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Original Article

Effect of compaction on multi-physical properties of hemp-black liquor composites

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ABSTRACT

This study aims to develop fully agro-based insulating building materials. Previous studies investigated several types of aggregates and binders to develop composites. The thermal conductivity of such composite appears mainly impacted by the density. This paper aims to investigate the effect of density on various multi-physical properties of composites, using the same formulation and adjusting the forming step. Thus, specimens are produced with the same hemp shiv to black liquor ratio but different compaction stress. The effect of compaction on bulk density, porosity, mechanical resistance, thermal conductivity and Moisture Buffer Value (MBV) is analyzed. It is shown that it is possible to reach thermal conductivity that is low enough to be considered as building insulating material. Thanks to this study, the required compaction stress to ensure targeted values of density, then thermal conductivity and MBV, is identified.

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1. Introduction

The agricultural waste valuation is one of the current challenges in the field of green building material [1–3]. Agricultural waste can be used to produce bio-based panels with low environmental impact (embodied energy and carbon footprint) and high hygrothermal efficiency [2,4–6]. As part of the ISO-BIO project [7], the developed thermal insulating panels are ecofriendly composites made of aggregates coming from local agriculture and green binders. The gluing effect is obtained by coupling thermal and mechanical treatment.

In a previous study, presented in [8], various composites were investigated with Biofibat® hemp shiv or corn cob residues as aggregates, and corn cob extract, flax fines extract, black liquor, BioChoice® lignin or PLA as binders. After mixing, the composites are produced by compacting and then heating leading to the gluing of the aggregates. This study showed that the developed composites had low thermal conductivity (ranging from 0.067 to 0.148 W/(m.K)) and excellent moisture buffering ability (MBV >2 g/(m^2.%RH)). More, the thermal conductivity increased linearly with density, whatever the kind of aggregate and of binder.

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The density of composites is mostly implied by the compaction stress applied during their production, among other factors. The influence of compaction on physical properties (density, thermal conductivity and compressive stress in particular) of composites has been highlighted by Balciunas [9] in the case of hemp-sapropel composites (sediment rich in organic matter) compacted at 20, 40 and 60% of their initial volume.

This study focuses on the composites obtained with hemp shiv and black liquor.

Hemp shiv are lightweight aggregates with high porosity and interesting hygrothermal properties [10]. This makes them perfectly suited for the development of bio-based insulating materials. They are the most commonly used aggregates in the literature for the production of building materials [2].

Black liquor is a renewable binder from local industry, is better for the environment (less CO_2 emission and preservation of fossil resources) than using petroleum-based binder (urea-formaldehyde resin or phenolic resin). More, the use of black liquor leads to a better hygrothermal properties of bio-based composites than the use of petroleum-based binder (polyvinyl acetate) as highlighted in previous study [11]. Black liquor is readily available component since ten tonnes of black liquor are produced per tonne of pulp using the Kraft pulping process [12,13]. Currently, black liquor is mainly used for the production of energy. However, efficient energy production is cumbersome and expensive [12,14,15]. It is therefore important to seek to enhance this co-product in a different way. Due to its chemical composition (Kraft lignin, polysaccharides, aromatics and aliphatics components), black liquor represents an interesting alternative for the synthesis of sustainable chemicals [16–18].

For this study, the composites are produced with the same hemp shiv to black liquor ratio but seven different compaction stresses applied during forming step (15.6, 31.2, 62.5, 125, 250, 500 and 1000 kPa).

Thus, this study investigates the effect of compaction conditions on density, porosity, mechanical, thermal and hygric performances of composites. It will then be possible to define production compaction regarding target values of multi-physical properties.

2. Materials and method

2.1. *Raw materials*

The composites are produced with Biofibat® hemp shiv as aggregates and black liquor as binder.

The Biofibat® hemp shiv were produced in 2016 year by CAVAC (Sainte-Gemme-la-Plaine, France). Its particle size distribution was determined by image analysis as described by Amziane et al [19]. The length of hemp shiv ranges from l_{10} = 5.5 mm to l_{90} = 19.4 mm, with an medium value l_{50} of 11.5 mm (Fig. 1). The width ranges from ranges from w_{10} = 1.7 mm to w_{90} = 4.8 mm, with an medium value w_{50} of 2.9 mm. The elongation ratio (length/width) ranges from 2.2 to 6.5.

Bulk hemp shiv have interesting thermal properties (with a thermal conductivity between 48 and 59 mW/(m.K) [9,20,10]) due to their low bulk density. They also show excellent ability to regulate ambient relative humidity (MBV about 2.31 g/(m^2.%RH) [10]).

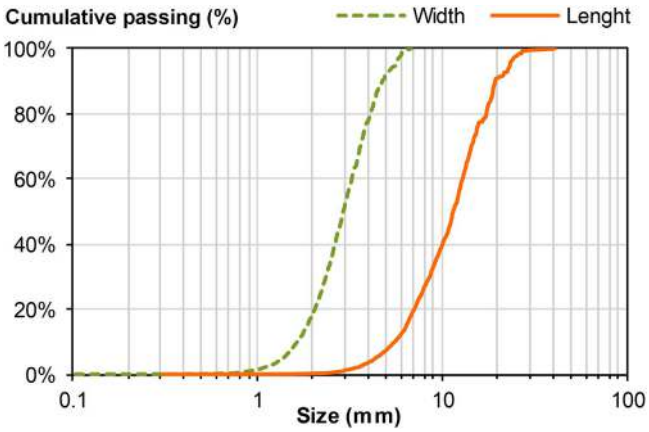

Fig. 1 – Particle size distribution of Biofibat® hemp shiv.

Table 1 – Chemical composition of kraft black liquor according to the safety data sheet.

Components	Content (% in weight)
Alkali lignin	14.1
Carboxylic acid	1.1
Acetic acid	4
Formic acid	10
Hydroxide acid	2.8
Polysaccharides	5.4
Sulfate	3.4
Sulfur	8.3
Sodium	15.9
Other components	35

The black liquor is a low cost by-product from the kraft paper industry [18]. It is composed of about 70% dry matter. Its chemical composition mainly includes lignin and inorganic components which mainly come from the paper industry process. It also includes few polysaccharides and some aromatic and aliphatic components (Table 1) [16,21]. Fig. 2 is a diagrammatic illustration of the black liquor structure. Durmaz et al and Nayeri et al also observed that the black liquor protect the ligno-cellulosic resources from fungal development [17,22].

2.2. *Composites*

For each kind of composites, six specimens (10 × 10 × 10 cm^3) are produced. Three of them are used for thermal and hygric characterization, the three others for mechanical characterization and measurement of skeleton density. The same black liquor to hemp shiv dry mass ratio of 15% is used. This value is chosen in order to ensure a good cohesion [23].

For preparation, the hemp shiv are mixed with the black liquor in a mixer with a flat paddle during 5 minutes. The mix is split into three parts to produce three specimens. Specimens are molded and compacted 5 times using an Instron 5988 testing machine fitted with a upper plunger, to ensure a good particles arrangement. They are maintained under compression and heated (for 2 hours at 190 °C), cooled to room temperature and demolded (Fig. 3).

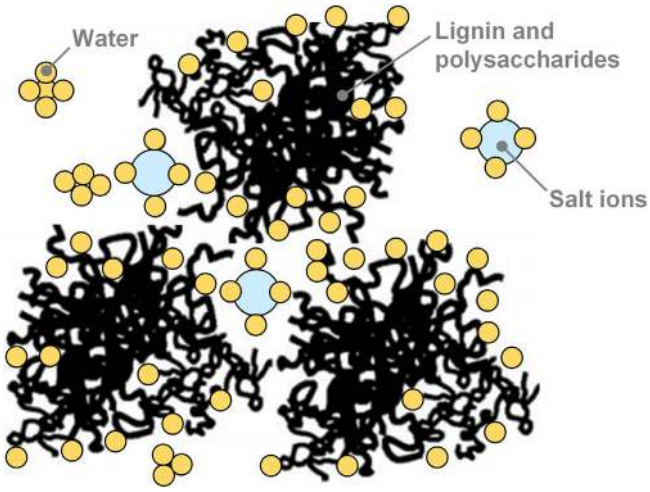

Fig. 2 – Diagrammatic illustration of the black liquor structure [21].

To study the effect of compaction on multi-physical properties, seven compaction levels are considered: 15.6, 31.2, 62.5, 125, 250, 500 and 1000 kPa.

2.3. *Characterization methods*

2.3.1. *Densities and porosity*

The density is the ratio of the weight to the size of the specimen (which is approximately $10 \times 10 \times 10$ cm^3). The weight is obtained with an analytical balance (Sartorius LP8200S, Göttingen, Germany). Each dimension (length, width, height) is the average of four values measured with an electronic caliper (readability of 0.1 mm). It is measured on the six specimens of each kind of composite.

The skeleton density ρ_s is the ratio of the weight to the skeleton volume of the specimen. This measurement is carried out using a pycnometer according to the standard ASTM D854 [24]. A known mass m_{sample} of dry and crumbled composites (for a volume of about 200 ml) is introduced into a pycnometer of about 600 ml. Then, the pycnometer is filled with toluene. It is regularly shaken to remove air bubbles. After ensuring that no air bubbles are trapped between the particles, the pycnometer is completely filled with toluene and the volume of toluene displaced by the crumble sample V_{sample} is determined following the equation (1). Three measurements are performed for each composite.

$$\rho_s = \frac{m_{sample}}{V_{sample}} = \frac{m_{sample}}{V_{pycno} - V_{toluene}} = \frac{m_2 - m_1}{m_4 - \frac{m_3 - m_2}{\rho_{toluene}}} \quad (1)$$

with:

- m_1: mass of the empty pycnometer with ground-glass stopper;
- m_2: mass of the pycnometer with ground-glass stopper and sample;
- m_3: mass of the pycnometer completely filled with toluene with ground-glass stopper and sample;
- m_4: mass of the pycnometer completely filled with water with ground-glass stopper;
- $\rho_{toluene}$: density of the toluene at the temperature of the measurement;
- ρ_{water}: density of water at the temperature of the measurement.

The total porosity n_t of a composite is the volume ratio of the porosity to the total volume of the specimen. It is then related to apperent and skeleton density of composite following (Equation (2)).

$$n_t = \frac{\rho_s - \rho_{app}}{\rho_s} \quad (2)$$

with:

- ρ_s: density of the skeleton of the sample;
- ρ_{app}: density of apparent density of the sample.

2.3.2. *Mechanical characterization*

For each composite, a compression test is performed on three specimens to determine the average compressive strength. The test is carried out on a testing machine (Zwick/Roell

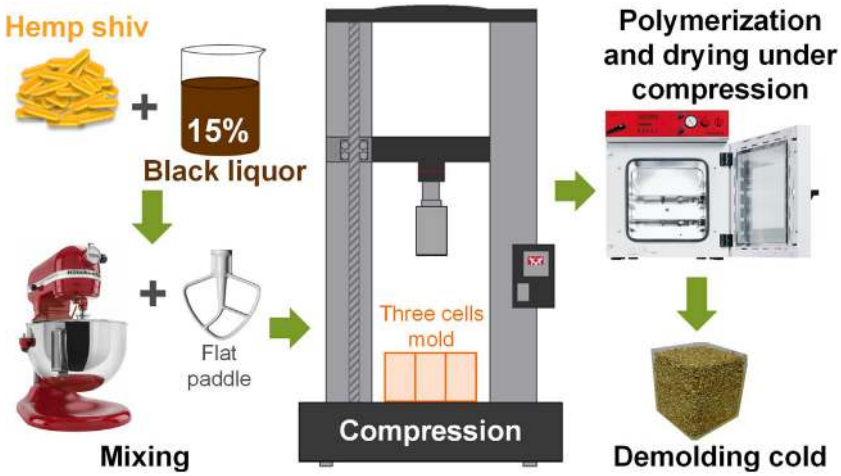

Fig. 3 – Flow chart of composites production.

ProLine, Metz, France) fitted with a 20 kN load cell (Zwick/Roell XForce, Metz, France). The load is applied on the specimen, by the monotonous displacement of the upper steel plate with cross-head speed of 0.05 mm s^{-1}.

Following the recommendations of the NF EN 826 standard [25], the results of the compression tests are studied using stress-strain curves as described by Viel et al [8].

2.3.3. *Thermal characterization*

For each composite, the thermal conductivity is measured three times on three pairs of specimens obtained by coupling three specimens, with the hot wire transient method [26] using commercial "*CT meter*" device (SMEE, Voiron, France). The heating power and time are 142 mW and 120 seconds. Previously to the measurement, specimens are stabilized at (23 °C; dry state) and at (23 °C; 50%RH).

The dry state is reached after the drying of composites in an oven at 60 °C until their mass stabilization (the variation must be lower than 0.1% between two consecutive weighing for three consecutive weighing with a 24-hours time step). Then, the composites are placed in desiccator at 23 °C. The measurements are performed once the weight stabilization of composites is reached (same stabilization criterion as previously). The stabilization at (23 °C; 50%RH) is performed in a climate chamber.

2.3.4. *Hygric characterization*

For each composite, the moisture buffer value is measured on three specimens, according to the Nordtest protocol [27]. It relates the amount of moisture uptake or release to the exchange area as a function of relative humidity (Equation (3)).

$$MBV = \frac{\Delta m}{A.(RH_{high} - RH_{low})} \quad (3)$$

with:

- MBV: moisture buffer value (g/(m^2.%RH));
- Δm: moisture uptake/release during the period (g);
- A: open surface area (m^2);
- $RH_{high/low}$: high/low relative humidity level (%).

Specimens are sealed on all their surfaces except one. After stabilization at (23 °C; 50%RH), the relative humidity is submitted to daily cyclic variations: 8/16 hours at 75/33%RH (absorption/desorption period), during 5 days in a climate chamber (Vötsch VC4060, Balingen, Germany).

3. Results

3.1. *Densities and porosity*

Table 2 gives the physical properties of composites versus compaction stress.

The apparent density of developed composites, at dry state, ranges from 128 to 247 kg/m^3. It increases with the compaction pressure following the equation given in Fig. 4 with a high correlation coefficient. So, it is possible to identify the compaction pressure to apply for a target density. The non-linear curve given by Fig. 4 is due to the mechanisms of compaction that take place during the composites production: (i) grain rearrangement and (ii) elastic and plastic deformations of particles [28]. The Cooper-Eaton model makes it possible to clearly distinguish these two phenomena [29].

Table 2 – Effect of compaction on apparent densities at (23 °C; 50%RH) and 23 °C; dry point), skeleton density and porosity of composites.

σ_p (kPa)	$\rho_{23°C-50\%RH}$ (kg/m^3)	$\rho_{23°C-dry}$ (kg/m^3)	ρ_s (kg/m^3)	n_{tot} (%)
15.6	135.8 ± 3.7	127.6 ± 3.7	1345.2 ± 46.4	90.5
31.2	144.4 ± 3.8	135.3 ± 3.6	1325.2 ± 11.4	89.8
62.5	155.0 ± 2.8	144.9 ± 2.7	1294.2 ± 5.4	88.8
125	171.6 ± 1.2	160.3 ± 1.2	1282.9 ± 6.7	87.5
250	191.4 ± 0.9	180.7 ± 0.9	1211.0 ± 7.2	85.1
500	229.4 ± 3.3	213.7 ± 3.1	1269.9 ± 2.1	83.2
1000	265.9 ± 1.3	247.0 ± 1.6	1283.0 ± 11.0	80.7

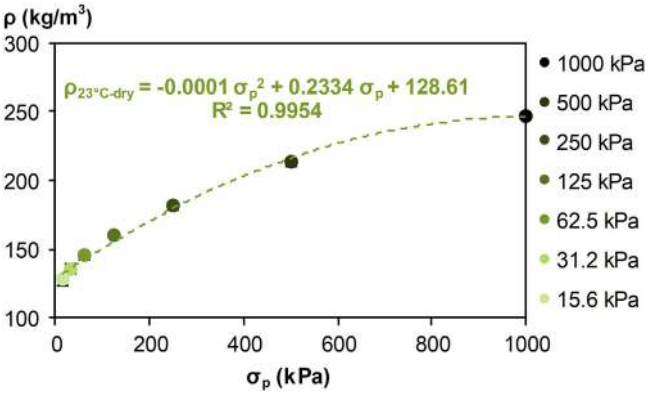

Fig. 4 – Apparent density of composites at (23 °C; dry point) versus the pressure applied (σ_p) during the composites process.

Furthermore, the density of composites increases by 7% from the dry point to the wet point at (23 °C; 50%RH).

The developed composites have very similar skeleton densities ranging from 1211 to 1345 kg/m^3. The average of these values is 1300 kg/m^3 with a variation of ± 3.33%. The formulations of the seven composites developed in this paper are the same, so it is consistent to have the same skeleton density.

Moreover, the hemp-starch composites developed by Bourdot et al, have a skeleton density close to this value. Indeed, they have a skeleton density of 1249 kg/m^3 with a variation of ± 1.02% [30].

Since the skeleton density is the same for all composites, the total porosity of the composites evolves linearly with their apparent density, following the relationship between these 3 values given Equation (2), and as highlighted in Fig. 5. At the dry point and it ranges from 80.7% to 90.5%.

3.2. *Mechanical resistance*

The developed composites made with hemp shiv show compacting behavior (continuous stress increased versus strain - Fig. 6). Thus, the mechanical performance is given by the compressive strength obtained for the longitudinal deformation ϵ = 10%.

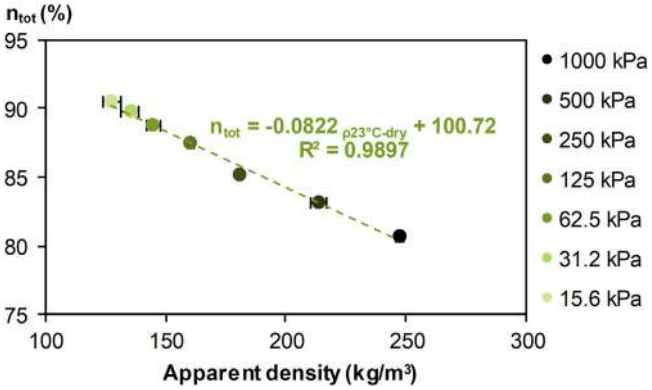

Fig. 5 – Porosity of the developed composites versus their apparent density at (23 °C; dry point).

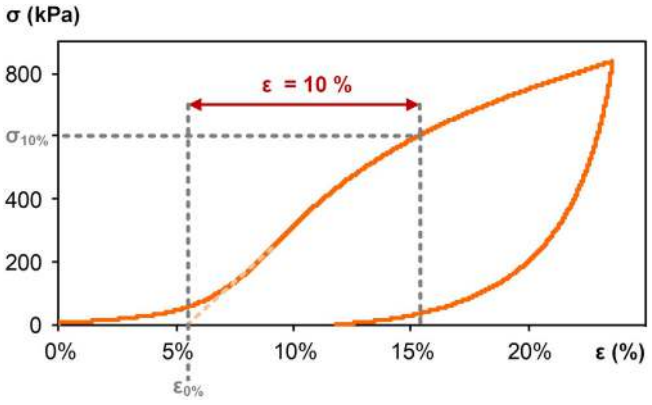

Fig. 6 – Stress-strain curve of the composites compressed at 31.2 kPa during the production process.

Table 3 – Stress at 10% deformation for each composites.

σ_p (kPa)	$\rho_{23°C-50\%RH}$ (kg/m^3)	$\sigma_{10\%}$ (kPa)	$\epsilon_{h=3m}$ (%)
15.6	134.7 ± 3.0	54.6 ± 10.8	0.50
31.2	144.9 ± 1.2	87.7 ± 0.1	0.28
62.5	150.6 ± 0.4	105.7 ± 6.2	0.24
125	169.1 ± 1.4	194.8 ± 12.9	0.19
250	203.2 ± 1.1	230.1 ± 6.5	0.22
500	221.8 ± 3.8	613.3 ± 7.8	0.09
1000	270.8 ± 16.7	802.7 ± 131.2	0.09

Table 3 and Fig. 7 show the mechanical properties measured on composites.

For a given compaction pressure (between 15.6 and 500 kPa) during the production of composites, the experimental values are very close to each other between the three specimens. In contrast, the composites compressed at 1000 kPa during the production process have experimental values with a larger measurement deviation (coefficient of variation of about 16%) due to a significant variation in apparent densities between the three specimens. The compressive strength ranges from 55 to 803 kPa. The composite compressed at 1000 kPa during the production process has the highest compressive strength and the composite compressed at 15.6 kPa has the lowest. Fig. 7 underlines that compressive strength evolves linearly with bulk density.

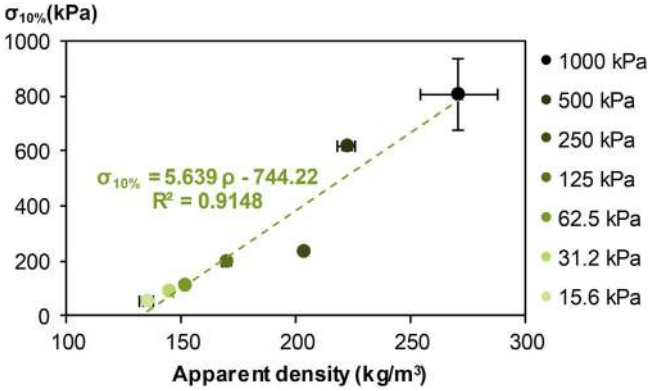

Fig. 7 – Stress at 10% deformation of the developed composites versus their apparent density.

Table 4 – Effect of compaction and humidity (dry and 50%RH) on thermal conductivity of hemp - black liquor composites at 23 °C.

σ_p kPa	ρ_{dry} (kg/m^3)	λ_{dry} (mW/(m.K))	$\rho_{50\%RH}$ (kg/m^3)	$\lambda_{50\%RH}$ (mW/(m.K))
15.6	127.6 ± 3.7	62.8 ± 1.1	134.6 ± 3.4	71.0 ± 1.1
31.2	135.3 ± 3.6	65.7 ± 1.9	143.3 ± 3.9	73.1 ± 1.8
62.5	144.9 ± 2.7	65.6 ± 1.3	153.7 ± 2.7	72.7 ± 1.0
125	160.3 ± 1.2	68.9 ± 0.9	170.1 ± 1.1	77.8 ± 1.0
250	180.7 ± 3.1	71.1 ± 1.5	190.4 ± 1.0	78.2 ± 3.0
500	213.7 ± 0.9	79.8 ± 2.8	225.4 ± 3.4	91 ± 1.9
1000	247.0 ± 1.6	93.2 ± 1.9	258.8 ± 1.6	101.5 ± 1.8

For 3 meters high wall, the stress induced by the composites density leads to $\epsilon_{h=3m}$ deformations lower than 0.50%. The mechanical properties meet the requirements to be used as building insulating panels without compaction risk.

Balciunas et al [9] also noticed on hemp-saprobel composites that the compressive strength (from broken before measurement to 2080 kPa) increases with the apparent density of composites (from 157 to 401 kg/m^3).

3.3. *Thermal conductivity*

The thermal conductivity of hemp - black liquor composites ranges from 62.8 to 93.2 mW/(m.K) at dry point and from 71.0 to 101.5 mW/(m.K) at wet point (Table 4). The composite with the lowest compaction pressure (15.6 kPa) meets the requirements to be classified as insulating building materials regarding the NF P 75-101 standard (λ <65 mW/(m.K)) [31].

Fig. 8 underlines that the thermal conductivity increases linearly with density with a high correlation coefficient. More, Fig. 8 also highlights that the slope is twice the slope obtained for agro-resources [10]. This may be attributed to the addition of binder and the compaction which reduces inter-particular and maybe intra-particular porosity. Thanks to the linear

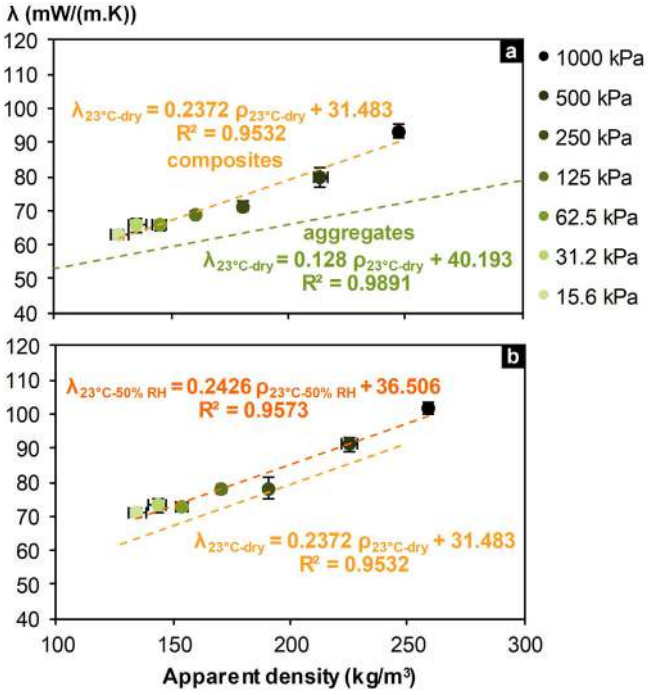

Fig. 8 – Thermal conductivity of hemp - black liquor composites versus their apparent density: (a) Comparison between the composites (yellow curve) and the agro-resources in bulk (green curve [10]) at dry point and (b) Comparison of thermal conductivity values between dry point (yellow curve) and wet point (orange curve).

Table 5 – Effect of compaction on MBV of composites (average value and standard deviation).

σ_p (kPa)	MBV_{abs} (g/(m².%RH))	MBV_{des} (g/(m².%RH))	MBV_{av} (g/(m².%RH))
15.6	2.50 ± 0.03	2.77 ± 0.02	2.63 ± 0.02
31.2	2.60 ± 0.09	2.86 ± 0.09	2.73 ± 0.09
62.5	2.82 ± 0.11	3.06 ± 0.11	2.94 ± 0.11
125	2.63 ± 0.07	2.87 ± 0.06	2.75 ± 0.06
500	2.31 ± 0.01	2.49 ± 0.01	2.40 ± 0.01
1000	2.31 ± 0.12	2.49 ± 0.10	2.40 ± 0.11

regression, it is possible to identify the required density to reach a target thermal conductivity.

The thermal conductivity at wet point is about 10% higher than the thermal conductivity at dry point. The slope versus density remains the same.

Balciunas et al [9] had the same findings on hemp-saprobel composites that the thermal conductivity increases with the apparent density of composite (λ = 53 to 73 mW/(m.K) for ρ = 147 to 401 kg/m³).

3.4. *Moisture buffer value (MBV)*

Following Table 5, the MBV ranges from 2.40 to 2.94 g/(m².%RH). All the composites are excellent hygric regulators according to the Nordtest classification (MBV >2 g/(m².%RH)) [27].

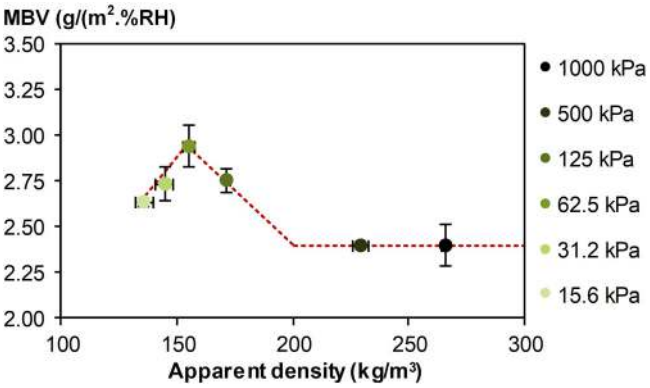

Fig. 9 – MBV of hemp-black liquor composites versus density.

The lowest MBV is obtained for the two composites with the highest densities (compacted at 500 and 1000 kPa). The highest MBV is obtained for the composite compacted at 62.5 kPa. Fig. 9 gives the MBV versus the apparent density of composites.

In a first time, the MBV increases up to 2.94 g/(m².%RH) for a density of 170 kg/m³. Then, it decreases down to 2.40 g/(m².%RH) for density of 200 kg/m³. Finally, the MBV remains constant. Such evolution may be explained:

- In a first time, by the increase in specific surface area with density which leads to higher sorption and thus higher MBV;
- Then, by the decrease in vapor permeability induced by lower inter-particular porosity which reduces the vapor penetration in the composite, and thus the MBV.

The combination of these two parameters leads to identify an optimum value of density, and thus of compaction pressure. In order to validate these hypothesis, complementary investigations regarding vapor permeability and specific surface area are required.

Bourdot et al find the same MBV with hemp-starch composites. Indeed, these composites have an average MBV of 2.63 g/(m².%RH) for an average density of 136 kg/m³ [30]. However, Maalouf et al find a slightly lower MBV with also hemp-starch composites. These composites have an average MBV of 2.46 g/(m².%RH) for an average density of 170 kg/m³ [32].

Collet and al find a lower MBV ranging from 1.94 to 2.15 g/(m².%RH) for the hemp concretes which have a density ranging from 430 to 460 kg/m³ [33]. This difference can be explained by the use of less binder in the case of the composites developed in this paper.

4. Conclusion

The developed composites are produced with hemp shiv and black liquor with several compaction pressures.

The composites density ranges from 128 to 347 kg/m³ at dry state. The density is correlated to the compaction pressure applied during the production. Regarding the total porosity of the composites, it is inversely proportional to their apparent density.

The thermal conductivity increases linearly with density between 62.8 and 93.2 mW/(m.K) at dry state (23 °C; 0%RH) and between 71.0 to 101.5 mW/(m.K) at wet point (23 °C; 50%RH).

All the developed composites are excellent hygric regulators as their MBV ranges from 2.40 to 2.94 g/(m^2.%RH). The MBV evolves with density and shows an optimal value for density around 170 kg/m^3.

Among developed composites, the composite with the lowest density shows high thermal and hygric performances. On the one hand, its thermal conductivity meets the requirements to make it considered as insulating building material. On the other hand, it is an excellent hygric regulator, with MBV of 2.77 g/(m^2.%RH) and has sufficient mechanical properties for its use (self bearing materials).

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