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RECYCLING OF RAW WHEAT HUSK TO MANUFACTURE MAGNESIA CEMENT BASED LIGHTWEIGHT BUILDING MATERIALS

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Abstract

In the last decades, the attention towards "carbon neutral" cement and agro-resources' by-products as new alternative raw building materials has grown exponentially. Within this context, the present study deals with the design of a lightweight insulating concrete based on raw wheat husk incorporated in a highly porous magnesia cement.

Concretes made from plant aggregates are commonly formulated with lime binders, with one of these materials being lime-hemp concrete. In this work, the possibility of substituting the traditionally employed calcic lime binder with an alternative magnesium binder was explored. In the majority of the research studies, the lignocellulosic particles associated with a mineral binder come from plant stems, as is the case of hemp hurds. Wheat husks, instead, are the main by-product of the industrial process for separating the grain kernel from the inedible hull of hulled wheat species, so that their use could significantly enhance their valorisation and meet zero-waste concept. Having examined physical and structural characteristics of these lightweight aggregates, specimens made out of wheat husk and both traditional lime binder (LWC) and alternative magnesium one (MWC) were manufactured. Both thermal and mechanical properties of the final concrete materials were studied and the results were compared to those obtained for hemp hurd concretes (LHC and MHC) manufactured with the same process.

The first results obtained for specimens cured for 10 days at 20°C and 50%RH, show higher apparent density for wheat husk concrete with an average compressive strength of 1.88±0.09 MPa in case of specimens made by alternative magnesium binder (MWC) and 0.16±0.03 MPa considering LWC. Thermal conductivity is 0.142±0.006 W/mK for LWC and 0.35±0.03 W/mK for MWC. Considering a suitable balance between thermal and mechanical properties, the use of wheat husks could be promising for the manufacture of new environment-friendly products as inner partition walls and thermal insulating components.

Keywords:

Wheat husks, magnesia cement, bio-based concrete, thermal conductivity; compressive strength.

1 INTRODUCTION

Over the last two decades, an increasing interest in green materials, technologies, and services have been observed. This is caused by the depletion of natural resources as well as the steadily increasing environmental responsibility and awareness of climate change. In this context, the development of eco-friendly materials containing locally available vegetable resources is a hot topic and interest has been paid to a wide range of agricultural waste (e.g. cereal by-products) which could potentially be used for this scope. Currently, the most important cereal crop in Europe are common wheat and spelt [Eurostat 2017]. It is cultivated for its grains which is the most important human food. Cereal by-products is an annually renewable source, available in abundant volume through the world. The traditional use of these husks and straws includes bedding for animals and livestock feeding. Under particular conditions they can also be incinerated to produce green electricity or heat. In terms of carbon footprint, burning cereal waste such as wheat husk makes no sense as it doesn't promote their valorization. Furthermore, cereal agro-wastes are difficult to pelletize due to their low-lignin and high-extractives content [Whittaker 2017].

According to November 2018, the world production of wheat was about 760 million metric tons [USDA 2018], where about one third produced by the European Union [Eurostat 2017]. Wheat husk is a lignocellulosic waste product which is about 20% of harvested wheat [McCartney 2006]. Considering that, approximately 50 million metric tons of wheat husk are produced annually in the European Union, which represents 1000 times the hemp hurds production in EU [Carus 2016].

The aim of this study is to evaluate the utilization of wheat husks to design light weight insulating concretes in the same way as for Lime Hemp hurd Concrete (LHC). At first, both commercial hemp hurd and wheat husk were characterized and the results were compared. The main investigations include: shape and particle-size distribution, water absorption, surface morphology, specific surface area (SSA) and bulk density. Two types of binder were selected in order to produce bio-based composites with both natural particles. The first one is a traditional lime binder, fully explored in literature and also used in industrial applications. The second one is a quite new hybrid organic-inorganic binder that could be an interesting alternative or even better to the previous. In particular, considering its composition based on magnesium oxide, magnesium sulphate solution ($MgSO_4 \cdot 7H_2O$) and a vegetal flour, it is expected to have a better environmental impact. Indeed, vegetal flour is obtained from renewable resources and the production of MgO from magnesite (MgCO3) requires lower temperatures compared to the conversion of CaCO3 to CaO. Additionally, concretes made from plant aggregates are commonly formulated with high alkaline pH cementitious or calcium lime binder for which the compatibility with polysaccharide carbohydrate-rich plants seems to be limited as delay or even lack of setting can occur [Peschard 2006; Laskowski 2007]. In this context, the use of more acidic magnesium binder as this hybrid organic/inorganic one (pH 9.8 -10) could be an interesting track to elaborate plant specific binder. In accordance with these aspects, Sassoni et al. [Sassoni 2014] designed new composites made with this organic/inorganic binder and hemp hurd as substitutes for traditional insulating materials. They exhibit very promising thermal and mechanical properties, generally comparable to those of commercially available products.

After choosing natural particles and binders, four mixtures were investigated (Tab.°1) and specimens were manufactured by mixing and manual tamping. Both thermal and mechanical properties of the final concrete materials were studied and the preliminary results were compared to those obtained for hemp concrete manufactured with the same process.

2 MATERIALS AND METHODS

2.1 Raw materials

Natural particles

Two different crop by-products were used for this work.

- Commercial hemp hurd, which is taken as reference as it is already used in association with a lime-based binder and marketed for individual housing construction.
- Wheat husk was selected as a local resource, coming from a cereal dehulling process operated by farmers' cooperative in San Felice sul Panaro (MO), Italy. In the present paper, wheat husk was proposed as an alternative lignocellulosic aggregate, obtained from a mechanical process of grain-hull separation.

Binders

Two different binders were used:

- The binder used as reference was a 50/50 wt% combination of a natural hydraulic lime (NHL3.5 according to EN459-1:2010) produced in Italy by T.C.S. srl and a hydrated calcic lime in the form of putty lime (CL90S according to EN459-1:2010) from La Banca Della Calce srl. All these materials are available as commercial products. The mixing water on binder mass ratio (Wm/B) was taken as 0.6 for all formulations.
- A hybrid organic-inorganic binder based on MgO, $MgSO_4$ and vegetal flour. Based on previous studies [Sassoni 2014] the molar ratio of $MgO/MgSO_4*7H_2O/H_2O$ was determinate as 6.4/1/15.3 and vegetal flour was added by 25 wt% of MgO weight.

2.2 Experimental procedures

Characterization of the natural particles

The measurements of bulk density, water absorption and particle size distribution by mechanical sieving and image analysis were performed following the recommendation of RILEM Technical Committee 236 Bio-aggregate based Building Materials [Amziane 2017]. In addition, the specific surface area of particles (SSA) were determined by gas adsorption (Gemini 2360). Microstructural analyses were performed by scanning electron microscopy (SEM, ESEM Quanta-200, FEI).

Formulations and specimens production

Four mixtures were prepared combining the two types of natural particles and the two binders selected, as presented in Tab.°1. The binder on aggregates mass ratios (B/A) equal to 2 was considered regarding practices and applications of hemp concrete.

Tab. 1: Mixtures notation.

Notations	Binders	Aggregates
LHC Lime hemp concrete	NHL3.5+CL90S	Hemp hurd
MHC Magnesium hemp concrete	$MgO+MgSO_4$ +vegetal flour	Hemp hurd
LWC Lime wheat concrete	NHL3.5+CL90S	Wheat husk
MWC Magnesium wheat concrete	$MgO+MgSO_4$ +vegetal flour	Wheat husk

The amount of water in the mixtures was equal to the sum of:

- Prewetting water of natural particles (Wp), fixed on the basis of water absorption tests considering the water uptake after 5 min of water immersion (Fig.°6).
- Mixing water (Wm), considering that binder mass ratio (Wm/B) was taken as 0.6 for lime concretes and 0.7 for magnesium concretes.

The mix proportions and the fresh density of concrete specimens are reported in Tab.°2.

The optimum mixing sequence for the production of lime hep concrete has not yet been established. Some authors form a slurry with water and binder before adding the hemp [Walker 2014; Arnaud 2012]. However, the high absorption ability of hemp hurds may cause problems in binder hydration and setting. This is the reason why some authors wet the hemp prior to adding the binder [Chabannes 2014]. Prewetting of the natural particles was also adopted in this work (mixing drum for 5min). Therefore, Wp/Aggregate was taken as 2 for wheat husk and 3 for hemp hurd. At the same time, in a separate concrete mixer, binder and a fixed amount of water were mixed for about 2 min. However, the high absorption ability of hemp hurds may cause problems in binder hydration and setting. This is the reason why some authors wet the hemp prior to adding the binder. The prewetted natural particles were subsequently added and mixing continued until a homogeneous mix was obtained. The total mixing time did not exceed 15 min.

Tab. 2: Mix proportions.

Mixture	LHC	MHC	LWC	MWC
W/B	2.1	2.2	1.6	1.7
Binder [kg/m^3]	228	227	288	363
Aggregate [kg/m^3]	114	114	144	182
Wm [kg/m^3]	137	159	173	254
Wp [kg/m^3]	343	341	288	363
Fresh density [kg/m^3]	822 ±53	842 ±29	891 ±34	1163 ±35

For thermal tests, the mixture was placed in 15 x 5 x 10 cm^3 moulds and compacted in 3 layers using a steel manual device. Each layer was subjected to a pressure of 5.8 kPa for 30 sec. The height of each single layer was equal to one-third of the total height of the concrete specimen (15 cm) and the mass of a single layer was equal to one-third of the total mass desired for the specimen, this mass being calculated according to the target density of the freshly-mixed concretes. Three pilot pins were inserted in the freshly prepared samples and left during curing in order to create holes to host the sensor (TR-1 needle) for thermal conductivity tests. The holes were positioned following the recommendations of the instrument manufacturer. For mechanical tests, the mixture was poured in cube moulds (5 x 5x 5 cm^3) and compacted in 1 layer using the same device, pressure and time adopted for specimens for thermal tests.

Specimen sizes and details regarding the compaction process for samples subjected to compressive and thermal tests are summarized in Tab.°3.

All specimens were demoulded after 24 h and stored in standard conditions (20°C and 50%RH) for 10 days prior to characterization.

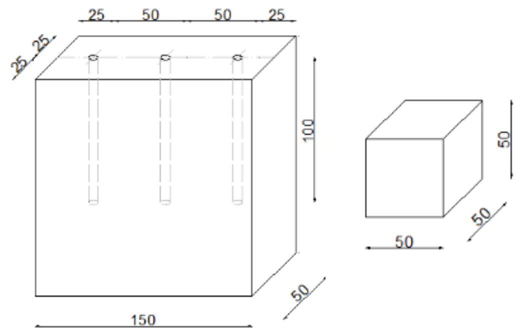
Characterization of bio based concretes

Thermal conductivity of concrete specimens was measured with thermal properties analyzer commercially known as KD2 Pro developed by Decagon Devices. It is constituted by a portable controller and a sensor (TR-1) designed primarily for soil, concrete and other granular or solid materials. Its operation is based on the infinite line heat source theory and it calculates the thermal conductivity by monitoring the dissipation of heat from the needle probe. Heat is applied to the needle for a set heating time, t_h, and temperature is monitored by that needle during heating and for an additional time equal to t_h after heating. The reading time, during which data are taken to compute thermal properties, was set at 10 min. The contact between needle and tested concrete is guaranteed by putting thermal grease in the hole for the emplacement of the needle. It is convenient to mention that, TR-1 sensor was previously calibrated before its use by a test tube supplied by the manufacturer. Considering the natural particles orientation after the concrete casting, thermal conductivity was measured parallel to aggregates orientation. These measurements were performed at a temperature of 20 ± 1 °C. Three values of thermal conductivity were measured for each hole in the concrete specimens.

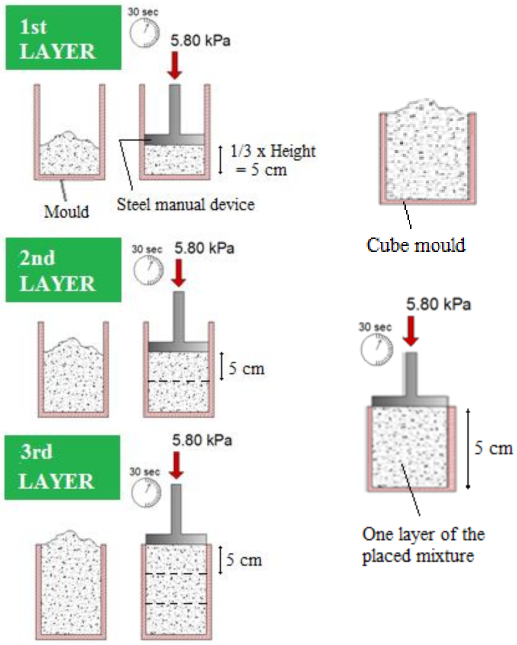
Tab. 3: Plant based concrete specimen sizes and compaction process for thermal tests and compressive tests.

Thermal tests	Compressive tests

Specimen sizes (mm)



Compaction process



After a curing period of 10 days, compressive strength (σ_c) was determined on 3 specimens (5 × 5 × 5 cm^3) for each concrete according to European standard EN 826. As shown in Fig.°1, depending on the stress-strain behavior of concrete specimens, the compressive strength (σ_{max} or σ_{10}) was identified. Tests were carried out by subjecting specimens to axial compressive strength in the compaction direction using an Instron 5567 electromechanical testing machine (maximum load 10 kN) at a constant displacement rate of 5 mm/min.

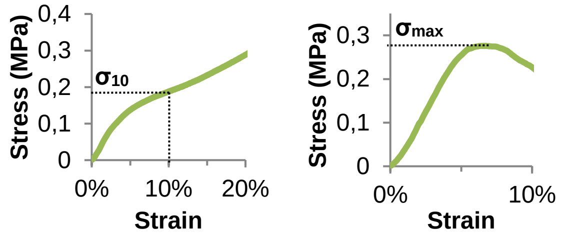


Fig. 1: Examples of stress-strain curves of concrete specimens.

3 RESULTS AND DISCUSSION

3.1 Characterization of the natural particles

Shape and Particle Size Distribution

The design of insulating concrete requires a special attention to the physical properties of the particles.

In this context, particle size distribution was evaluated for both natural particles. Conventional mechanical sieving offers an initial approach to the distribution of the width of particles (Fig.°2). Notably, it enables to evaluate the amount of finest particles contained in the hemp hurd and wheat husk.

As shown in Fig°2, wheat husks are characterized by a higher amount of fine particles (less than 0.5 mm) than hemp hurd. Only retained mass fractions on sieve size ≥ 0.5 mm were used in this study, so 11 mass% of hemp hurd particles and 24 mass% of wheat husk particles were discarded.

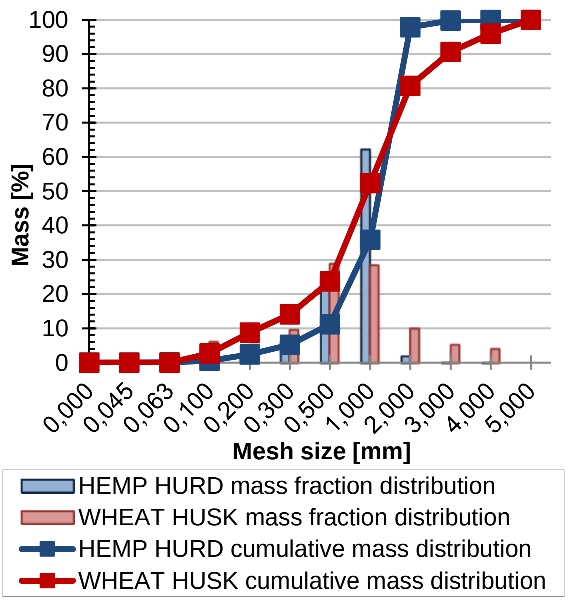


Fig. 2: Particle size distribution of wheat husk and hemp hurd obtained by mechanical sieving.

Considering only particles in the mesh size range 0.5mm - 5mm, hemp hurd has about 62 mass% of particles retained on sieve size 1mm. Assuming ellipsoidal, elongated and flaky shape, when these particles can pass lengthways through the sieves, their width may be oriented along the diagonals of the square holes. In this case, only those particles whose width is greater than $2^{1/2}d$ are retained by a sieve whose mesh size measures d. Therefore, this indicates that the width of 62 mass % of hemp hurds ranges from 1.4mm to 2.8mm. Instead of this, wheat husk particles show a more homogeneous mass distribution through the mesh size from 0.5mm to 4mm.

In this study mechanical sieving is used to supplement the image analysis techniques presented herein.
A comparison between the size distribution of hemp hurd and wheat husk is done in Fig.°3. Important information can be drawn from this analysis.

Wheat husk width ranges from 0.3 to 6.1 mm and the maximum length is about 13.8 mm. In the case of hemp hurd, the width distribution ranges from 0.3 mm to 5.6 mm and the length can reach up to 19.1 mm.

Even though the similar width size range which characterizes both particles, for the same cumulative area (Y axis), wheat husk particles width is smaller than hemp hurd one. In addition, the length size distribution is really similar for these two type of natural particles and only about 3% of hemp hurd cumulative area has a length size between 13.8mm and 19.1mm.

As a consequence, the elongation factor (EF), defined here as the length on width ratio (L/W) is higher for wheat husk particles (Tab.°4). This reflect a more spherical shape for hemp hurd aggregates.

The wheat husk length distribution presents a smaller size range in comparison to hemp hurd aggregates (Fig.°3). This latter difference could be due to their origin, since it is known that wheat husk sizes are strongly dependent on cereal grains size, therefore it shows a low variation in a representative statistical sample. In contrast, hemp hurd aggregates come from an industrial grinding process of hemp stems and thus generate a more dispersed distribution.

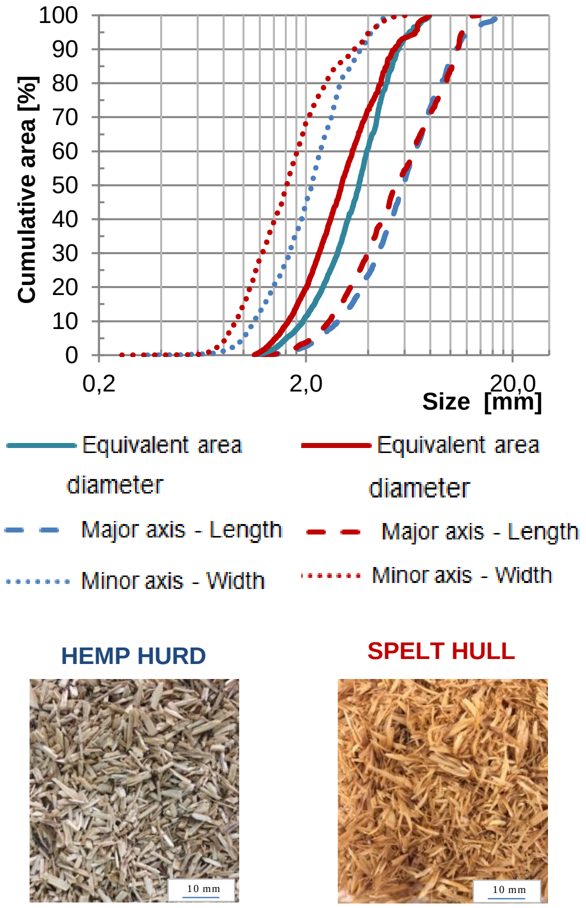


Fig. 3: Cumulative Particle Size distribution of wheat husk and hemp hurd aggregates.

Considering specimen preparation shown in Tab.°3, when the concrete is placed in the mould and compacted, size distribution and shape of the vegetable aggregates have a significant effect on the product characteristics in terms of granular stacking. In fact, these properties determine how the particles orient themselves and pack under applied pressure.

Tab.4: Geometrical characteristics for a cumulative distribution of 50%. Median width (W_{50}), equivalent area diameter ($\Phi_{s\text{-}50}$), length (L_{50}), and elongation factor (EF_{50}).

Median parameters	Wheat husk	Hemp hurd
W_{50} (mm)	1.61	2.13
$\Phi_{s\text{-}50}$ (mm)	2.98	3.61
L_{50} (mm)	5.50	5.94
EF_{50} (L_{50}/W_{50})	3.42	2.79

Microstructure and porosities

The cross-sectional view of single aggregate particles was investigated by scanning electron microscopy (SEM) and representative images are reported in Fig.°4.

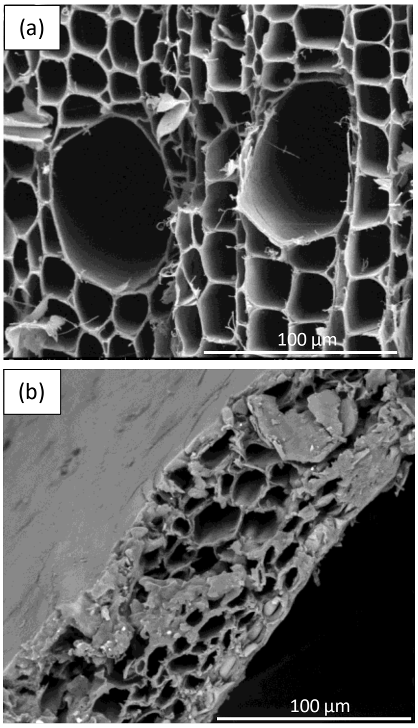


Fig. 4: SEM micrographs of hemp hurd (a) and wheat husk (b).

Fig.°4a highlights the highly porous nature of hemp hurd which consists of two types of macro-pore channels of different sizes. The first type results from the vascular bundles and is characterized by round-like or elliptic large pore channels with a diameter in the range 50–90 µm. The second type, having a honeycomb structure, derives from the ground tissues and surrounds the larger pores. These pore channels are polygonal or round-shaped with a diameter in the range 20–30 µm. Both types of channels are parallel and oriented along the major axis of the particle. In addition, the inner walls of the channels are porous, leading to the formation of a hierarchical pore structures and connected pore geometry. These observations are in agreement with earlier descriptions in the literature [Jiang 2017].

Fig.°4b shows a SEM micrograph of wheat husk taken with the same magnification as the one reported for hemp hurd (Fig.°4a). The structure of a wheat husk is rather different, starting with its thickness which is about 90 µm. The internal porous structure of wheat husk is composed of small vessels with a diameter of less than 30 µm.

The aggregate surface morphology and especially its roughness is an important factor which could have a great influence on aggregate-matrix adhesion.

Fig.°5a reports the longitudinal section view of a hemp hurd particle. It is characterized by channels parallel to each other and to the growth direction of the hemp stem. Instead, the wheat husk surfaces are characterized by peculiar morphologies (Fig.°5b, Fig.°5c) considering adaxial or abaxial surface of the husk. The adaxial surface has higher roughness with respect to the abaxial one and it presents a specific type of epidermis cells (aerial surface hairs) rich in silica, called trichomes.

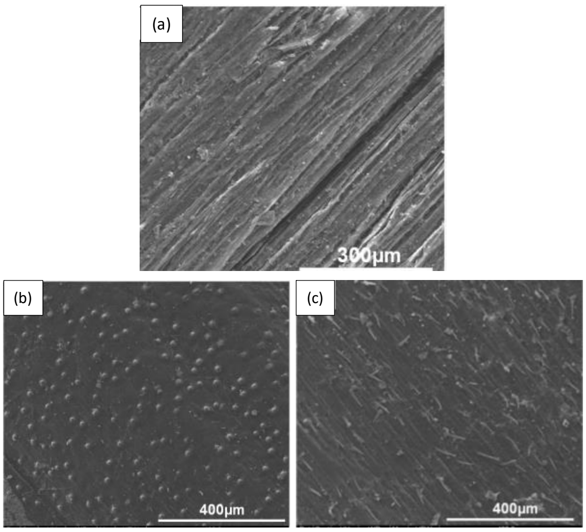


Fig. 5: SEM micrographs of (a) hemp hurd longitudinal section, wheat husk (b) abaxial surface (Outer/convex face) and (c) adaxial surface (inner/concave face).

They perform several functions, such as retaining moisture under continuous light during sunny days and protecting the plant and the wheat grain from leaf-consuming vermin. The abaxial surface of wheat husk is smoother than the inner one, but it is rich in protuberances (Fig.°5b) where silica is usually found.

SSA and water absorption

The SSA of hemp hurd was measured to be 1.05 m^2/g, which is higher than the value of 0.69 m^2/g obtained for wheat husk. These results are in agreement with SEM pictures presented in Fig.°4, where a significantly higher porosity was observed for hemp hurd. It means that hemp hurd particles expose more effective surface available for interaction with the surrounding matrix. Considering this aspect, the primary physical mechanism that permits adhesion between aggregates and matrix is mechanical interlocking. Surface roughness and porosity create anchorage points and enables binder penetration. This is a crucial factor for mechanical properties of a concrete.

In Fig.°6 the water absorption behavior of natural particles in function of the immersion time is reported.

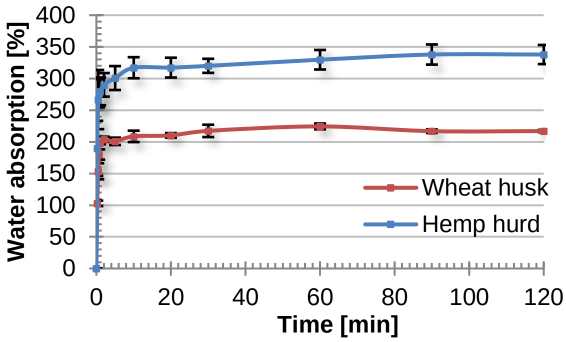


Fig. 6: Water absorption as a function of time of wheat husk compared to hemp hurd aggregates.

The results of water absorption tests underline that wheat husks absorb less water than hemp hurds. After the first minute of water immersion, the amount of water absorbed by wheat husk was about 200% whereas the corresponding value for hemp hurd was about 280%. During this first stage, water fills the open particle porosity driven by capillarity forces [Banks 1973]. Hemp hurd has an absorption kinetic of 1.7 times higher than that of wheat husks. The difference in the amount of water absorbed between hemp hurd and wheat husk can be explained by the higher SSA and open porosity of the former.

In particular, the fast water absorption of natural aggregates during the first minute of immersion is an essential aspect to be considered during concrete mixing. Indeed, it creates a competition for water between binder and natural particles: hurds and husks tend to absorb the water which the binder needs for hydration with a disrupting effect on the setting of concrete. Water presence and content, moreover, influence not only mechanical properties, but also thermal and acoustic performances of concrete [Collet 2014]. Therefore, the water absorption capacity of the aggregate provide important information about the amount of water needed for the concrete mix design.

Bulk density

The bulk density of wheat husk and hemp hurd was 146±2 kg/m^3 and 127±1 kg/m^3, respectively. It means that wheat husk requires lower storage and transportation space, thus providing lower handling costs. The difference in bulk density between the two kinds of aggregate depends upon particle size distribution, shape and chemical composition.

The bulk density of hemp hurd is very close to the values found in literature [Bourdot 2017]. For wheat husk, the bulk density is similar to the values given in literature for rice husk, which ranges from 100-200 kg/m^3 [Chabannes 2014]. This is strongly linked to the size and shape of particles. Indeed, both rice husks and wheat husks have a boat-like shape consisting of two interlocking halves and their dimensions depend on the size of the rice/cereal grain.

3.2 Characterization of bio based concretes

Apparent density

The average apparent density of the different concretes after 10 days are given in Tab.°5.

The density of LHC (405 kg/m3) correspond to the usual density of Lime Hemp Concrete wall mixture destined for use as an insulting filing material in a wall timber frame. It is recommended by the professional rules in hemp construction and studied in several research work [Chabannes 2018]. The LWC concrete reached an apparent density of 501 kg/m3. Considering the same B/A=2 and the same natural particle, the mixtures made with magnesium cement are denser than those with lime cement. In particular, the apparent density is 35% and 20% higher for specimens made with wheat husks and hemp hurd, respectively.

Tab. 5: Average apparent densities after 10 days of curing (20°C and 50%RH).

Mixture	Apparent density (kg/m^3)
LWC	501±37
LHC	405±24
MWC	783±34
MHC	500±17

Compressive strength (σ_c)

As it shown in Fig°7, the type of aggregate used to manufacture concrete specimens affect the concrete's behavior in compression. In the case of LWC and MWC the cubes continuously deform under stress. After the first part of the curves, where a linear quasi-elastic behavior is observed, the deformation does not lead to the fracture of the compressed specimen, but induces a continuous increase of stress.

Tab°6 reports the average compressive strength for different concrete specimens.

Tab. 6: Average compressive strength after 10 days of curing (20°C and 50%RH).

Mixture	Compressive strength (MPa)
LWC	0.16±0.03 (Strain=10%)
LHC	0.30±0.03
MWC	1.88±0.09 (Strain=10%)
MHC	0.63±0.03

As shown in Tab.°6, the use of magnesium binder instead of the lime one increases the concrete compressive strength for both types of aggregates. It should be mentioned here that the mechanical properties of the lime-based concrete probably would have been higher following prolonged curing. In fact, the low carbonation rate of hydrated lime generally lasts for months and even years. The strenght after 10 days of curing is thus mainly given by the calcium silicate hydrates (C-S-H) which are formed following hydration of C2S present in the natural hydraulic lime (NHL3.5) used in the mix.

Thermal conductivity

The results of thermal conductivity measurements are reported in Tab°7.

It was found that the thermal conductivity is higher for concrete made with wheat husk. This could be related to their higher apparent density compared to hemp hurd concretes. It is known from literature [Collet 2014], that thermal conductivity increases with the increasing of specimen's apparent density.

An interesting observation is that the LWC and MHC concretes have the same apparent density (Tab°5), but really very different thermal conductivity (Tab°7).

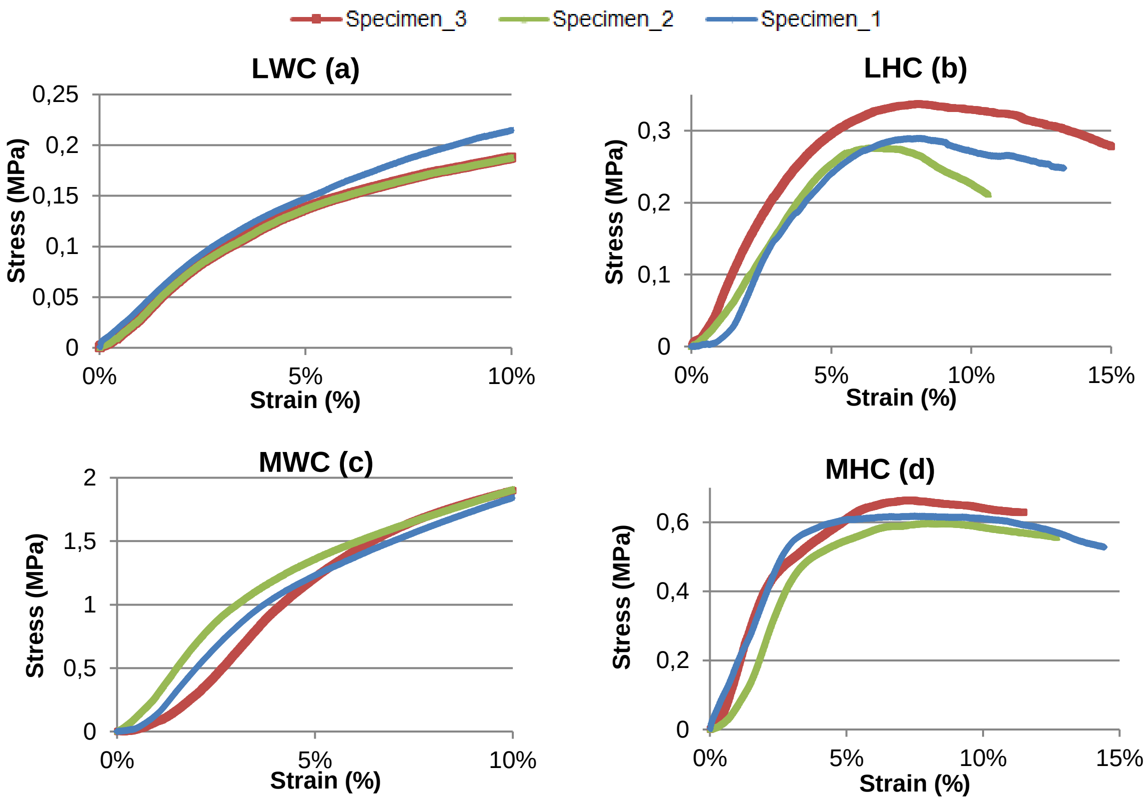


Fig. 7: Stress-strain curve of concrete specimens*: lime wheat husk concrete_LWC (a), lime hemp hurd concrete_LHC (b), magnesium Wheat husk concrete_MWC (c), magnesium hemp hurd concrete_MHC (d).*

The main motivation could be connected to their water content. In fact, the W/B mass ratio for LWC is equal to 1.6 (Tab.°2) and it lost about the 44% of its weight after 10 days of curing. Instead, MHC which was manufactured with a W/B mass ratio equal to 2.2, lost about 40% of its weight after 10 days of curing. Hence, MHC has a higher water content than LWC which could explain the higher thermal conductivity of the former.

Tab. 7: Average thermal conductivity after 10 days of curing (20°C and 50%RH).

Mixture	Thermal conductivity (W/mK)
LWC	0.142±0.006
LHC	0.118±0.008
MWC	0.35±0.03
MHC	0.225±0.007

4 CONCLUSION

In this study, the possibility of using wheat husks as aggregate in lightweight concrete was investigated. With this purpose, wheat husk was compared to commercial hemp hurd. The image analysis highlight similar width size ranges (from 0.3mm to 6.1mm) and smaller length distribution of wheat husk aggregates. However, the most important differences between the two particles are observed in the porosity network. Indeed, wheat husk is characterized by little vessels whose diameter (less than 30 μm) are smaller than vascular bundle structures observed in hemp particles.

The smaller SSA observed for wheat husk influences the particle water absorption capacity. Indeed in water absorption test, as early as one minute of water immersion, the amount of water absorbed by wheat husk was about 30% smaller than hemp hurd.

Tests done after 10 days of curing on concrete specimens show interesting results. Compressive tests performed on concrete made with calcic lime binder (LHC and LWC) highlight the weakness of wheat husk concrete, for which the compressive strength is about 50% smaller compared to the hemp hurd one.

However, mechanical behavior under compression confirms the interest of hybrid organic/inorganic binder for future development with both type of natural particles. Indeed, according to the results obtained, the lightweight concretes manufactured with this binder (MHC and MWC) highlight higher compressive strength than those made with calcic lime (LHC and LWC). In particular, the compressive strength of MWC is 12 times bigger than LWC and the value observed for MHC is 2 times bigger than those of LHC.

Considering the high value of thermal conductivity obtained for both MWC and MHC, further research could be focused on improving the thermal properties of lightweight concrete made with hybrid organic/inorganic binder without amending its mechanical performances.

5 ACKNOWLEDGMENTS

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