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Fire behaviour of hemp, clay and gypsum-based light biobased concretes and renders

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ABSTRACT

Greenhouse gas emissions from cement manufacturing account for about 8 to 10 percent of total CO₂ emissions worldwide. To reduce these emissions, researchers are developing new concrete manufacturing techniques and processes to reduce high energy consumption and environmental impacts. Biobased concretes are eco-friendly insulating materials for building industry that can respond to this problem. However, there is a lack of knowledge regarding how these plant-based fillers ignite and contribute to heat release in case of fire. In this work, the fire behaviour of a series of hemp-based earth and/or gypsum concretes covering a large range of densities (180–1500 kg/m³) is investigated using the cone calorimeter at an incident heat flux of 50 kW/m². The fire performances are mainly monitored by the thermal inertia of the materials. Only the lightest concretes ignite with a density threshold for ignition occurrence around 500 kg/m³. For a density of 261 kg/m³, the critical heat flux of an earth-hemp concrete was found to be close to 27 kW/m². The flaming period remains very short in all cases.

Keywords:

Biobased concretes

Raw earth

Gypsum

Hemp building materials

Cone calorimeter

Fire behavior

1. Introduction

Due to environmental concern, professional building workers, researchers and companies are currently devoting great efforts to reduce the environmental impact of building materials. For this purpose, using bio or georesources rather than fossil resources is part of the solution. A complementary way is to reduce the energy to heat or cool buildings during their service life by using insulating materials.

Raw earth offers a set of advantages, including its local availability, the capability to fully reuse the material after a building end-of-life, and its low embodied energy, in comparison to other binders like cement or lime [1–3]. Raw earth is a traditional material which can be used according to a variety of processes for various structural and non-structural applications in modern buildings [4]. Of course, the choice of raw earth is of first importance [5–7], and the variability of obtained properties requires a special attention [8]. Several studies investigated

how raw earth can be stabilized by cement, lime or gypsum [9]. Global warming impact of these binders depend on several parameters such as the energy used or nature of cement and lime.

At the production stage, gypsum is about 6 times less impacting than CEM I cement and 5 times less impacting than lime in terms of global warming [10–12]. Gypsum bring several advantages: a very fast setting kinetic and likely a more protective fire behaviour than clay-based materials since gypsum is used for a long time as protective panels against fire [13]. Moreover, its variability is easier to control than for clay. Gypsum binder can be used alone with plant particles, or it can be associated with clay in a given proportion.

Many works have already been devoted to assess the properties of plant-based concretes [14–15], including mechanical properties [16–17], heat conductivity [16], hygrothermal properties [3,17], acoustic properties [18–19] or environmental impact [20–21]. Some papers have focused more particularly on earth-hemp [19,22–23] or

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gypsum-hemp concretes [24–26].

Despite their promising future for building industry, there is still a lack of data about the association of mineral binders with plant particles, especially about their reaction-to-fire and their fire resistance. Several trade documents or scientific papers have presented some results, which are generally quite good. Lanos reported several examples of fire resistance tests on biobased materials (based on hemp or straw) [27]. Volhard studied two earth-straw concretes with different densities and concluded that these concretes exhibit a good fire resistance [28]. Regarding the reaction-to-fire (i.e. the direct contribution to heat release), Lanos reported SBI (Single Burning Item) rating of various biobased materials, including some earth-hemp concretes [27]. Most of them are rated A2 or B, but some F ratings are also reported without details. Note that the additional rankings concerning smoke and dripping are always at the best level (i.e. s1, d0).

Nevertheless, each report focused on one specific product (or a limited range of products), which does not allow to extrapolate results when the density, the binder or plant nature are different than the ones tested. To the best of our knowledge, no comprehensive study has been published in open literature about the reaction-to-fire of an extended series of biobased concretes. The present study aims to provide an overview of the flammability of these materials in order to estimate the fire hazard from a couple of parameters as the concrete density, the fiber content or the nature of the binder. Around 20 concretes based on hemp and different binders were prepared covering a large range of density (180–1500 kg/m³). Their flammability was assessed using a well-known bench-scale test, namely cone calorimeter.

2. Materials and methods

The plant particles used in this study are hemp shiv. Hemp shiv comes from Poitou Chanvre production. Shiv length and width are 7.6 (±1.3) and 2.0 (±0.4) mm respectively.

Earth is a fine natural kaolin (trademark: Blankalite 78 from Soka – France) with a true density of 2600 kg/m³. 99% of particles have <20

µm-size. Earth 2 was collected from earthworks performed close to Nocé (61340, Normandy, France). It was used in more than 30 hemp-clay operations. A granulometry, sedimentometry, and methylene blue value characterization showed it has a high fraction (37%) of normally active clay. Gypsum was a 0/200 µm natural hemihydrate gypsum plaster (trademark: neige 1R from Plâtres Vieujeot/Platre.com France).

Sand was a siliceous 0/4 mm sand coming from sablières de la Seine in Bernières sur Seine (27700). Exfoliated vermiculite with a density ranging from 65 to 160 kg/m³ comes from Nestaan NV (Belgium).

Fig. 1 shows the different constituents used for preparing biobased concretes.

Samples were prepared as follows:

- Plant particles and binder were weighted to produce 6 to 7 samples for each formulation. Each component weight varied from 500 g to 5 kg depending on the recipe followed. Weighing was done with a ± 0.1 g balance.
- Materials were first mixed dried. Then water was added in adequate proportion to obtain a good workability, based on the expertise of craftsmen that produced the samples. Water to binder ratio depends on the water absorption of plant particles, on the binder water demand and on the recipe. Water to binder ratio varied from 1 to 1.5 for clay (i.e. earth) based samples and from 0.4 to 1.5 for gypsum-based samples.
- A first 10 × 10 × 10 cm³ mould was completely filled by a moderate manually compaction. The mass introduced in the mould was recorded.
- The other moulds with the same mix were filled with the same mass of material, to obtain repeatable samples;

Cardboard moulds were used as it was convenient to produce a large number of samples (Fig. 2). They were used for manufacturing operations by adding a rigid plastic sheet between mixture and cardboard to ensure clean surface conditions. 1 day after manufacturing, mould and rigid plastic sheets were removed so the sample could dry. When



Fig. 1. Constituents of biobased concretes.



Fig. 2. Packed samples (at the bottom) and unpacked samples (at the top-left).

samples were stabilized in mass in room condition, they were packed again in cardboard moulds and sent to the laboratory.

After preparation, concretes were stored in cone calorimeter room up to mass stabilization (weight change over 24 h < 1%). This occurs after several weeks. The atmosphere of the room was not controlled and relative humidity varied in the range 20–35 %. The influence of moisture is discussed below.

Concretes are called as XYZ with X referring to the binder (E for the main type of earth used in this study, E2 for the second type of earth, G for gypsum and GE for a gypsum-earth mix), Y to the filler (H for hemp and V for vermiculite) and Z to the density. For example, GH423 is the concrete based on gypsum and hemp and presenting a density of 423 kg/m³. When the binder was a mix of earth and gypsum, the concretes were called GaE(100-a)HZ with “a” the weight content of gypsum in binder. For example, G75E25H184 refers to a concrete containing a binder based on 75 wt% of gypsum and 25 wt% of earth and presenting a density of 184 kg/m³. In some concretes, especially the densest ones, sand was also added. Table 1 lists all the concretes prepared with the water content used during the preparation.

Density was calculated considering a volume of 10x10x10 cm³. Nevertheless, for some samples, shrinkage occurs during curing and leads to a slight reduction in volume. This reduction was assessed

Table 1
List of concretes prepared and their composition.

	Earth (wt%)	Gypsum (wt%)	Sand (wt%)	Water (wt%)	Hemp (w%)	Hemp after curing (w%)
EH261	33	0	0	50	18	35
EH430	27	0	27	32	14	20
EH512	42	0	0	50	8	15
EH965	16	0	63	17	5	5
EH1297	16	0	64	17	3	3
GH320	0	32	0	50	18	33
GH423	0	42	0	41	17	26
GH649	0	44	0	44	11	18
GH1070	0	63	0	33	4	5
GH1328	0	69	0	29	2	3
G25E75H179	18	6	0	62	14	35
G50E50H254	20	20	0	39	22	34
G75E25H184	6	18	0	62	14	33
G25E75H1333	20	7	52	19	3	3
G50E50H1445	13	13	53	18	3	3
G75E25H1453	7	20	52	19	3	3
E2H259	33	0	0	50	18	35
EV631	37	0	0	43	20*	35*

* vermiculite (not hemp).

around 9%. Therefore, the reported densities must be considered as minimum values. Density was calculated after mass stabilization in the conditions already indicated above.

Hemp flammability was tested at microscale using pyrolysis-combustion flow calorimetry (PCFC - FTT, East Grinstead, UK) according to method A (anaerobic pyrolysis) and method B (aerobic pyrolysis) of ASTM D7309 standard. A few mg-sample was heated at 1 K/s up to 750 °C under nitrogen or air. Gases released are sent to a combustor at 900 °C in an excess of oxygen, ensuring a complete combustion. Heat release rate curves are plotted versus pyrolysis temperature. HRR is calculated using oxygen depletion method. Indeed, 1 kg of oxygen consumed corresponds to 13.1 MJ of energy released according to empirical Huggett’s relation [29].

Cone calorimeter (FTT, East Grinstead, UK) tests were usually performed at 50 kW/m² using piloted ignition for 20 min according to ISO 5660 standard [30]. Some additional tests were performed at different heat fluxes or without spark igniter. The 10x10x10 cm³ samples were directly put on the sample-holder without rockwool or aluminium foil. A frame was used to ensure that heat flux was only absorbed by the surface sample (and not by the side faces) (Fig. 3). When a balance was used during test, mass was recorded each 30 s. Usually two tests were performed for each concrete. Statistical analysis was carried out on concrete EH261. The char front was measured at the end of the test by measuring the black layer thickness using a ruler on the side faces. Usually, the thickness was similar on the four sides. When the thickness was significantly higher on one side, only the thickness on the other sides was considered. In cone calorimeter test, heat front moves through the thickness. When it reaches the sample bottom, the temperature inside the sample increases because heat cannot be removed. This temperature increase accelerates the decomposition. 10 cm-thick samples ensure that they can be considered as semi-infinite. In other words, the heat front does not reach the bottom of the samples during the fire test.

For some tests, the distance between radiant cone and the upper surface was increased to 60 mm (versus 25 mm in standard test) to measure the surface temperature using an infrared camera (Optris CT). The mean temperature of the zone at the center of the upper surface was measured on the range 125–900 °C. Emissivity was considered to be equal to 1 which is reasonable for such rough surface materials and for an approximative surface temperature measurement.

Single Burning Item (SBI) simulations were performed using Con-eTools software (FTT, East Grinstead, UK) from the cone calorimeter experiments at 50 kW/m². TTI in SBI simulations is an input data. It was taken to be equal to the value measured in cone calorimeter or 30 s maximum (when no ignition was measured).

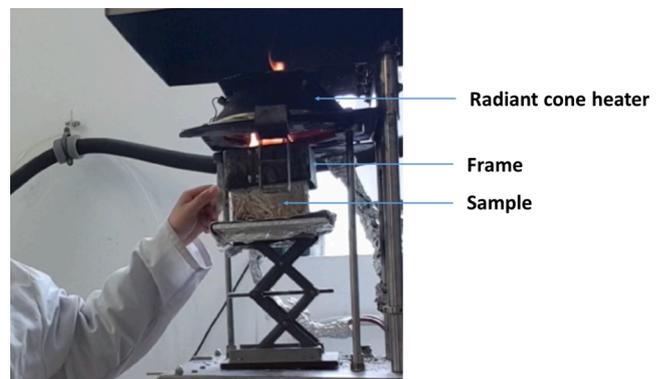


Fig. 3. Side view of the set-up in cone calorimeter test.

3. Results

3.1. Hemp flammability at the microscale

The flammability of hemp was analyzed at the microscale using the pyrolysis combustion flow calorimetry. Pyrolysis is mainly anaerobic during a fire, as long as oxygen is consumed in the flame. When the flame vanished, thermo-oxidation, i.e. aerobic pyrolysis, occurs. The flaming period in cone calorimeter is limited for the biobased concretes (see below), however, both conditions were tested using the pyrolysis combustion flow calorimetry.

The results in Fig. 4 show that HRR curve has only one peak in anaerobic pyrolysis. This peak is around 106 W/g and centered at 335 °C. This is in good agreement with previous works [31–32]. Total heat release is close to 10 kJ/g (a little bit higher than the previously reported values) and residual fraction (i.e. the ratio between the final mass and the initial mass) is 0.19. Heat of complete combustion is 12.8 kJ/g. Even if hemp is mainly composed by hemicellulose, cellulose and lignin, the decomposition steps of these three components merge, resulting in only one peak.

Under aerobic pyrolysis, two peaks are observed. The first one, 170 W/g at 288 °C, results from the decomposition of the main components which occurs earlier in presence of oxygen. The second one, 181 W/g at 383 °C, is due to the thermo-oxidation of the char. Consequently, no significant char is left and THR is much higher (16.4 kJ/g) as well as heat of complete combustion (16.8 kJ/g).

Considering these last values, it is possible to calculate the theoretical maximum heat released during the cone calorimeter test. Since the hemp content never exceeds 35 wt% in the samples studied, thus the heat release cannot be higher than 5.9 kJ per gram of concrete. When the hemp content is 5 wt%, this value is even much lower (<1 kJ/g). Since the piloted ignition is based on the gas phase combustion energy density [33], it is clear that such samples would hardly ignite. Comparatively, these heat release values are very low: recent results showed that flame out in cone calorimeter occurs in few seconds for an alginate-based foam with a density of 40 kg/m³ (lower than the current biobased concretes) and exhibiting a heat of combustion of 3 kJ/g [34]. Common polymers including those used as insulating materials exhibit much higher heat of combustion (20–40 kJ/g).

3.2. Density of biobased concretes

The density of biobased concretes is mainly monitored by adjusting the content of biofillers (here, hemp) and changing the compaction during casting (compaction level was not studied here, all sample were manually compacted in mould). Therefore, biofillers content and density are in close relation (see Fig. 5). Hemp content is calculated on a dry basis, i.e. by considering only the amount of hemp, binder (earth or

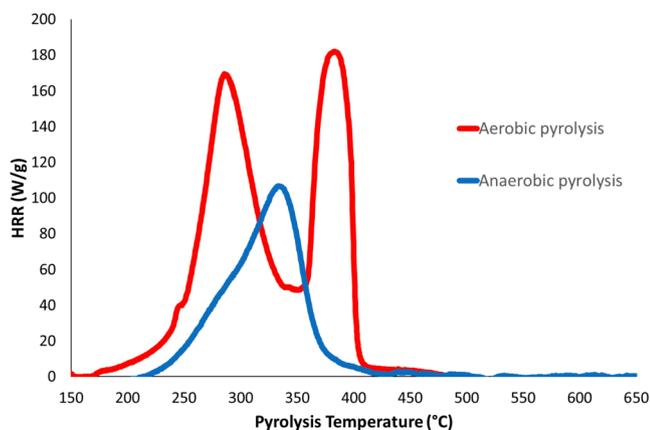


Fig. 4. HRR curves of hemp in PCFC (aerobic and anaerobic pyrolysis).

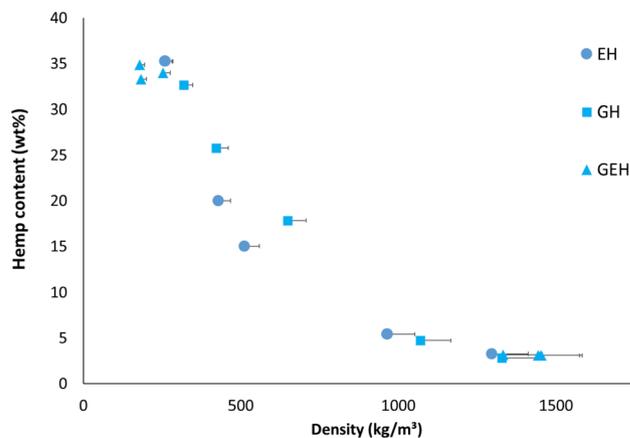


Fig. 5. Hemp content versus density in casted hemp biobased concretes after mass stabilization.

gypsum) and sand. However, for gypsum-based concretes, the gypsum is hydrated during curing (it changes from semi-hydrate to dihydrate form) and water consumed by this process is considered. In these concretes, sand is also often added to limit the shrinkage during curing, similarly to professional building workers practice for rendering. Densities in the range 180–600 kg/m³ are reached for hemp content between 15 and 35 wt%.

Several parameters may control the flammability. High-density concretes need more time to be heated up to pyrolysis temperature: indeed, the energy absorbed to increase the temperature from ambient to pyrolysis temperature is proportional to the product of density and specific heat. Moreover, higher density usually corresponds to higher heat conductivity. A high conductivity allows a higher heat transfer from the surface to the bulk. A second parameter is the hemp concentration. Indeed, ignition occurs when the amount of fuels in gas phase reaches a threshold concentration. When the hemp concentration is too low, this threshold may not be reached. Fig. 6 plots the hemp concentration (i.e. the hemp density in concrete, in kg/m³) versus the density. For low-density concretes, both parameters appear to be relatively independent. For density ranging from 250 to 500 kg/m³, the hemp concentration may vary roughly from 75 to 110 kg/m³. Its value is lower than 50 kg/m³ for the densest concretes. The reason is that the compaction may change the density at a same hemp content. Moreover, the binders (including sand in some formulations) can have different densities.

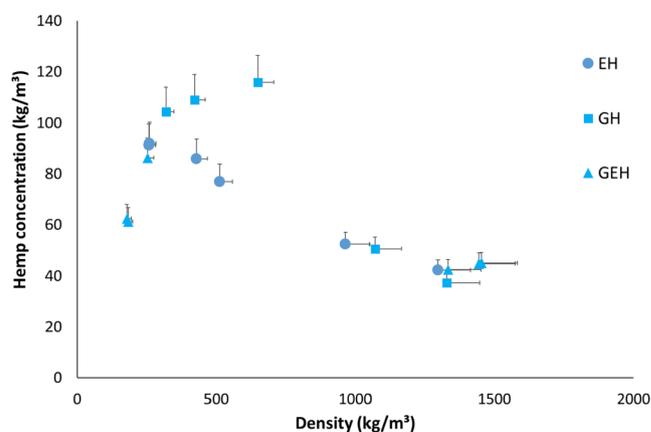


Fig. 6. Hemp concentration versus density in biobased concretes after mass stabilization.

3.3. Flammability of biobased concretes at bench scale – Preliminary remarks

This work is focused on the ignition, the peak of heat release rate and the mass loss during cone calorimeter test. Smoke production is another important aspect of the fire behaviour. But it was found to be negligible or very low for these materials (in open as well as in close conditions, i.e. in smoke chamber – Figure S1 in supporting information).

Typical heat release rate (HRR) and mass loss curves are shown in Fig. 7. Fig. 7A presents the curves for ignited and non-ignited concretes. For ignited concretes, ignition occurs in <30 s and is followed by a narrow peak of heat release rate. This peak varies depending on density but does not exceed 130 kW/m². It is followed by flame out after only few dozens of seconds. In other words, the flame is not sustained. The HRR curve does not go down to 0 due to the thermo-oxidation of hemp after flame out. The plateau remains lower than 20 kW/m². Nevertheless, the measurement of such low HRR values is not accurate and cannot be used for correct heat release calculations. Indeed, even EV631 containing no fuel exhibits a low but non-negligible heat release rate (noisy curve).

For non-ignited hemp-based concretes, there is no pHRR but the curve stabilizes to a constant HRR value (still lower than 20 kW/m²). Higher is the density, lower is the plateau. For the densest concretes, the HRR remains close to 0 during the whole test.

Fig. 7B shows the mass loss (in grams) during the test. The mass loss increases linearly during the whole test, even for ignited concretes where a short flaming period occurs at the beginning. The mass loss reaches around 45–50 g for the lightest concretes. Its value is still 20–25 g for the densest one. Obviously, the relative mass loss (i.e. in wt%) is much lower for the dense concretes. The mass loss is related to the hemp decomposition, the release of water (either free or constitutive) and the decomposition of gypsum. The mass loss for EV631 is low but non-

negligible (around 15 g due to water release from earth and vermiculite decomposition).

Water absorption in a biobased concrete depends on its composition. According to Colinart et al. [22], water content of hemp-earth concrete (density 196 kg/m³) after drying at 105 °C increases from 2 to 5% (4–10 g) when relative humidity increases from 20% to 50%. For densest concretes, hemp content and then water content (in percentage) is lower. Even if these values are limited, they are not negligible and should be accounted so the mass loss is not fully assigned to hemp decomposition.

The overall mass loss is an indicator but it hides a more complex reality. Indeed, water release, gypsum and hemp decomposition occur at different temperatures. As there is a heat gradient through the thickness of the samples, the front of hemp pyrolysis does not coincide with the front of water release or the front of gypsum decomposition.

The role of water content in flammability must be considered, especially because the relative humidity (RH) has varied in the storage room (in a moderate RH range 20–35%) during the study. Some additional experiments were carried out in Supporting Information S2. Results confirm that the water absorption in the studied range has no significant influence on flammability.

Eight tests were performed on EH261 at different periods of the study to ensure the reliability of measurements. Table 2 shows the mean, minimum and maximum values of TTI, pHRR and residual fraction as well as the standard deviations. Data for pHRR and residual fraction vary in a narrow range. On the contrary, data for time-to-ignition are more scattered even if this concrete ignites quickly in all cases. This observation is also valid for other concretes. Especially, few of them do not ignite in all cases. For example, PH320 ignites only one time over two tests. Of course, the occurrence of ignition has a great effect on pHRR but not on residual fraction as explained below. Two reasons for such variability may be proposed. First, some of these concretes are close to the flammability limit, i.e. their amount of fuels is just high enough to ignite. Second, the observation of the sample surface shows that some hemp fibres are apparent and not fully covered by the binder, especially for lighter concretes. Such free plant particles may promote ignition. This point will be investigated in the future. These variabilities confirmed our choice to test a large number of samples to reach correct findings when testing these kinds of materials with a cone calorimeter.

3.4. Influence of heat flux and thermal behavior

EH261 was chosen to study the influence of heat flux. While this material is one of the most flammable ones, it may be considered as a worse case from fire hazard point of view. Fig. 8 shows the HRR curves for EH261 at different heat fluxes. Two curves are shown for heat flux of 30 kW/m². At this heat flux, only one of both tests led to ignition. As usual, time-to-ignition decreases when heat flux increases (69 s at 30 kW/m² and <10 s at 70 kW/m²). Peak of heat release rate increases but the change is relatively limited (from around 60 kW/m² at heat flux of 30 kW/m² to 110 kW/m² at 70 kW/m²). The peak is much lower when no ignition occurs (<20 kW/m²).

Residual fraction also decreases when heat flux increases, from 0.90 at 30 kW/m² to 0.81 at 70 kW/m². Residual fraction is similar for both tests at 30 kW/m², confirming that the short flaming period has no impact on mass loss. The char front also increases: 1.5–2 cm at 30 kW/m² to 4.5 cm at 70 kW/m².

Materials can be divided into two categories according to their

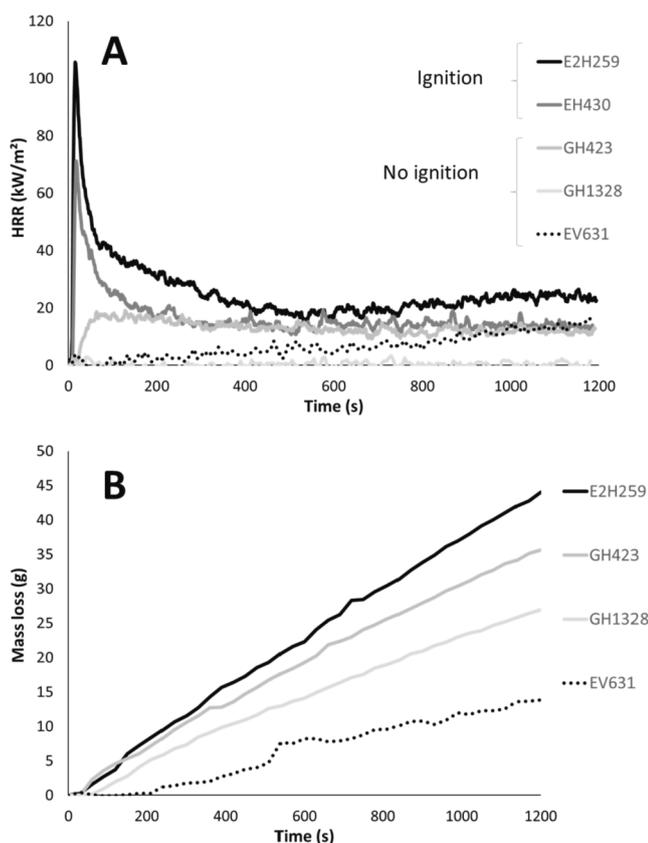


Fig. 7. HRR curves (A) and mass loss curves (B) for several concretes in cone calorimeter tests at 50 kW/m².

Table 2

Mean values of flammability and standard deviations for EH261 (minimum and maximum values are done in brackets).

	TTI (s)	pHRR (kW/m ²)	Residual fraction
Mean value	17 (6–23)	91 (82–103)	0.85 (0.83–0.87)
Standard deviation	6	7	0.02

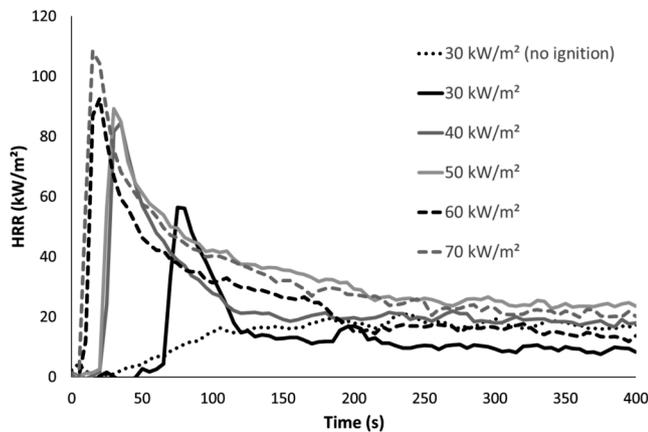


Fig. 8. HRR curves of EH261 at different heat fluxes.

thermal behaviour [35]. In thermally-thick materials, a heat gradient is observed through the sample thickness. Heat conductivity allows transferring heat from the surface to the bulk. A linear relation between the reciprocal of time-to-ignition square root and heat flux is observed in this case (Equation (1)). The time-to-ignition does not depend anymore on the thickness. On the contrary, for thermally thin materials, the whole volume is heated at the same temperature and burns at the same time. Heat conductivity does not influence the time-to-ignition. The reciprocal of time-to-ignition linearly changes with heat flux (Equation (2)).

$$TTI = \frac{\pi}{4} k \rho c \left[\frac{T_{ig} - T_o}{\varepsilon \times HF - CHF} \right]^2 \quad (1)$$

$$TTI = l \rho c \frac{T_{ig} - T_o}{\varepsilon \times HF - CHF} \quad (2)$$

with k the heat conductivity, c the specific heat, ρ the density, l the thickness, ε the emissivity, HF the heat flux, T_{ig} the temperature at ignition and T_o the room temperature.

Drawing the reciprocal of \sqrt{TTI} versus heat flux is a suitable way to identify the thermal behaviour of a material. Moreover, extrapolating these curves to 0 allows calculating the critical heat flux (CHF), i.e. the minimum heat flux for ignition. Nevertheless, in practice, both curves are quite often linear and it is hardly possible to conclude about the thermal behaviour. Another approach is to compare calculated CHF to experimental ones.

Considering Equation (1) (i.e. a thermally thick behaviour), CHF was 7 kW/m^2 . On the contrary, Equation (2) (thermally thin behaviour), CHF was found to be 27 kW/m^2 (Fig. 9). From the tests described above,

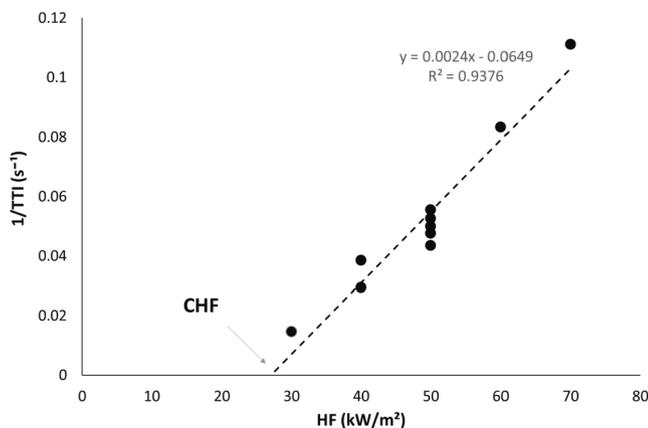


Fig. 9. TTI^{-1} versus heat flux for EH261.

it is clear that CHF should be close to 30 kW/m^2 . This value is quite high in comparison to many solid fuels as polymers (except halogenated or thermostable ones) [33]. It is noteworthy that this value is probably a minimum value for biobased concretes, because most of them do not ignite even at 50 kW/m^2 .

This result also means that the light sample tested can be considered as a thermally thin one, despite its high thickness (10 cm). In fact, for insulating materials, heat conductivity is low and cannot contribute efficiently to cool down the surface by transferring heat to the bulk. Therefore, TTI on such materials depends on the thickness directly absorbing the heat flux from the cone, according to Equation (2) [34]. This thickness may be assessed if thermophysical properties are known. Heat conductivity was measured close to 0.08 W/m.K . This value agrees with values reported by Labat et al. and Colinart et al. on biobased concretes of similar density [22,36]. Specific heat and emissivity were chosen equal to 1.5 kJ/kg.K and 1 respectively. A temperature at ignition of 450 $^{\circ}\text{C}$ was chosen (see below about the measurements of surface temperature). The best agreement between calculated and experimental TTI is found for a thickness of 2.5 mm (see Fig. 10). TTI calculations with Equation (1) are also shown for comparison purpose. Such thickness appears quite reasonable. For a lightest alginate foam (40 kg/m^3), the critical thickness above which TTI is constant was found to be around 1.5 cm [34]. Calculated TTI is not in agreement with experimental values only for the lowest heat flux, but this heat flux is very close to the CHF. Moreover, ignition was not observed for one test among two.

3.5. Influence of density on flammability

First, we evaluated if hemp concentration is the main parameter driving the flammability. Figure S3 in Supporting Information shows no clear correlation between reciprocal of time-to-ignition (TTI^{-1}) and hemp concentration. Low ($<50 \text{ kg/m}^3$) as well as high (greater than 110 kg/m^3) hemp concentrations do not lead to ignition. Ignition occurs for intermediate hemp concentrations. Therefore, the discussion focuses on the role of density on flammability.

On the other hand, Fig. 11 shows a clear correlation between the time-to-ignition and the density for all materials studied. The densest materials do not ignite and thus TTI^{-1} can be considered to be null. On the contrary the lightest materials ignite. A rough threshold of density can be proposed: above 500 kg/m^3 , no biobased concrete ignites. Ignition occurs for almost all concretes with a density lower than 500 kg/m^3 (except GH423). The lightest concretes exhibit a lower TTI. However, the relation between time-to-ignition and density for ignited materials is rough due to the uncertainties already discussed. Nevertheless, in all cases, time-to-ignition varies in a narrow range (6–29 s).

Flame out occurs after only few dozens of seconds. The burning time does never exceed 60 s. It is the reason why the mass loss rate does not seem to increase during the flaming period (remind that the mass loss was measured each 30 s). Moreover, during the most part of the test (at least 95%, i.e. 19 min over 20 min), pyrolysis can be considered as aerobic while oxygen is not consumed in the flame.

It can be noted that most of concretes undergoing ignition are based on earth or a mix of earth and gypsum. However, it is not possible to conclude about the role of gypsum to prevent ignition since there is only one gypsum-based concrete exhibiting a low density (GH320). Nevertheless, it is noteworthy that it exhibits the highest TTI (29 s) among the lightest concretes (and only one sample was ignited). Further work is needed to assess properly the role of gypsum.

Right after ignition, the heat flux absorbed by the material is increased by the heat flux from the flame. During several dozens of seconds, the hemp pyrolysis rate increases and pHRR is much higher than for non-ignited concretes as shown in Fig. 12. The pHRR of non-ignited materials does not exceed 20 kW/m^2 and tends to continuously decrease when density increases. This heat release is partly due to the pyrolysis of biofillers but also to the measurement uncertainties. Indeed, the HRR curve is noisy with positive values even for EV concrete

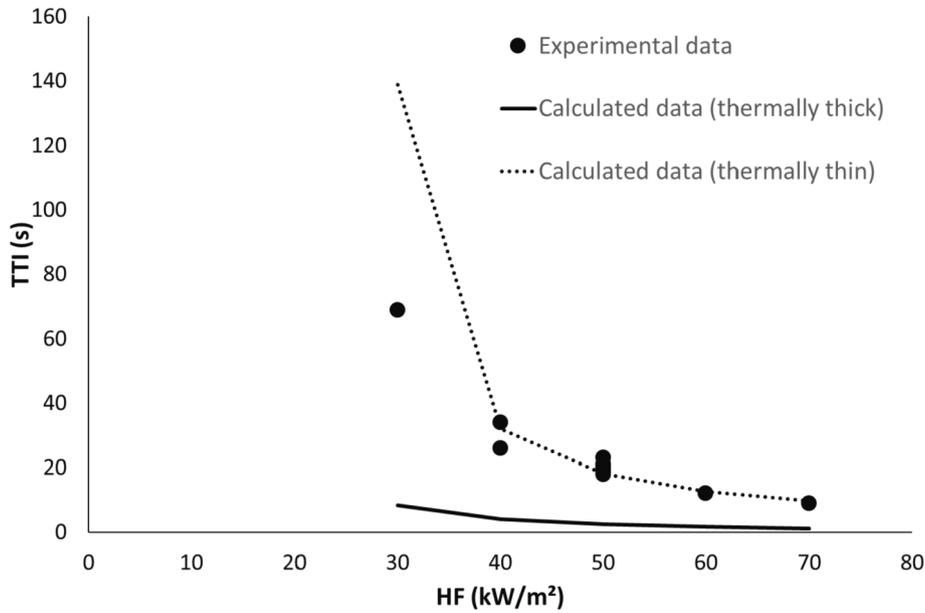


Fig. 10. Experimental and calculated TTI versus heat flux for EH261 (for thermally-thin model, thickness was fixed to 2.5 mm).

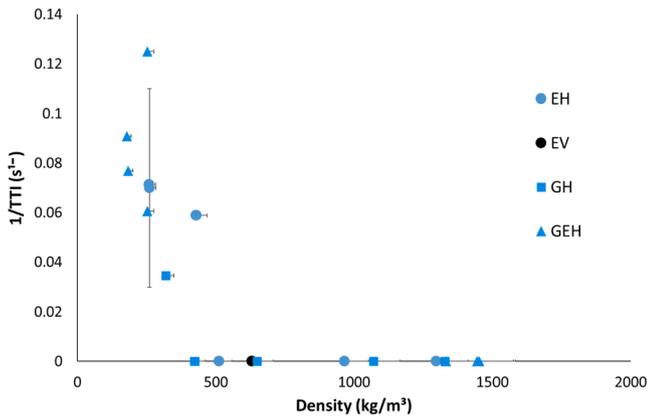


Fig. 11. Reciprocal of the time-to-ignition versus density.

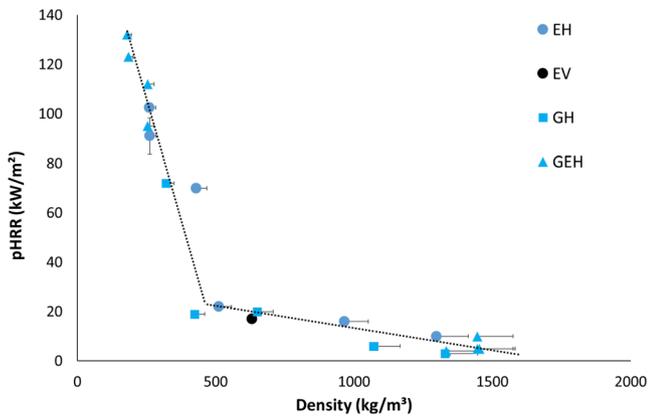


Fig. 12. pHRR versus density.

which does not contain any fuel. The comparison between GH423 and EH430 highlights the influence of ignition on pHRR. While GH423 does not ignite, EH 430 exhibits a time-to-ignition of 22 s. Its pHRR is 69 kW/m² versus lower than 20 kW/m² for GH423.

The peak of heat release rate increases when density decreases,

especially for concretes which ignite. In other words, the mass loss rate reaches maximum values for the lightest concretes. However, these concretes do not contain the highest hemp concentration (see Fig. 6). Then, it may be assumed that the pyrolysis zone reaches a higher thickness for these concretes, maybe because the radiation from the radiant cone penetrates deeper. This may be also ascribed to their low heat conductivity. Indeed, the heat absorbed cannot be easily removed and then a thicker layer of concrete reaches a temperature high enough for pyrolysis.

During burning, the mass loss is due to the pyrolysis of hemp, the release of absorbed water and the decomposition of gypsum. As already explained, hemp pyrolysis is aerobic and leads to a complete filler decomposition (no residue). Dihydrate calcium sulphate releases two molecules of water, losing 20.9 wt% of its mass.

Residual fraction is plotted on Fig. 13. It increases with the density and comes from 0.75 for the lightest concretes to 0.98 for the densest ones. Once again, a critical density threshold can be found around 500 kg/m³: above this value, the residual fraction increases slowly with the density. Below it, the decrease in residual fraction is much faster. Note that it is not due to the increase in mass loss rate during flaming period. The mass loss during these few dozens of seconds is not high enough to account for the lower residual fraction of these concretes. See also Supporting Information S4 about this point.

When comparing EH to GH at similar density, residual fraction may

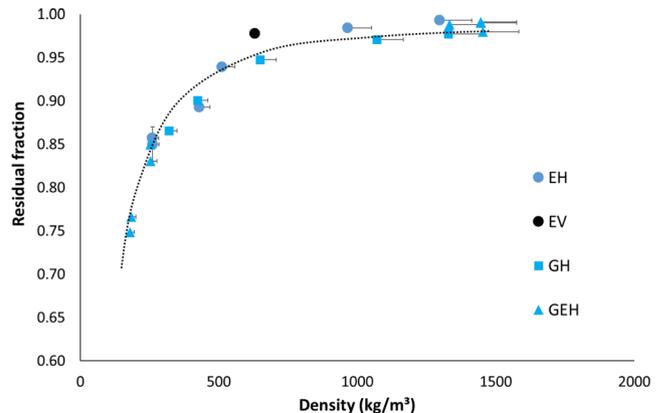


Fig. 13. Residual fraction versus density.

be slightly lower for GH. This may be assigned to the additional water release due to gypsum decomposition. Theoretical maximum mass loss rate can be calculated considering the complete decomposition of hemp and water release from gypsum. Note that two-step dehydration of gypsum occurs at moderate temperature ($<300\text{ }^{\circ}\text{C}$). During the two steps, 1.5 molecule and half a molecule of water are released respectively (i.e. 15.7 and 5.2 wt%) [37]. The release of absorbed water was not considered; therefore, these calculations should be considered carefully as a rough approximation. Fig. 14 plots the ratio between the experimental mass loss and the theoretical one versus density. It clearly appears that gypsum-based concretes have a lower ratio. In other words, the heating front may move more slowly through the thickness of these concretes, leading to lower decomposition of hemp and gypsum.

Fig. 15 shows some pictures of residues from hemp-based concretes after cone calorimeter tests. The lower part of the samples corresponds to the initial aspect of the concretes. Note that the sample E2H259 has a different colour due to the binder (second earth used called E2). Lower is the density, higher is the hemp content, and more apparent are the hemp particles. The upper part corresponds to the char layer formed during the test. Its mechanical strength is negligible, especially for the lightest concretes. When density increases, the char layer thickness decreases from 6 cm to around 0 cm, evidencing that the front char moves faster in the lightest concretes (Fig. 16). However, the measurements are relatively scattered and do not allow to conclude about the effect of binders (earth vs gypsum).

3.6. Influence of binders

Density was found to be the main parameter controlling the flammability. Some results may suggest that the binder has a small influence on reaction-to-fire, especially gypsum may reduce the pyrolysis rate as discussed above (Fig. 14). The comparison of concretes EH261, GH320, G25E75H179, G50E50H254 and G75E25H184 allows assessing the influence of the binders for light concretes with a high amount of hemp. Note that the density of these concretes varies in the range $179\text{--}320\text{ kg/m}^3$. Once again, Fig. 17 shows that pHRR as well as residual fraction are mainly controlled by density and the nature of binder does not seem to be influent. Nevertheless, considering only G25E75H179 and G75E25H184 exhibiting the same hemp content and density, the second one shows a slightly lower pHRR and a higher residual fraction. Especially its mass loss (in % of maximum mass loss) is only 55% versus 66% for the first one.

Further investigations are needed to better assess the role of gypsum on flammability.

A last comment can be done about the influence of the earth nature on flammability. Two earths were used (E and E2) and two concretes with the same density were prepared: EH261 and E2H259. Both exhibit similar performances (time-to-ignition, pHRR, residual fraction). Char

front may be slightly higher for E2H259 but it is assumed that the nature of earth is negligible on the reaction-to-fire.

3.7. Surface temperature at ignition

Ignition occurs when the concentration of fuels in gas phase reaches a threshold concentration. An equivalent but more convenient criterion for ignition is to consider a threshold temperature at sample surface. This temperature is obviously dependent on the material.

Temperature at ignition was measured for some concretes using infrared camera. Note that the distance between cone and sample surface is increased to 60 mm for these experiments. Therefore, even if the heat flux at the surface is still 50 kW/m^2 , the times-to-ignition are slightly different from those reported previously. Fig. 18 shows the surface temperature during cone calorimeter test for some concretes that undergo ignition, i.e. concretes with a low density. Arrows indicate the occurrence of ignition. It can be observed that ignition occurs for a temperature within the range $400\text{--}500\text{ }^{\circ}\text{C}$.

Heating rate depends on the thermophysical properties of a material, namely specific heat, heat conductivity and density. A low density should lead to a higher heating rate and to an earlier ignition. However, there is no clear correlation between the density and the time to reach this temperature. GEH184 is the lightest concrete and it is heating faster. Its time-to-ignition is the lowest. Nevertheless, ignition occurs earlier for EH430 than for EH261. Moreover, GH320 exhibits the lowest heating rate. This may be ascribed to the release of water due to the decomposition of gypsum. This endothermic phenomenon (around 500 J/g [37–38]) lowers the heating rate. The surface temperature increases after ignition and stabilizes at a temperature ranging from $650\text{ }^{\circ}\text{C}$ (for EH184) to $600\text{ }^{\circ}\text{C}$ (for GH320).

The surface temperature evolution during cone calorimeter test is plotted in Fig. 19 for the series of earth-based concretes. The density of these concretes ranges from $261\text{ to }1297\text{ kg/m}^3$. Only the two lightest concretes exhibit ignition. The heating rate as well as the maximum temperature reached decrease when density increases.

Interestingly, EH512 and EH965 reach high surface temperature ($500\text{ }^{\circ}\text{C}$ or more) without igniting. This temperature is higher than the temperature at ignition measured for EH261 and EH430. Moreover, EH512 contains a similar hemp concentration as EH261 and EH430 (77 kg/m^3 versus 92 and 86 kg/m^3). Therefore, the non-flammable behaviour of EH512 cannot be assigned to a too low hemp concentration or a too low temperature surface. The heating rate is probably the main point which explains why ignition occurs for EH261 and EH430 and not for EH512. Indeed, if the heating rate is too low, the decomposition occurs over a longer period. Then, the fuels are released more slowly and the threshold fuel concentration to reach ignition may not be reached. Since the heating rate depends on the product of specific heat and density, this result highlights once again the role of density as the main parameter monitoring the flammability.

3.8. SBI simulations using ConeTools

ConeTools is a software allowing to calculate rating in SBI test from cone calorimeter data. The method was developed and showed a quite good agreement between experimental and calculated ratings from a series of 40 building materials [39–41]. Later, other papers attempted to use ConeTools to predict SBI rating [42]. It is noteworthy that ConeTools allows predicting the rating but is not able to fit accurately the experimental HRR curve in SBI.

SBI rating was calculated for several concretes using ConeTools. Rating depends on FIGRA_{max} (i.e. the maximum value of FIGRA - FIGRA is defined as the growth rate of the burning intensity, and is calculated as the maximum value of the function “heat release rate/elapsed test time” and THR600 (i.e. the total heat release after 600 s). FIGRA_{max} was systematically found to be the most severe criterion. Fig. 20 shows the FIGRA_{max} for some flammable concretes (for denser

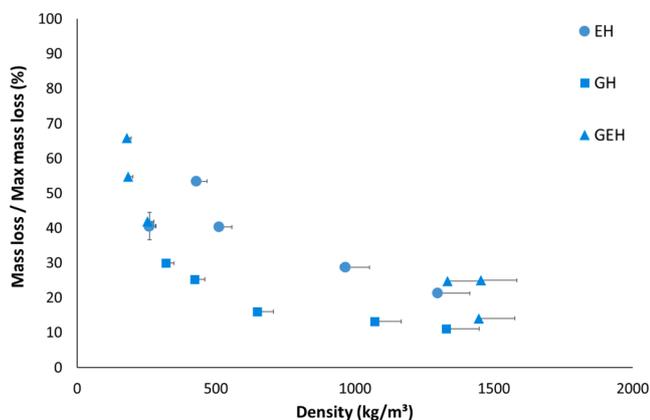


Fig. 14. Mass loss (in % of maximum mass loss) versus density.

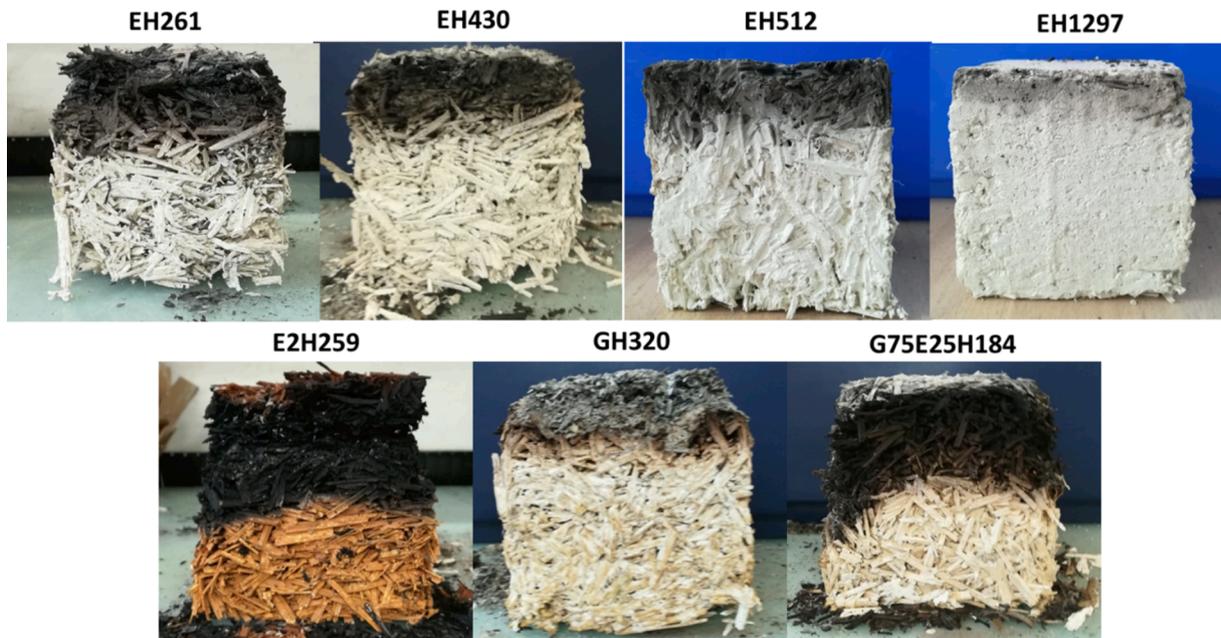


Fig. 15. Some samples after cone calorimeter tests (at 50 kW/m²).

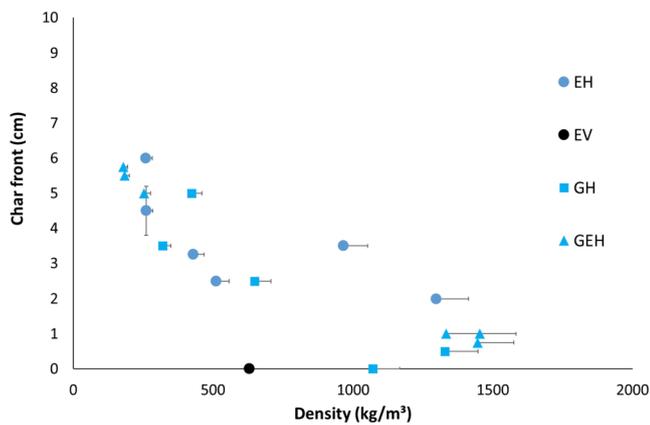


Fig. 16. Char front versus density.

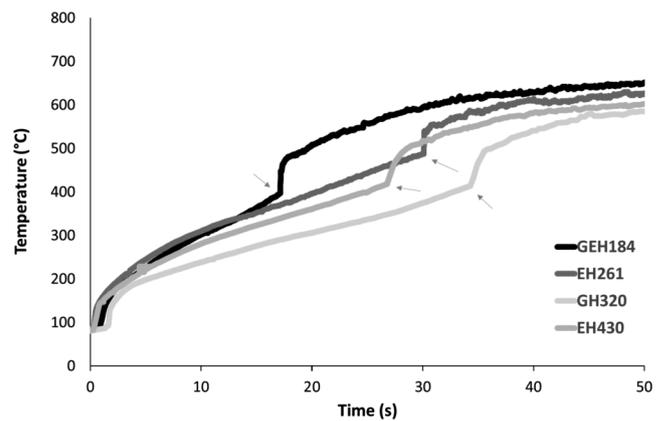


Fig. 18. Temperature at surface during cone calorimeter at 50 kW/m² for several biobased concretes (arrows indicate the occurrence of ignition).

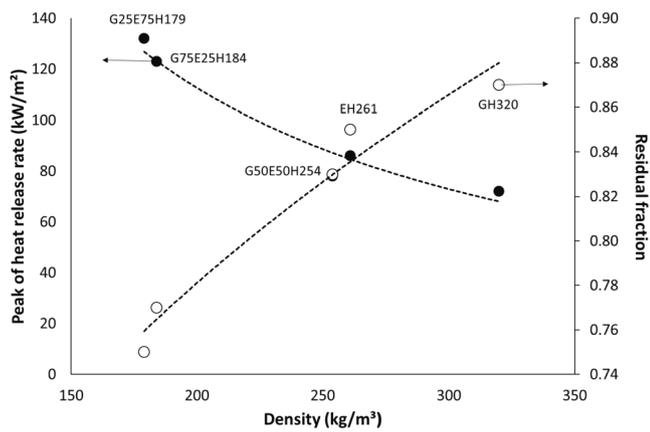


Fig. 17. Peak of HRR and residual fraction versus density for concretes containing a high hemp content and various binders (for GH320, data correspond to the ignited sample).

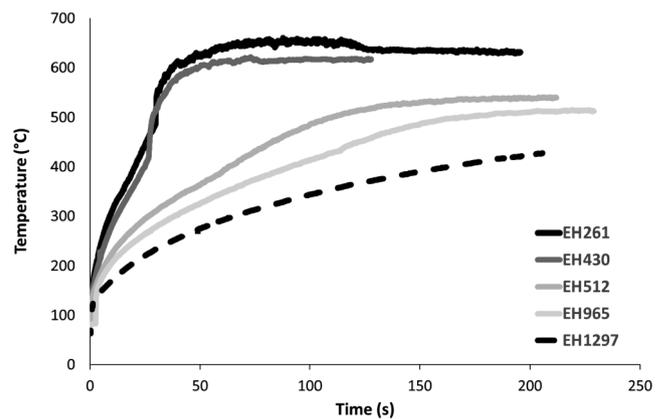


Fig. 19. Temperature at surface during cone calorimeter at 50 kW/m² for earth-based concretes with various densities.

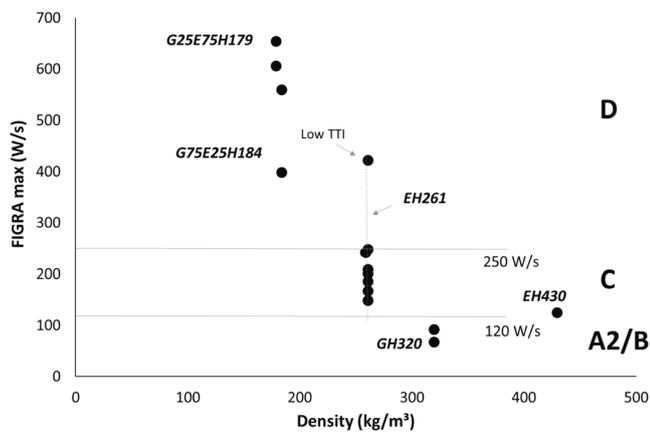


Fig. 20. Calculated FIGRA_{max} versus density.

concretes, rating is systematically found to be A2/B with very low values for FIGRA_{max} and THR600). Rating ranges from D (>250 W/s for the lightest concretes) to A2/B (FIGRA_{max} < 120 W/s for GH320 despite its ignition). For EH261, most cone tests lead to a rating C (FIGRA_{max} < 250 W/s) except one (rating D) due to its low TTI in cone calorimeter test. This result highlights the strong influence of TTI on simulations.

According to these predictions, rating should be B for biobased concretes with a density exceeding 500 kg/m³ in agreement with most of data provided by Lanos [27]. A gypsum-straw concrete with a density of 550 kg/m³ and a thickness of 75 mm was also B-rated according to SBI test sponsored by one of us and performed by FCBA. Conversely, a clay-straw concrete with a density of 400 kg/m³ and a thickness of 200 mm was rated C (FIGRA_{0,2MJ} equal to 130.1 W/s) which is also in agreement with our findings [43].

Note that these predictions should be only suitable for 10 cm-thick concretes. Nevertheless, the front char in cone calorimeter test never exceeds 6 cm. Then the predictions are assumed still proper as long as the material is not too thin.

4. Conclusion

Fire behaviour of earth and gypsum-based concretes containing hemp was assessed using cone calorimeter for a wide range of density. The flammability is mainly a function of the density. The role of binder seems to be of lesser importance. Especially ignition is observed for low-density concretes even if the flame is not sustained more than 60 s. A threshold density roughly close to 500 kg/m³ is identified. Even if their surface temperature may reach high values, the heating rate of higher-density concretes is too low and fuel release is spread over a long period of time, preventing that fuel concentration in gas phase reaches a critical value for ignition. While the flame vanishes after <1 min, it does not significantly accelerate the decomposition rate during a long period, and residual fraction is not affected by ignition. Time-to-ignition values appear relatively scattered, and this point needs more investigations.

From ignition point of view, these concretes, at least the lightest ones, can be considered as thermally thin since heat transfer from the surface to the bulk is negligible. Critical heat flux for ignition is high (27 kW/m²) for one of the most flammable concretes. Peak of heat release rate remains low in most cases but reaches 100–130 kW/m² in case of ignition for the lightest concretes. Residual fraction and char front after 20 min also depend on the density. Smoke production is always negligible for these concretes.

Finally, SBI rating was predicted using ConeTools. If most concretes exhibit a very good rating (A2/B), some of them among the lightest ones are rated C or D. Further works are required to compare this prediction to SBI tests.

Further work will be also carried out to investigate the influence of other bioresources and binders to flammability, especially for low-

density concretes.

CRediT authorship contribution statement

Rodolphe Sonnier: Conceptualization, Data curation, Methodology, Project administration, Supervision, Validation, Writing – original draft, Writing – review & editing. **Oumaima Belkhane:** Data curation, Investigation, Methodology, Validation, Writing – review & editing. **Laurent Ferry:** Validation, Writing – review & editing. **Laurent Aprin:** Validation, Writing – review & editing. **Pierre Delot:** Conceptualization, Funding acquisition, Validation, Writing – review & editing. **Coralie Garcia:** Conceptualization, Funding acquisition, Validation, Writing – review & editing. **Arthur Hellouin de Menibus:** Conceptualization, Data curation, Funding acquisition, Methodology, Supervision, Validation, Writing – review & editing. **Hélène Lenormand:** Validation, Writing – review & editing. **Marc Potin:** Conceptualization, Funding acquisition, Validation, Writing – review & editing.

Declaration of Competing Interest

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 the properties (density, hemp content) influencing their fire behavior.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.conbuildmat.2022.127230>.

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