobased concretes characterised (more than 150 – cf. Table in appendix A), this study aims at distinguishing concretes that ignite from those that do not ignite. Even though density is a relevant parameter (as discussed above), it is not satisfying to consider only this parameter to predict their flammability. Indeed, many light

bioconcretes (of density $< 600 \text{ kg.m}^{-3}$) do not ignite (e.g. several bioconcretes based on sunflower pith).

Burning rate is a balance between the energy released from the material and the energy needed to heat and pyrolyze it. The first term will be here called exothermic contribution and the second one endothermic contribution. For biobased concretes, the exothermic contribution comes from the bioresources, since the mineral part, that is, the binder, does not burn, and consequently does not release energy. On the other hand, the endothermic contribution comes from the energy stored by the material during heating, the bioresource pyrolysis and the possible binder endothermic decomposition.

Lyon et al. proposed a criterion for piloted ignition of combustible solids [38]. Regardless of the solid, the onset of piloted ignition of combustible polymers is predicted by a gas phase combustion energy density of 1.9 MJ.m^{-3} , which describes the lower flammability limit of fuel vapor air mixtures [38]. Thus, in order to represent the exothermic contribution in the combustion process, the chosen parameter was the energy density (MJ.m^{-3}) of the bioconcrete. It is the product between the THR of the bioresource (as measured via the PCFC in anaerobic conditions, considering a complete combustion, which is a reasonable hypothesis, because no flame inhibitor was used), the density of the bioconcretes (ρ) and the fraction of bioresources in the bioconcrete (Eq. 1).

$$\text{Exothermic contribution} = \text{THR} \left(\frac{\text{kJ}}{\text{g}} \right) \times \rho \left(\frac{\text{kg}}{\text{m}^3} \right) \times \text{bioresource fraction} \quad (1)$$

The thermal properties of a material which are relevant to fire behaviour include the thermal conductivity (k), the density (ρ) and the specific heat capacity (c_p) [39]. However, as discussed above, for thermally thin materials such as bioconcretes, the heat conduction is limited because of its low thermal conductivity [26]. Consequently, after disregarding the thermal conductivity, the two remaining thermal properties pertinent to fire behaviour are the density (ρ) and the specific heat capacity (c_p).

During combustion, the binders tend to absorb energy and slow down the heating process and, therefore, delay the burning rate and the ignition. However, some binders, such as gypsum, in addition to absorbing energy, undergo an endothermic decomposition, which reduces even more the pyrolysis rate and, consequently, slows down the fire spread. The conventional specific heat capacity (c_p) does not consider this endothermic phenomenon. In order to include this contribution, the conventional specific heat capacity was adapted in this study. This modified thermal property was calculated by adopting the equation of the thermal decomposition to volatile fuel H_g (J.g^{-1}), which is described by Lyon and Quintiere [38] (Eq. 2). The first term represents the energy required to heat the material (bioresource and binder) from room temperature to the ignition temperature considering its specific heat capacity. In this work, $c_p(T)$ is of course the mean value of the weighted specific heat capacities of the binder and the bioresource. As explained above, $c_p(T)$ was estimated as the average value of c_p

Table 4

Main values obtained in the Calvet calorimeter and in the literature for the constituents of the bioconcretes used in the criterion for ignition prediction.

Bioconcrete constituent	Measured specific heat capacity* ($\text{J.g}^{-1}.\text{K}^{-1}$)	Specific heat capacity in the literature ($\text{J.g}^{-1}.\text{K}^{-1}$)	Reference	Measured endothermic enthalpy (J.g^{-1})	Enthalpy in the literature (J.g^{-1})	Reference
Gypsum	1.04	0.95	[31]	563.3	560	[27]
Earth	1.01	0.94	[32]	40.5	N/A	-
Lime	1.09	1	[33]	54.3	1150	[28]
Rice husk	2	1.5	[34]	N/A	2016.8	[30]
Wheat straw	1.7	1.63	[35]	N/A	2016.8	[30]
Hemp shiv	1.6	1.6	[36]	N/A	2016.8	[30]
Sunflower pith	1.5	1.3	[37]	N/A	2016.8	[30]

* Measured specific heat capacities are an average between the specific heat capacity at room temperature and at higher temperatures ($100 \text{ }^\circ\text{C}$ for the bioresources and $300 \text{ }^\circ\text{C}$ for the binders).

measured using Calvet calorimeter between room temperature and 300 °C for the binder and between room temperature and 100 °C for the bioresource. The second term is the crystalline heat of fusion, which is null here since there is no melting phenomenon. The last term is the heat required to vaporize a unit mass of the material (binder decomposition or bioresource pyrolysis). T_0 is room temperature (25 °C) and T_{ign} was supposed to be 450 °C, which is close to the surface temperature reached during burning as evidenced in our previous work [10]. This temperature also corresponds quite well to the end of the pyrolysis range of bioresources measured in PCFC (in anaerobic conditions).

$$Hg = \int_{T_0}^{T_{ign}} c_p(T) dT \left(\frac{J}{g} \right) + \Delta h_m \left(\frac{J}{g} \right) + \Delta h_v \left(\frac{J}{g} \right) \quad (2)$$

The Eq. 2 was applied for each biobased concrete constituent (binder and bioresource) in order to find the thermal energy needed to heat and decompose each bioconcrete (H_g). Then this value was divided by the pyrolysis temperature range ΔT (i.e. range between ambient temperature and pyrolysis temperature, 25 °C and 450 °C respectively) to calculate the modified specific heat capacity (c_{pmod} in $J.g^{-1}.K^{-1}$) for each bioconcrete, as shown in the Eq. 3.

$$C_{pmod} = \frac{Hg \left(\frac{J}{g} \right)}{\Delta T (^\circ C)} \quad (3)$$

In order to illustrate this calculation, the bioconcrete **EW296** is used as an example. This biobased concrete is composed of 64 % earth and 36 % wheat straw. The specific heat capacity of the earth used is 1.01 $J.g^{-1}.K^{-1}$ and its decomposition energy 40.5 $J.g^{-1}$. It results in a value of 469.75 $J.g^{-1}$ for H_g of the binder ($1.01 J.g^{-1}.K^{-1} \times 425 K + 40.5 J.g^{-1}$). The specific heat capacity of wheat straw is 1.7 $J.g^{-1}.K^{-1}$ and its decomposition energy relative to the mass loss fraction (measured in the PCFC in anaerobic conditions) equals to 1512.6 $J.g^{-1}$ ($2016.8 J.g^{-1} \times 0.75$), which results in 2235.1 $J.g^{-1}$ for H_g of the bioresource ($1.7 J.g^{-1}.K^{-1} \times 425 K + 2016.8 J.g^{-1} \times 0.75$). Consequently, the total H_g value, after considering the proportions of each constituent ($0.64 \times 469.75 J.g^{-1} + 0.36 \times 2235.1 J.g^{-1}$), equals to 1105.3 $J.g^{-1}$. Finally, this value is divided by the pyrolysis temperature range (i.e. range between ambient temperature and pyrolysis temperature i.e. 425 K), to obtain a modified specific heat capacity of 2.6 $J.g^{-1}.K^{-1}$. If gypsum is used instead of earth, the modified specific heat capacity becomes 3.4 $J.g^{-1}.K^{-1}$, illustrating the major influence of dehydration. Note that the decomposition of lime may also enhance the endothermic contribution, but it occurs at much higher temperature. It has been evidenced in our previous study [10] that the surface temperature can reach 600 °C for the lightest concretes, but only after ignition. At ignition, the surface temperature does not exceed 450 °C. Therefore, in this study, the decomposition of lime was not considered. For the calculation of endothermic contribution, only the small measured endothermic enthalpy (54.3 $J.g^{-1}$ – cf. Table 4) was considered.

It is noteworthy to point out that the same unit of measurement of the conventional specific heat capacity was kept for the modified specific heat capacity (i.e. $J.g^{-1}.K^{-1}$).

Thus, it is possible to calculate the volumetric modified heat capacity P ($J.m^{-3}.K^{-1}$) as the endothermic contribution. As shown in Eq. 4, it is the product of the density and the modified specific heat capacity.

$$P = C_{pmod} \left(\frac{J}{g.K} \right) \times \rho \left(\frac{g}{m^3} \right) \quad (4)$$

In order to obtain the two contributions (endothermic and exothermic) with the same units of measurement, P was multiplied by ΔT , that is, 425 K (i.e. the range between room temperature and pyrolysis temperature) and then, finally, comparing these two contributions leads to Fig. 10.

Biococoncretes which ignite are characterized by a high exothermic

contribution and/or a low endothermic contribution. In other words, these properties allow revealing an area where most of the biobased concretes ignite when they are tested in the cone calorimeter at 50 $kW.m^{-2}$, which are represented by the full figures with a red outline.

In the following part, we propose a flammability criterion to separate two areas: the flammable zone, where ignition tends to occur and the non-flammable zone, where bioconcretes are not prone to ignition.

4.5. Criterion to predict the ignition of bioconcretes (flammability criterion)

Based on the criterion for piloted ignition of combustible solids already discussed, Lyon and Quintiere [38] proposed a critical heat release rate (called HRR^*) for piloted ignition that is independent of the fuel type. Its value was evaluated at 24 $kW.m^{-2}$.

According to this criterion, ignition occurs if:

$$m_f'' \times h_c > HRR^*$$

m_f'' being the fuel mass flux (in $g.s^{-1}.m^{-2}$) and h_c its combustion energy (in $kJ.g^{-1}$). The fuel mass flux can be deduced from the thickness of the pyrolyzed zone (black layer on the biobased concrete) obtained after the 20 min test in the cone calorimeter at 50 $kW.m^{-2}$. Indeed, it was previously shown that the mass loss rate is constant over the whole test.

Fig. 11 shows the thickness of the pyrolyzed zone after 20 min (at the end of the test) versus the endothermic contribution, previously defined. The thickness L (in m) roughly decreases when this contribution increases according to the Eq. 5:

$$L = 2.6734 \times \text{Endothermic contribution}^{-0.761} \quad (5)$$

Despite some uncertainties due to the thickness measurements (indeed, the pyrolysis front does not move exactly at the same rate on all the samples faces), all bioconcretes exhibit the same tendency, regardless of the nature of the binder and the bioresource.

The pyrolysis rate ($m.s^{-1}$) can be calculated by dividing the thickness of the pyrolysis zone L , calculated with the Eq. 5, by the test duration (i.e. 20 min):

$$\text{Pyrolysis rate} = \frac{L (m)}{\text{Test duration} (s)} \quad (6)$$

Finally, the experimental fuel mass flux m_f'' ($g.s^{-1}.m^{-2}$) was calculated according to the Eq. 7. Note that this fuel mass flux corresponds only to the bioresource, since the binder does not release heat.

$$m_f'' = \text{Pyrolysis rate} \left(\frac{m}{s} \right) \times \rho \left(\frac{g}{m^3} \right) \times \text{bioresource fraction} \times (1 - \mu) \quad (7)$$

with μ being the residue of the biobased resource (i.e. 15 %).

The mass loss rate remains constant during the test, so the fuel mass

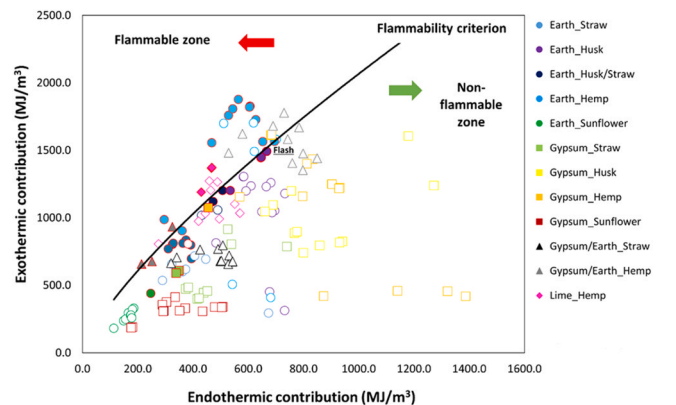


Fig. 10. Exothermic contribution versus the endothermic contribution as a flammability criterion.

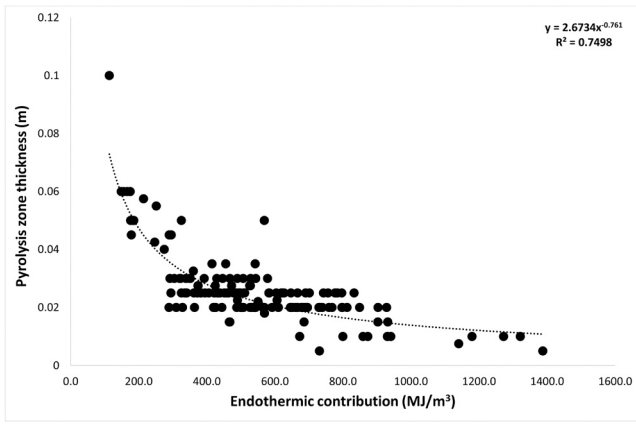


Fig. 11. Pyrolyzed zone thickness versus the endothermic contribution.

flux can be considered as constant. Finally, considering the relation proposed by Lyon and Quintiere between HRR^* , m_f' and h_c , a critical value for the combustion energy H_c^* ($\text{kJ}\cdot\text{g}^{-1}$) was obtained as follows:

$$H_c^* = \frac{24 \left(\frac{\text{kW}}{\text{m}^2} \right)}{m_f' \left(\frac{\text{g}}{\text{m}^2 \cdot \text{s}} \right)} \quad (8)$$

From H_c^* , the critical exothermic contribution ($\text{kJ}\cdot\text{m}^{-3}$) was calculated as follows:

$$\text{Exothermic contribution}^* = H_c^* \left(\frac{\text{kJ}}{\text{g}} \right) \times \text{bioresource fraction} \times \rho \left(\frac{\text{g}}{\text{m}^3} \right) \times (1 - \mu) \quad (9)$$

Note that the residue of the biobased resource used above (15 %) is lower than those measured at the PCFC under anaerobic conditions (Table 1). Therefore, this value is conservative (i.e. it is disadvantageous and provides a margin of safety).

Finally, the criterion for ignition defined in Fig. 10 is given in the Eq. 10:

$$\text{Exothermic contribution} = f(c_{p_{\text{mod}}} \times \rho \times \Delta T) \quad (10)$$

This criterion is defined by the black curve in the Fig. 10. This boundary is only approximative. Some materials are able to ignite in the non-flammable area but are very close to the boundary (this might be tackled by applying a safety penalty coefficient if this criterion should be used in design standards). Similarly, some materials do not ignite but are seen in the flammable zone. This is probably due to several limits of this approach: the thermophysical properties are not accurate (an average was made to obtain the specific heat capacity values used in this study, for example), combustion is assumed to be complete (since the THR measured via PCFC was considered). In addition, bioconcretes are considered to be thermally thin, which is probably not accurate anymore for the denser ones, but this hypothesis has no relevance since denser biobased concretes do not ignite. Finally, some values are only approximate (residue of the biobased resource, heat of pyrolysis). Nevertheless, this criterion allows a reasonable estimation of the risk of ignition for a specific bioconcrete from several properties which are relatively easy to measure: the energy released by the bioresources, the bioconcrete density, the specific heat capacity of constituents and their heat of decomposition. Moreover, once these properties measured, the risk can be predicted for any combination of binder and bioresource.

5. Conclusion

The fire reaction of a large set of biobased concretes according to the

Euroclass system was predicted. Four different bioresources and three different binders were used to obtain conclusions which are relevant for all bioconcretes (or most of them).

The rating A1 and A2 was calculated based on the GHC and on the proportion of the constituents of each formulation. The binders do not release energy when burned, therefore it was possible to calculate the critical bioresource fraction for the four plant aggregates studied in the present work.

Fire behaviour of different biobased concretes was assessed using cone calorimeter tests on more than 150 samples to cover a large range of composition and density. Ignition can be predicted considering two contributions: one is the heat which can be released by the bioconcretes (i.e. by their organic fraction) and one is the heat needed to heat and decompose the constituents of the bioconcretes (binder and bioresources). A flammability criterion has been independently defined, separating a flammable zone (where bioconcretes can ignite) from a non-flammable zone. This criterion considers the role of the binder and the impact of its endothermic decomposition on the heating process of the biobased concrete.

It was found that the density of the biobased concretes has a great influence on the thickness of the pyrolyzed zone, that is, in the fire behaviour of these concretes. Nevertheless, other parameters also have an important role. When the biobased concrete is burned, its organic part is responsible for releasing energy and the more energy this material releases, the greater the likelihood of the concrete to ignite. In addition, the binder is also an important parameter to be analysed. In the flammable zone, the materials are often made of earth. Indeed, earth is inert, while gypsum (and maybe lime) decomposes endothermically and then enhance the heat needed to decompose the constituents. The decomposition of gypsum is more likely to begin to consume the energy of the fire before lime. Therefore, gypsum would be the best binder (of the 3 studied here) regarding the fire behaviour of the biobased concretes. However, additional studies should be carried out on the influence of these materials, especially lime, since not much can be concluded about it.

In closing, based on the values of the GHC (i.e. the A1, A2 or worst rating) and the cone tests (i.e. the prediction of the SBI test), this article makes it possible to properly estimate the fire reaction behaviour of biobased concretes according to the Euroclass.

Lastly, further work will be carried out to study the fire resistance of the biobased concretes, as well as to investigate the smouldering fire, since this phenomenon is very common in insulating materials.

CRedit authorship contribution statement

Laurent Aprin: Writing – review & editing, Validation, Supervision, Conceptualization. **Pierre Delot:** Writing – review & editing, Supervision, Methodology. **Arnaud Regazzi:** Writing – review & editing, Validation, Supervision, Conceptualization. **Placide Uwizeyimana:** Writing – review & editing, Validation, Supervision, Conceptualization. **Arthur Hellouin de Menibus:** Writing – review & editing, Validation, Supervision, Methodology, Conceptualization. **Marc Potin:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Sherryhane Labeni:** Investigation, Data curation. **Laurent ferry:** Writing – review & editing, Validation, Supervision, Conceptualization. **Rodolphe Sonnier:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization. **Tania Lopes:** Writing – original draft, Methodology, Investigation, Formal analysis, Data curation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The work was supported by some companies or associations dealing with the use of biobased concretes as

insulating materials or renders. These companies are represented through some co-authors of the article. The work has been carried out loyally, not to promote these materials but to identify a criterion to predict the ignition of biobased concretes in order to evaluate their fire reaction.

Data availability

Data will be made available on request.

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Appendix A. (formulations & fire behaviour)

Formulations	Binder fraction	Bioresource fraction*	Fire behaviour**
EW296	0.64	0.36	D
EW298	0.64	0.36	D
EW281	0.64	0.36	
EW287 (MF1)	0.64	0.36	
EW339 (MF2)	0.64	0.36	
EW384	0.71	0.29	C
EW373	0.71	0.29	C
EW363	0.76	0.24	
EW440	0.76	0.24	C
EW352	0.80	0.20	A2/B
EW495	0.81	0.19	C
EW509	0.81	0.19	
EW474	0.83	0.17	
EW488	0.83	0.17	
EW608	0.85	0.15	
EW1288	0.97	0.03	
ER521	0.64	0.36	
ER522	0.64	0.36	
ER578	0.64	0.36	A2/B
ER579	0.64	0.36	C
ER596 (MF1)	0.64	0.36	
ER597(MF2)	0.64	0.36	A2/B
ER477	0.64	0.36	C
ER618	0.71	0.29	
ER599	0.71	0.29	
ER756	0.76	0.24	
ER737	0.76	0.24	
ER581	0.80	0.20	A2/B
ER805	0.81	0.19	
ER910	0.81	0.19	
ER902	0.83	0.17	
ER889	0.83	0.17	
ER1192	0.95	0.05	
ER1384	0.97	0.03	
ERR426	0.64	0.36	C
ERR457	0.64	0.36	C
ERR387	0.64	0.36	
ERR499	0.71	0.29	
ERR499	0.71	0.29	
EH469	0.64	0.36	
EH482	0.64	0.36	C
EH500	0.64	0.36	A2/B
EH453	0.64	0.36	
EH414	0.64	0.36	
EH261	0.64	0.36	C
EH608	0.71	0.29	A2/B
EH606	0.71	0.29	A2/B
EH690	0.76	0.24	
EH681	0.76	0.24	
EH430	0.80	0.20	C
EH804	0.81	0.19	
EH767	0.81	0.19	
EH902	0.83	0.17	
EH894 (Flash)	0.83	0.17	A2/B
EH512	0.85	0.15	
EH965	0.95	0.05	
EH1297	0.97	0.03	
ES158	0.64	0.36	

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(continued)

Formulations	Binder fraction	Bioresource fraction*	Fire behaviour**
ES177	0.64	0.36	
ES172	0.64	0.36	
ES235	0.64	0.36	C
ES159	0.71	0.29	
ES167	0.71	0.29	
ES186	0.71	0.29	
ES187	0.71	0.29	
ES121	0.71	0.29	
ES208	0.76	0.24	
GW249	0.68	0.32	
GW242	0.68	0.32	C
GW376	0.68	0.32	
GW407	0.74	0.26	
GW296	0.79	0.21	
GW303	0.79	0.21	
GW609	0.83	0.17	
GW365	0.84	0.16	
GW376	0.84	0.16	
GW349	0.85	0.15	
GW355	0.85	0.15	
GR488	0.68	0.32	
GR467	0.68	0.32	
GR535	0.68	0.32	A2/B
GR882	0.74	0.26	
GR603	0.79	0.21	
GR610	0.79	0.21	
GR1041	0.83	0.17	
GR710	0.84	0.16	
GR661	0.84	0.16	
GR786	0.85	0.15	
GR777	0.85	0.15	
GH479	0.68	0.32	
GH481	0.68	0.32	
GH320	0.68	0.32	A2/B
GH423	0.74	0.26	
GH650	0.79	0.21	
GH634	0.79	0.21	
GH649	0.83	0.17	
GH743	0.84	0.16	
GH743	0.84	0.16	
GH775	0.85	0.15	
GH772	0.85	0.15	
G25E75H1333	0.97	0.03	A2/B
G50E50H1453	0.97	0.03	A2/B
G75E25H1445	0.97	0.03	A2/B
GH1328	0.97	0.03	
GS225	0.68	0.32	
GS248	0.68	0.32	
GS214	0.68	0.32	
GS229	0.74	0.26	
GS227	0.74	0.26	
GS140	0.74	0.26	
GS136	0.74	0.26	
GS301	0.79	0.21	
GS285	0.79	0.21	
GS368	0.84	0.16	
GS406	0.84	0.16	
GS435	0.85	0.15	
GS431	0.85	0.15	
EGW273	0.66	0.34	
EGW257	0.66	0.34	
EGW372	0.73	0.27	
EGW373	0.73	0.27	C
EGW459	0.78	0.22	
EGW476	0.78	0.22	
EGW495	0.82	0.18	
EGW527	0.82	0.18	
EGW495	0.82	0.18	
EGW498	0.82	0.18	
EGW539	0.84	0.16	
EGW556	0.84	0.16	
EGW555	0.84	0.16	
EGH454	0.66	0.34	
EGH415	0.66	0.34	
G25E75H179	0.65	0.35	D

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Formulations	Binder fraction	Bioresource fraction*	Fire behaviour**
G50E50H254	0.65	0.35	A2/B
G75E25H184	0.65	0.35	D
EGH592	0.73	0.27	
EGH627	0.73	0.27	
EGH685	0.78	0.22	
EGH722	0.78	0.22	
EGH743	0.82	0.18	
EGH781	0.82	0.18	
EGH858	0.84	0.16	
EGH805	0.84	0.16	
LH449	0.71	0.29	A2/B
LH450	0.71	0.29	A2/B
LH265	0.71	0.29	A2/B
LH466	0.74	0.26	
LH436	0.74	0.26	A2/B
LH524	0.77	0.23	
LH498	0.77	0.23	
LH517	0.81	0.19	
LH488	0.81	0.19	
LH699	0.85	0.15	
LH629	0.85	0.15	
LH757	0.87	0.13	
LH758	0.87	0.13	

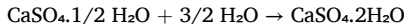
*bioresource fraction after curing

**calculated using ConeTools software

The method presented here requires calculating the final (i.e. after curing) bioresource and binder fractions as accurately as possible.

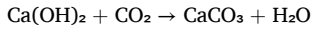
A fraction of the water added during manufacturing is used to hydrate the binder and another fraction of the water is fixed by the bioresource or removed during drying. Only the part used to hydrate the binder (gypsum) is considered for the final calculation of the bioresource fraction.

- For **gypsum**, the complete hydration reaction is:



It implies that 145 g of gypsum mobilizes 27 g of water. For example, in the GH320 formulation which contains 32 g of plaster and 18 g of hemp (and 50 g of water), only 6 g of water is necessary for hydration ($= 32 \times 27 / 145$). Consequently, hemp represents 32 % of the final product ($= 18 / (18 + 32 + 6)$).

- For **hydraulic lime**, $\text{Ca}(\text{OH})_2$ does not hydrate but carbonates, according to the following reaction:



It implies that 74 g of lime becomes 100 g of CaCO_3 if carbonation is complete. For example, in the LH449 which initially contains 64 g of $\text{Ca}(\text{OH})_2$ and 36 g of hemp (and water), the bioconcrete after curing and carbonation contains 86.5 g of CaCO_3 and 36 g of hems, i.e. 29.5 % of hemp.

- For **earth**, we considered that all water was removed during drying.

Obviously, these calculations must be considered as approximative.

Appendix B. (Properties)

Formulation	Endothermic contribution (MJ.m ⁻³)	Exothermic contribution (MJ.m ⁻³)	TTI (s)	pHRR (kW.m ⁻²)	Residual fraction (after 20 min)
EW296	325.70	809.86	10	18	0.90
EW298	328.90	817.79	10	17	0.89
EW281	310.60	768.82	26	64	0.90
EW287 (MF1)	N/A	N/A	56	22.5	0.89
EW339 (MF2)	N/A	N/A		16	0.91
EW384	374.70	847.66	14	99	0.93
EW373	363.90	823.19	10	23	0.93
EW363	323.10	662.11		21	0.92
EW440	392.50	804.20	11	72	0.94
EW352	289.60	535.04		24	0.91
EW495	394.50	715.07	7	68	0.95
EW509	405.90	735.57		17	0.96
EW474	362.30	612.41		28	0.94
EW488	373.00	630.75		41	0.95
EW608	446.60	693.12		16	0.98
EW1288	673.30	293.66		6	0.99
ER521	582.40	1313.93		19	0.94
ER522	583.60	1316.45		19	0.94

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Formulation	Endothermic contribution (MJ.m ⁻³)	Exothermic contribution (MJ.m ⁻³)	TTI (s)	pHRR (kW.m ⁻²)	Residual fraction (after 20 min)
ER578	645.60	1456.56	16	48	N/A
ER579	647.30	1460.34	17	73	N/A
ER596 (MF1)	N/A	1501.92		17	0.94
ER597 (MF2)	N/A	1504.44	45	30	0.94
ER477	535.30	1202.04	43	59	0.90
ER618	610.50	1254.95		23	0.95
ER599	592.00	1216.99		19	0.94
ER756	618.80	1270.92		24	0.96
ER737	665.10	1239.67		31	0.96
ER581	483.50	813.40		24	0.93
ER805	649.00	1071.71		17	0.97
ER910	732.90	1210.30		18	0.97
ER902	696.20	1073.38		25	0.97
ER889	686.10	1057.91		29	0.97
ER1192	676.80	417.20		11	0.99
ER1384	731.10	290.64		12	0.99
ERR426	472.80	1120.84	12	83	0.93
ERR457	507.20	1202.31	9	79	0.93
ERR387	431.00	1017.04		19	0.93
ERR499	489.90	1057.23		20	0.93
ERR499	489.90	1057.23		20	0.93
EH469	528.80	1771.69	29	89	0.92
EH482	543.80	1821.96	26	93	0.92
EH500	564.60	1891.13	15	67	N/A
EH453	511.60	1713.85		23	0.91
EH414	468.10	1568.32	28	99	0.90
EH261	295.90	986.58	19	86	0.85
EH608	606.70	1853.80	45	56	0.94
EH606	604.30	1846.49	38	58	0.94
EH690	627.50	1740.06		23	0.95
EH681	619.00	1716.37		31	0.95
EH430	360.60	903.00	22	69	0.89
EH804	652.60	1604.98		32	0.95
EH767	622.30	1530.36		21	0.96
EH902	701.60	1611.50		21	0.96
EH894 (Flash)	695.10	1596.68	54	28	0.97
EH512	382.10	806.40		22	0.94
EH965	542.30	506.63		16	0.98
EH1297	681.10	408.56		10	0.99
ES158	166.50	297.27		24	0.78
ES177	185.80	331.91		23	0.82
ES172	181.00	323.11		24	0.78
ES235	247.40	439.92	11	66	0.82
ES159	149.10	241.13		25	0.81
ES167	156.00	252.29		28	0.79
ES186	174.00	281.24		26	0.79
ES187	175.20	283.35		26	0.80
ES121	113.50	183.52		68	N/A
ES208	178.30	260.08		23	0.79
GW249	361.00	683.73	10	68	0.87
GW242	350.00	662.93	12	59	0.87
GW376	544.50	1028.74		22	0.91
GW407	554.30	897.03		20	0.92
GW296	384.20	539.90		35	0.90
GW303	393.40	552.67		26	0.90
GW609	762.00	925.68		14	0.95
GW365	450.20	527.20		21	0.92
GW376	463.60	542.94		N/A	N/A
GW349	422.80	451.30		32	0.92
GW355	429.60	458.66		22	0.92
GR488	713.70	1230.77		46	0.91
GR467	682.40	1176.84		36	0.92
GR535	783.70	1348.20		20	0.93
GR882	1213.20	1790.46		14	0.96
GR603	789.50	1013.04		19	0.94
GR610	798.60	1024.80		22	0.94
GR1041	1312.30	1457.40		13	0.97
GR710	882.10	944.83		20	0.95
GR661	821.10	879.53		23	0.95
GR786	957.60	935.46		27	0.96
GR777	947.50	925.58		25	0.96
GH479	705.40	1810.62		24	0.91
GH481	708.30	1818.18		24	0.91
GH320	472.40	1209.60	59	72	0.87
GH423	585.70	1288.04		19	0.90

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Formulation	Endothermic contribution (MJ.m ⁻³)	Exothermic contribution (MJ.m ⁻³)	TTI (s)	pHRR (kW.m ⁻²)	Residual fraction (after 20 min)
GH650	856.80	1639.76		26	0.94
GH634	835.80	1599.44		34	0.94
GH649	822.30	1362.90		20	0.95
GH743	927.10	1482.48		20	0.95
GH743	927.80	1483.68		22	0.95
GH775	948.70	1384.27		17	0.95
GH772	945.20	1379.09		21	0.96
G25E75H1333	873.20	419.90		4	0.99
G50E50H1453	1140.30	457.70		5	0.99
G75E25H1445	1321.50	455.18		5	0.98
GH1328	1387.20	418.32		3	0.98
GS225	313.70	421.76		23	0.85
GS248	345.40	464.26		25	0.87
GS214	298.70	400.61		25	0.81
GS229	301.20	345.33		23	0.87
GS227	298.50	342.32		21	0.86
GS140	185.10	212.18		26	0.77
GS136	180.00	206.29		24	0.76
GS301	380.80	376.15		24	0.90
GS285	360.60	356.18		19	0.90
GS368	444.90	364.47		24	0.91
GS406	489.80	401.13		20	0.92
GS435	516.40	384.89		23	0.93
GS431	512.20	381.80		34	0.94
EGW273	348.10	748.57		26	0.89
EGW257	327.00	703.15		22	0.89
EGW372	433.50	819.89		55	0.92
EGW373	434.70	822.09		59	0.92
EGW459	503.10	838.68		17	0.94
EGW476	520.80	868.22		21	0.94
EGW495	502.80	715.50		28	0.95
EGW527	535.10	761.42		48	0.95
EGW495	503.10	715.79		19	0.95
EGW498	506.00	719.98		20	0.95
EGW539	532.60	697.03		32	0.96
EGW556	549.20	718.74		22	0.96
EGW555	548.30	717.71		18	0.95
EGH454	591.00	1717.63		25	0.91
EGH415	539.70	1568.70		22	0.91
G25E75H179	218.30	676.62	11	132	0.75
G50E50H524	331.40	960.12	25	78	0.83
G75E25H184	255.90	695.52	13	123	0.77
EGH592	704.30	1805.38		22	0.93
EGH627	744.90	1909.52		21	0.93
EGH685	762.40	1726.45		22	0.94
EGH722	804.40	1821.71		23	0.94
EGH743	765.40	1483.48		23	0.96
EGH781	804.70	1559.69		24	0.96
EGH858	858.20	1531.53		19	0.96
EGH805	805.80	1438.00		22	0.96
LH449	466.4	1367.7	100	32	0.92
LH450	468.1	1372.5	76	43	0.92
LH265	275.2	806.9		32	0.86
LH466	459.1	1273.0		54	0.93
LH436	429.5	1191.0	140	29	0.92
LH524	488	1266.4		26	0.93
LH498	464.1	1204.3		45	0.93
LH517	444.3	1032.1		32	0.95
LH488	419.9	975.4		41	0.94
LH699	550.8	1102.3		44	0.96
LH629	495.5	991.6		39	0.96
LH757	569.2	1034.4		19	0.97
LH758	569.5	1035.1		21	0.97

Appendix C. (Abbreviation List)

Abbreviation	Meaning
ASTM	American society for testing and materials
c_p	Specific heat capacity
Cpmod	Modified specific heat capacity
FAA	Federal aviation administration

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Abbreviation	Meaning
FIGRA	Fire growth rate index
GHC	Gross heat of combustion
h_c	Combustion energy
H_g	Thermal decomposition to volatile fuel
HRR	Heat release rate
ISO	International organization for standardization
k	Thermal conductivity
MDF	Medium density fibreboard
m_f^r	Fuel mass flux
PCFC	Pyrolysis combustion flow calorimetry
pHRR	Peak of heat release rate
RFPC	Réseau français de la construction paille
RH	Relative humidity
SBI	Single burning item
THR	Total heat release
TpHRR	Temperature at the peak of heat release rate
TTI	Time to ignition
ρ	Apparent density
μ	Bioresource residue

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