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New thermal insulation fiberboards from cake generated during biorefinery of sunflower whole plant in a twin-screw extruder

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A B S T R A C T

The objective of this study was to manufacture new thermal insulation fiberboards by thermo-pressing. The starting material was a slightly deoiled cake (17.6% oil content), generated during the biorefinery of sunflower (*Helianthus annuus* L.) whole plant in a co-rotating (Clextral BC 45, France) twin-screw extruder. All fiberboards produced were cohesive mixtures of proteins and lignocellulosic fibers, acting respectively as binder and reinforcing fillers in what could be considered as a natural composite. The molding experiments were conducted using a 400 ton capacity heated hydraulic press (Pinette Emidecau Industries, France). The influence of molding conditions on board density, mechanical properties and heat insulation properties was examined. Molding conditions included mold temperature (140–200 °C), pressure applied (150–250 kgf/cm^2) and molding time (40–76 s), and these greatly affected board density and thus the mechanical and heat insulation properties. Board density increased with increasingly extreme molding conditions, rising from 500 to 858 kg/m^3. The mechanical properties increased at the same time (from 52 to 660 kPa for flexural strength at break, from 5.9 to 49.4 MPa for elastic modulus, from 0.5 to 7.7 kJ/m^2 for Charpy impact strength, and from 19.2 to 47.1° for Shore D surface hardness). Conversely, heat insulation properties improved with decreasing board density, and the lowest thermal conductivity (88.5 mW/m K at 25 °C) was obtained with the least dense fiberboard. The latter was produced with a 140 °C mold temperature, a 150 kgf/cm^2 pressure applied and a 40 s molding time. A medium mold temperature (160 °C) was needed to obtain a good compromise between mechanical properties (272 kPa for flexural strength at break, 26.3 MPa for elastic modulus, 3.2 kJ/m^2 for Charpy impact strength, and 37.3° for Shore D surface hardness), and heat insulation properties (99.5 mW/m K for thermal conductivity). The corresponding board density was medium (687 kg/m^3). Because of their promising heat insulation properties, these new fiberboards could be positioned on walls and ceilings for thermal insulation of buildings. The bulk cake also revealed very low thermal conductivity properties (only 65.6 mW/m K at 25 °C) due to its very low bulk density (204 kg/m^3). It could be used as loose fill in the attics of houses.

1. Introduction

Sunflower (*Helianthus annuus* L.) is cultivated for the high oil content of its seeds. Oil represents up to 80% of its economic value. The industrial process for oil production consists of four successive stages: trituration, pressing, extraction of the residual oil using hexane, and refining (Isobe et al., 1992; Rosenthal et al., 1996). Extraction yields are close to 100% with very good oil quality. However, the use of hexane for oil production is an increasingly controversial issue and could be prohibited due to its carcinogenicity (Galvin, 1997). Consequently, numerous solvents have been considered, including water (Rosenthal et al., 1996).

Several researchers have studied the aqueous extraction of sunflower oil (Evon et al., 2007, 2009; Hagenmaier, 1974; Southwell and Harris, 1992) that is an environmentally friendly alternative to solvent extraction. It can be conducted using whole seeds (Evon et al., 2007) or from a press cake (Evon et al., 2009) in a Clextral BC 45 (France) co-penetrating and co-rotating twin-screw extruder that enables efficient mechanical lysis of the cells. Three essential unit operations are carried out in a single step and in continuous mode: conditioning and grinding of the initial material, liquid/solid extraction, and liquid/solid separation. A filter section is positioned on the barrel to collect an extract (filtrate) and a raffinate (cake), separately. However, the introduction of a lignocellulosic residue upstream from the filtration module is essential to enable liquid/solid separation.

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When applied to the whole plant, aqueous fractionation in a twin-screw extruder does not require the addition of a lignocellulosic residue (Evon et al., 2010a) due to the natural abundance of fibers in sunflower stalk (Maréchal and Rigal, 1999), and twin-screw extrusion technology thus appears to be an original and powerful solution for the biorefinery of sunflower whole plant. Under optimal operating conditions, oil extraction yield reaches 57%, and residual oil content in cake is 14.3% of the dry matter. These conditions lead to the co-extraction of proteins but also pectins and hemicelluloses. The corresponding protein extraction yield is 44%, and residual protein content in cake is 7.3% of the dry matter.

Cake moisture content is relatively high (at least 62%), and so it is first dried to facilitate conservation. It has a porous structure, and is mainly composed of lignocellulosic fibers (around 58% of the dry matter), but also cell debris from the kernel breakdown process. Actually, it is lixiviated matter where soluble molecules (proteins, pectins...) and lipids are partly removed, although plant structural molecules are not extracted. The cake is thus suitable for use in animal feeds and for energy production in pellet burning furnaces. Nevertheless, new valorizations of the cake as a mixture of proteins and lignocellulosic fibers can be also considered (Orliac et al., 2002, 2003; Rouilly et al., 2001, 2003, 2006a,b).

The cake's thermo-mechanical behavior has been previously studied (Evon et al., 2010b). DSC (differential scanning calorimetry) measurements indicate that denaturation of cake proteins is almost complete, and the DMTA (dynamic mechanical thermal analysis) spectrum obtained from dried and ground samples reveals a significant peak at high temperature (around 180 °C) which, as already observed with industrial sunflower cake (Geneau, 2006), is associated with the glass transition of proteins. Because the cake is a mixture of proteins and lignocellulosic fibers, it can be considered as a natural composite, and can thus be successfully processed into cohesive, biodegradable and value-added fiberboards by thermo-pressing, proteins and lignocellulosic fibers acting respectively as internal binder and reinforcing fillers (Evon et al., 2010b, 2012a).

The mechanical properties for bending of fiberboards, increase with temperature, pressure and length of time of thermo-pressing (Evon et al., 2010b, 2012a). The highest flexural strength at break (11.5 MPa) and the highest elastic modulus (2.2 GPa) are obtained from a cake with a dry matter residual oil content of 14.5%, and under the following molding conditions: 500 mg/cm^2 for cake quantity, 200 °C for the temperature of the two aluminium plates of the heated hydraulic press, 320 kgf/cm^2 for pressure applied, and 60 s for molding time (Evon et al., 2010b). The fiberboard thickness is only 3.9 mm, its density is quite high (1035 kg/m^3) and DMTA analysis reveals a significant vibratory oscillation peak at low temperature (between −20 and −14 °C), which is attributed to the β-transition of proteins (glass transition of their side chains) (Rouilly et al., 2006b; Zhang et al., 2001). No significant transition is observed between 0 °C and 200 °C, meaning that proteins ensure the agromaterial's cohesion without any phase change in this temperature range. Finally, lignocellulosic fibers' entanglement also act as reinforcement. Because of its promising flexural properties, such a fiberboard would be potentially usable as inter-layer sheet for pallets in the handling and storage industry or for the manufacture of biodegradable, multi-board containers, e.g. composters, crates for vegetable gardening (Evon et al., 2010b).

Another industrial application of fiberboards made from renewable resources is heat insulation of buildings (walls and ceilings), where the main advantages of vegetable fibers are abundance, low cost (the majority are agricultural residues), minimal environmental impact, independence from fossil resources, and their natural capacity for thermal insulation (Saiah et al., 2010). Insulation boards can be made from maize husks and cobs (Paiva et al., 2012; Pinto et al., 2011; Sampathrajan et al., 1992), a mixture of durian peels and coconut coir fibers (Khedari et al., 2003, 2004), cellulose (Nicolajsen, 2005), wastes from tissue paper manufacturing and corn peel (Lertsutthiwong et al., 2008), kenaf fibers (Ardente et al., 2008), flax and hemp fibers (Benfratello et al., 2013; Korjenic et al., 2011; Kymäläinen and Sjöberg, 2008), cotton stalk fibers (Zhou et al., 2010), jute fibers (Korjenic et al., 2011), coconut fibers (Alavez-Ramirez et al., 2012; Panyakaew and Fotios, 2011), sunflower pith (Vandenbossche et al., 2012), date palm fibers (Chikhi et al., 2013), etc.

The thermal conductivity of insulation boards is often influenced by their densities (Benfratello et al., 2013; Chikhi et al., 2013; Khedari et al., 2003, 2004; Lertsutthiwong et al., 2008; Panyakaew and Fotios, 2011; Vandenbossche et al., 2012; Zhou et al., 2010), and low-density materials have the lowest thermal conductivities. As an example, the thermal conductivity of an insulation board from sunflower pith is only 38.5 mW/m K at 25 °C with a board density of 36 kg/m^3 (Vandenbossche et al., 2012). It is comparable to that of conventional insulation materials like expanded polystyrene (37.4 mW/m K with a board density of 50 kg/m^3), rock wool (35.6 mW/m K with a board density of 115 kg/m^3), and glass wool (35.4 mW/m K with a board density of 26 kg/m^3). Thermal conductivity is higher with medium-density materials: 46–68 mW/m K at room temperature for coconut husk insulation boards with board densities of 250–350 kg/m^3 (Panyakaew and Fotios, 2011), 81.5 mW/m K for a cotton stalk fibers insulation board with a board density of 450 kg/m^3 (Zhou et al., 2010), 89.9–107.9 mW/m K for hemp fibers insulation boards with board densities of 369–475 kg/m^3 (Benfratello et al., 2013), 103.6 mW/m K for a coconut coir insulation board with a board density of 540 kg/m^3 (Khedari et al., 2003), and 150 mW/m K for a date palm fibers insulation board with a board density of 754 kg/m^3 (Chikhi et al., 2013). Nevertheless, such boards are viable options for use in building insulation (walls and ceilings).

Heat insulation properties, of fiberboards from cake generated during the biorefinery of sunflower whole plant in a twin-screw extruder are also promising, even if the corresponding board densities are quite high (904–966 kg/m^3) (Evon et al., 2012b). Indeed, thermal conductivity at 25 °C is rather low for the three fiberboards tested, and it decreases from 135.7 to 103.5 mW/m K with an increase in board thickness from 5.4 to 10.2 mm. Thus, the thickest fiberboard (density 917 kg/m^3) gives the best thermal insulation. Fiberboards from such a cake with lower density and higher thickness would perhaps produce a significant improvement in their heat insulation properties.

This study aimed to manufacture by thermo-pressing, new thermal insulation fiberboards with medium density (from 500 to 900 kg/m^3), and high thickness (more than 10 mm and up to 20 mm), from cake generated during the biorefinery of sunflower whole plant in a twin-screw extruder, and to evaluate the influence of molding conditions (temperature, pressure, and time) on their mechanical (flexural properties, Charpy impact strength, and Shore D surface hardness) and heat insulation properties.

2. Materials and methods

2.1. Material

Thermo-mechanical fractionation in the twin-screw extruder was carried out using a batch of sunflower (*Helianthus annuus* L.) whole plant of the oleic type (La Toulousaine de Céréales, France) (Table 1), harvested in September, i.e. at plant maturity. Whole plant was previously dried in a ventilated oven (50 °C, 48 h) and crushed using a hammer mill (Electra VS 1, France) fitted with a 15 mm screen. The moisture content of the powdered plant (batch of around 250 kg) was 7.2 ± 0.1% (French standard NF V 03-903).

Table 1
Chemical composition of the sunflower whole plant used for the experiment and of the cake obtained, after its thermo-mechanical fractionation in the Clextral BC 45 twin-screw extruder (% of the dry matter).

Material	Sunflower whole plant	Cake
Minerals	8.01 (±0.04)	6.97 (±0.01)
Lipids	24.11 (±0.04)	17.57 (±0.06)
Proteins	11.46 (±0.20)	9.19 (±0.18)
Cellulose	24.81 (±0.59)	30.99 (±0.19)
Hemicelluloses	8.10 (±0.30)	12.64 (±0.16)
Lignins	10.27 (±0.16)	15.47 (±0.01)
Water-soluble components	18.55 (±0.40)	12.36 (±0.09)

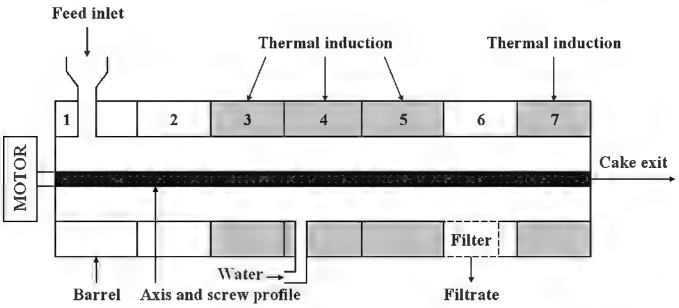

Fig. 1. Schematic modular barrel of the Clextral BC 45 twin-screw extruder used for the thermo-mechanical fractionation of sunflower whole plant ($\theta_c = 80\,°C$).

2.2. *Twin-screw extruder*

Thermo-mechanical fractionation was conducted with a Clextral BC 45 (France) co-rotating twin-screw extruder. The extruder had seven modular barrels, each 200 mm in length, and different twin-screws which had segmental screw elements each 50 and 100 mm long (Fig. 1). Four modules (modules 3, 4, 5 and 7) were heated to 80 °C by thermal induction and cooled by water circulation. A filter section consisting of six hemispherical dishes with 1 mm diameter perforations, was positioned on module 6 to enable the filtrate to be collected. Screw rotation speed (S_S), sunflower whole plant feed rate (Q_S), and barrel temperature (θ_c) were monitored from a control panel.

2.3. *Thermo-mechanical fractionation of sunflower whole plant in the twin-screw extruder*

Sunflower whole plant was fed into the extruder inlet port by a Clextral 40 (France) volumetric screw feeder on the first module. Water was injected using a Clextral DKM K20-2-P32 (France) piston pump at the beginning of module 4 (Fig. 1). The screw profile chosen in this study (Fig. 2) had already been used successfully for the aqueous extraction of oil from sunflower whole plant (Evon et al., 2010a). The trituration zone was located in modules 2 and 3, and consisted of a succession of 10 monolobe paddles and 5 bilobe paddles, 5 cm apart. The extraction zone, situated in modules 4 and 5, was composed of a second series of 5 bilobe paddles. The reverse pitch screws were positioned in module 7, immediately downstream from the filtration module, to press the liquid/solid mixture.

Twin-screw extrusion was performed for 30 min before any sampling to ensure stabilization of operating conditions. Recorded operating conditions included feed rates of sunflower whole plant and water, temperature, plus motor current. On stabilization, the filtrate and the cake were immediately collected for a period of 90 min to avoid any variation in outlet flow rates. Sample collection time was determined with a stopwatch, and carried out once. The filtrate and the cake were then weighed.

The cake was dried in a ventilated oven (60 °C, 24 h) immediately after collection. Particle size distribution inside the cake was then determined using a Retsch AS 300 (Germany) vibratory sieve shaker with a 500 g test sample mass. The sieve acceleration was 1.5×g, and sieving time 10 min.

Oil extraction yield in the twin-screw extruder was calculated according to the following formula:

$$R_L = \frac{(Q_S \times L_S) - (Q_C \times L_C)}{Q_S \times L_S} \times 100 \qquad (1)$$

where R_L is the oil extraction yield based on the residual oil content in the cake (%), Q_S the inlet flow rate of the sunflower whole plant (kg/h), Q_C the flow rate of the cake (kg/h), L_S the oil content in the sunflower whole plant (%), and L_C the oil content in the cake (%).

The protein extraction yield in the twin-screw extruder was calculated according to the following formula:

$$R_P = \frac{(Q_S \times P_S) - (Q_C \times P_C)}{Q_S \times P_S} \times 100 \qquad (2)$$

where R_P is the protein extraction yield based on the residual protein content in the cake (%), P_S the protein content in the sunflower whole plant (%), and P_C the protein content in the cake (%).

The energy consumed by the motor was determined according to the following formulae:

$$P = U \times I \times \cos\varphi \times \frac{S_S}{S_{max}} \qquad (3)$$

where P is the electric power supplied by the motor (W), U the motor's operating voltage ($U = 460$ V), I the current feeding the motor (A), cos φ the theoretical yield of the extruder motor (cos $\varphi = 0.95$), and S_S and S_{max} the test speed and maximum speed (600 rpm) of the rotating screws (rpm), respectively.

$$\mathrm{SME} = \frac{P}{Q_S} \qquad (4)$$

where SME is the specific mechanical energy consumed by the motor per unit weight of sunflower whole plant (W h/kg).

2.4. *Thermo-pressing*

The cake was molded by thermo-pressing inside an aluminium mold, using a 400 ton capacity Pinette Emidecau Industries (France) heated hydraulic press, producing 150 mm × 150 mm square fiberboards. Cake quantity for all experiments was 258 g (i.e. 1147 mg/cm^2). Two fiberboards were manufactured for all the

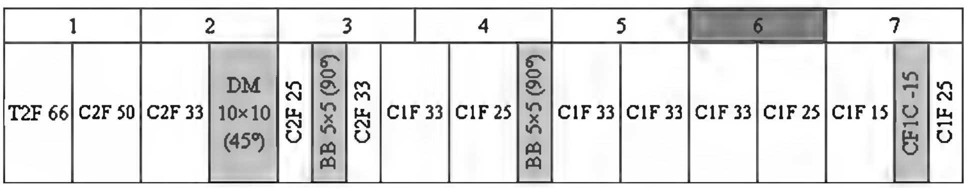

Fig. 2. Screw configuration of the Clextral BC 45 twin-screw extruder used for the thermo-mechanical fractionation of sunflower whole plant. T2F, trapezoidal double-thread screw; C2F, conveying double-thread screw; C1F, conveying simple screw; DM, monolobe paddle-screw; BB, bilobe paddle-screw; CF1C, reverse screw; the numbers following the type of the screw indicate the pitch of T2F, C2F, C1F and CF1C screws, and the length of the DM and BB screws.

Table 2
Operating conditions used for cake production and results of the thermo-mechanical fractionation of the sunflower whole plant in the Clextral BC 45 twin-screw extruder (θ_c = 80 °C).

S_S(rpm)	Q_S(kg/h)	Q_L[a] (kg/h)	Q_C(kg/h)	Q_F[b] (kg/h)	I (A)	P (W)	SME (W h/kg)
62.5	5.7	20.2	12.1	13.7	9.8 (±0.4)	445.5 (±17.2)	78.5 (±3.0)

[a] Q_L is the inlet flow rate of the water.
[b] Q_F is the flow rate of the filtrate.

thermo-pressing conditions tested (including temperature of the aluminium mold, pressure applied, and molding time). Their thickness was measured immediately after thermo-pressing at twelve points, with a 0.01 mm resolution electronic digital sliding caliper, and the mean value (t_0) recorded. Then, fiberboards were equilibrated in a climatic chamber (60% RH, 25 °C) for three weeks before any analyses. A first fiberboard was used to assess mechanical properties for bending, and a second one for measuring: heat insulation properties, Shore D surface hardness, and finally Charpy impact strength.

2.5. Analytical methods

The moisture contents were determined according to French standard NF V 03-903. The mineral contents were determined according to French standard NF V 03-322. The oil contents were determined according to French standard NF V 03-908. The protein contents were determined according to French standard NF V 18-100. An estimation of the three parietal constituents (cellulose, hemicelluloses, and lignins) was made using the ADF-NDF method of Van Soest and Wine (1967, 1968). An estimation of the water-soluble components was made by measuring the mass loss of the test sample after 1 h in boiling water. All determinations were carried out in duplicate.

2.6. Mechanical properties for bending

A JFC 5-kN H5KT (France) universal testing machine fitted with a 100 N load cell was used to assess the flexural properties of the test specimens according to French standard NF EN 310. Such properties included energy-to-break (E), breaking load (F), flexural strength at break (σ_f), and elastic modulus (E_f). The energy-to-break for each specimen, was estimated by the area under the load deformation curve from zero to rupture, calculated using the trapezium method. After fiberboard equilibration in the climatic chamber, test specimens were cut 30 mm wide and their thickness was measured at three points and their length at two points, with a 0.01 mm resolution electronic digital sliding caliper. The mean values of thickness (t) and length (l) were recorded to calculate the volume of specimens, which were all weighed to calculate their mean apparent density (d). The test speed was 3 mm/min and the grip separation was 120 mm. All determinations were carried out four times.

2.7. Charpy impact strength

A Testwell Wolpert 0–40 daN cm (France) Charpy machine was used to assess the impact strength of the unnotched test specimens according to the French standard NF EN ISO 179. It included absorbed energy (W), and resilience (K). The test specimens, 60 mm long and 15 mm wide, were cut after fiberboard equilibration in the climatic chamber. Their thickness was measured at three points with a 0.01 mm resolution electronic digital sliding caliper, and the mean value (t) was recorded to calculate their section. Impact strength measurements were made at 23 °C according to the three point bending technique, and grip separation was 25 mm. All determinations were carried out twelve times.

2.8. Shore D surface hardness

A Bareiss (Germany) durometer was used to assess the Shore D surface hardness of the fiberboards, according to the French standard NF EN ISO 868. All determinations were carried out 48 times (24 times for each side of the fiberboard).

2.9. Heat insulation properties

Thermal conductivity (λ) and thermal resistance (R) of fiberboards were determined at three temperatures (10 °C, 25 °C, and 40 °C) according to the ISO 8302 08-91 standard, using a Lambda-Meßtechnik GmbH Dresden EP 500 (Germany) λ-Meter hot plate apparatus. The area measured was 150 mm × 150 mm, and because of swelling in the climatic chamber, fiberboards had to be cut before measuring to obtain the required dimensions. The difference of temperature between the two plates was 5 K. Measurements were also made with the bulk cake using a polycarbonate box 1 mm thick and 50 mm high. Here, the solid, cake particles were evenly distributed in the box, and a 'box effect' correction made on the thermal conductivity measurements. The cake was equilibrated in the climatic chamber (60% RH, 25 °C) for three weeks before being tested.

3. Results and discussion

3.1. Cake production by twin-screw extrusion

Thermo-mechanical fractionation of whole plant and aqueous extraction of sunflower oil were conducted simultaneously in the Clextral BC 45 twin-screw extruder using a screw configuration previously optimized (Evon et al., 2010a). A filtrate and a cake were collected continuously from the filter section positioned in the barrel on module 6 (Fig. 1). Lipids and water-soluble components, mainly proteins but also pectins and hemicelluloses, were partly extracted during the process. Operating conditions used for cake production and results of the thermo-mechanical fractionation are given in Table 2. Cake moisture content was 67.8 ± 0.2%, and it was dried in a ventilated oven immediately after production to facilitate conservation. It was a powder consisting of inhomogeneous particles (Figs. 3 and 4), with chemical composition shown in Table 1. It was richer in oil (17.6% of the dry matter for oil content) compared with other cakes described in previous studies (from 13.1 to 14.5%) (Evon et al., 2010a,b), leading to an oil extraction yield (R_L, yield based on the residual oil content in the cake) of 45.9 ± 0.2%, and a corresponding protein extraction yield (R_P, yield based on the residual protein content in the cake) of 40.4 ± 1.2%.

The low efficiency of aqueous, whole plant extraction of sunflower oil could be explained by the relatively low (3.6) ratio of water to solid (Q_L/Q_S) in the twin-screw extruder compared to the ratio used for more efficient experiments (up to 4.1) (Evon et al., 2010a,b). It was also due to a low extruder filling ratio (0.09 kg/h rpm), defined as the input flow rate of sunflower whole plant to screw rotation speed ratio (Q_S/S_S), resulting in an unsatisfactory liquid/solid separation of filtrate and cake in the reverse pitch screws. Optimal efficiency of sunflower oil extraction with water (57%), was obtained in a previous study with an extruder

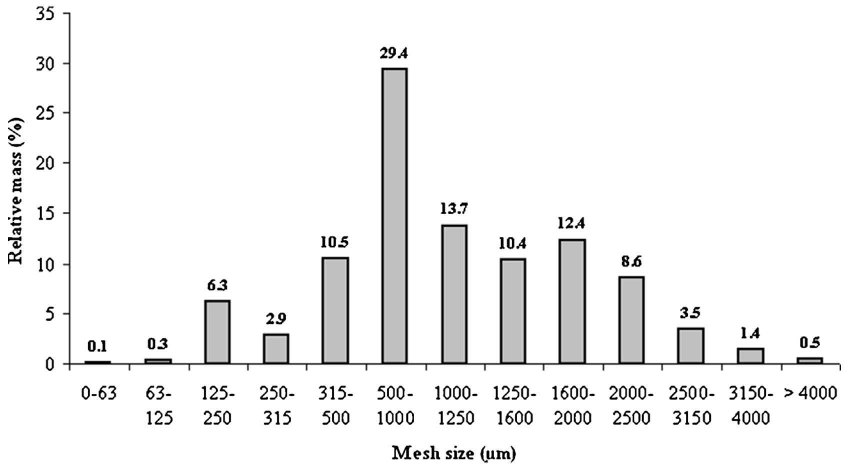

Fig. 3. Particle size distribution in the cake.

filling ratio of 0.17 kg/h rpm (Evon et al., 2010a). A high filling ratio increases compression of matter in the reverse pitch screws, which is essential for efficient separation of liquid and solid phases by filtration.

Because lipids and proteins were partly extracted by water during the process, their residual content in the cake decreased compared to initial values in the whole plant: from 24.1 to 17.6% of the dry matter, and from 11.5 to 9.2% of the dry matter, respectively (Table 1). Logically, the same tendency was also observed with water-soluble components: from 18.6 to 12.4% of the dry matter. Conversely, cellulose and lignins were not extracted because these two biopolymers are insoluble in water. Thus, in parallel, a significant increase relative to their initial values was observed: from 24.8 to 31.0% of the dry matter, and from 10.3 to 15.5% of the dry matter, respectively.

In conclusion, the chemical composition of the cake (Table 1) confirmed that it was a mixture of lignocellulosic fibers and proteins whose denaturation in the twin-screw extruder was almost complete (Evon et al., 2010b). Therefore, it could be considered as a natural composite capable of being transformed into fiberboards by thermo-pressing. Moreover, because the cake oil content was higher than in previous studies, it was reasonable to suppose that residual oil in fiberboards would contribute to make them less water-sensitive and also more durable than truly deoiled thermo-pressed agromaterials, in spite of their overall hydrophilic character. Lastly, the particle size distribution inside the cake revealed the presence of two different populations (Fig. 3). On the one hand, the largest particles (above 1 mm) were mainly composed of lignocellulosic fibers originating essentially from the sunflower stalk and, on the other, the smallest population contained not only smaller fibers but also spherical particles, mean diameter around 500 μm, from the kernel breakdown process. Most of the proteins contained in the cake were present within this population.

3.2. Influence of thermo-pressing conditions on mechanical properties of fiberboards

Five fiberboards were manufactured using different thermo-pressing conditions (Table 3 and photographs in Fig. 4). Conditions included temperature of the aluminium mold, pressure applied, and molding time. The mold temperature, in particular, varied from 140 to 200 °C and was chosen as the main criterion causing variation in fiberboard density (Evon et al., 2010b). The highest temperature (200 °C), producing glass transition of proteins in the cake

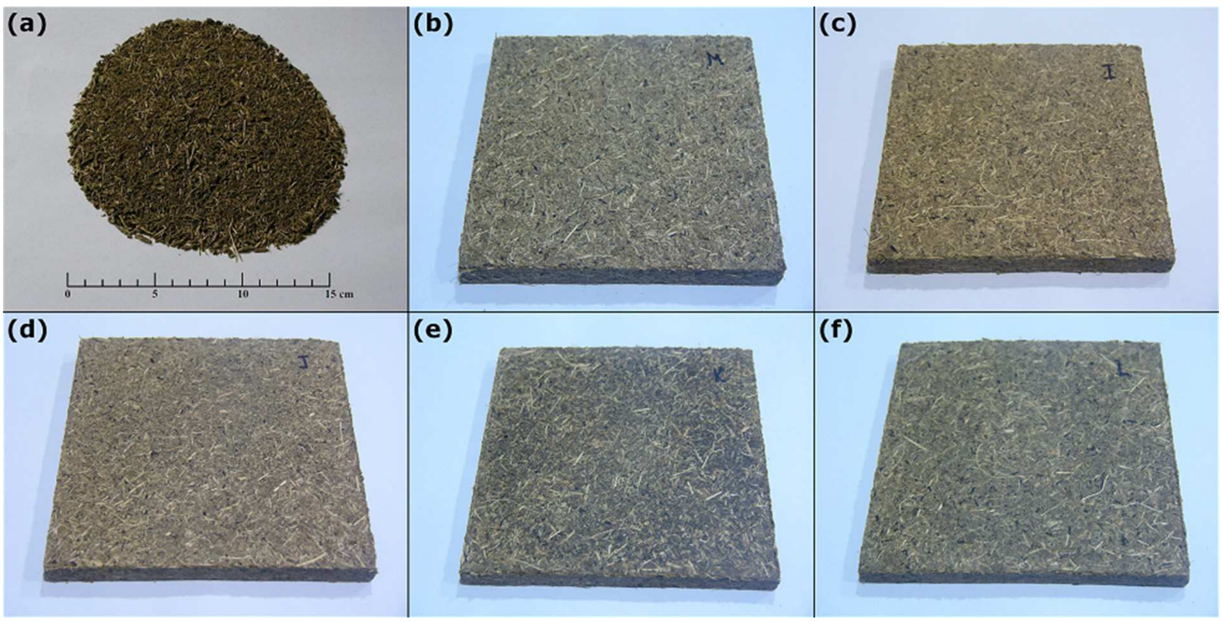

Fig. 4. Photographs of the cake (a) and of the five fiberboards (b, trial 1; c, trial 2; d, trial 3; e, trial 4; f, trial 5).

Table 3
Thermo-pressing conditions for the manufacture of the five fiberboards.

Trial	1	2	3	4	5
Temperature (°C)	140	140	160	180	200
Pressure (kgf/cm^2)	150	250	250	250	250
Time (s)	40	76	76	76	76

during molding, corresponded to that giving the best mechanical properties for bending in a previous study (Evon et al., 2010b). The lowest temperature (140 °C) conversely, was chosen to be near the protein glass transition temperature. A previous study using the DSC technique, showed that this temperature for sunflower proteins was clearly influenced by their water content (Rouilly et al., 2001), with a large drop (from 181 to 5 °C) observed as this protein water content increased from 0 to 26.1% of the dry matter, confirming that water acted as a plasticizer for the proteins. Cake moisture was 2.6 ± 0.1% at molding, and the glass transition temperature of proteins in this case was therefore estimated to be close to 140 °C. Moreover, no temperature higher than 200 °C was tested because thermal degradation of most of the organic compounds in the cake (i.e. lipids, proteins, hemicelluloses, and cellulose) occurs in the 200–375 °C temperature range (Evon et al., 2012b). More specifically, another study indicated that thermal degradation of sunflower proteins from an industrial cake occurred above 250 °C and below 350 °C (Geneau, 2006). A second thermal degradation stage in the cake from whole plant, situated at around 425 °C, is also observed. This corresponds to the thermal degradation of lignins only (Evon et al., 2012b).

The pressure applied varied from 150 to 250 kgf/cm^2. It was lower than for other studies: up to 320 kgf/cm^2 (Evon et al., 2010b), from 320 to 360 kgf/cm^2 (Evon et al., 2012b), and from 250 to 500 kgf/cm^2 (Evon et al., 2012a). The objective for such a choice was to minimize the density of the materials obtained, in order to manufacture fiberboards with medium-density. Molding time varied from 40 to 76 s, which was quite similar to values used in a previous study (30 s and 60 s) (Evon et al., 2010b), and was sufficient for sunflower proteins to reach a rubbery state during molding. In addition, for the fiberboard molded at 200 °C (trial 5), the low molding time (76 s) was also a guarantee against initiation of thermal cake degradation during molding.

The low cake moisture at molding (only 2.6%) was chosen to minimize vapor generation during thermo-pressing and so restrict the risk of defects like blisters inside the fiberboards. Cake quantity for all the experiments was 1147 mg/cm^2, and this led to the manufacture of thicker fiberboards compared with other materials from sunflower cake described in previous studies (Evon et al., 2010b, 2012a,b): at least 10.3 mm thick (and up to 15.2 mm) immediately after thermo-pressing, and at least 13.0 mm (and up to 20.2 mm) after climatic chamber conditioning (60% RH, 25 °C) for three weeks (Table 4).

All the fiberboards were cohesive. As previously observed (Evon et al., 2010a,b, 2012a,b), proteins acted as an internal binder inside fiberboards, and they contributed to ensure cohesion of the agro-material, in addition to the entanglement of lignocellulosic fibers also acting as reinforcement. Conditioning in the climatic chamber was conducted immediately after thermo-pressing in order to assess the mechanical and heat insulation properties of fiberboards from equilibrated materials. It resulted in water sorption, and this increased with a decrease of temperature, pressure, and molding time (Table 4). Indeed, the moisture content of fiberboard from trial 1 was 8.3 ± 0.2% after conditioning, instead of only 6.8 ± 0.1% for the fiberboard from trial 5. This led to the five fiberboards swelling, and the relative increase in their dimensions tended to increase further with decreasing thermo-pressing conditions: +32.5% for trial 1 instead of +27.0% for trial 5 for thickness, and +4.2% for trial 1 instead of +2.6% for trial 5 for length (Table 4).

The density of the equilibrated fiberboards was clearly influenced by the thermo-pressing conditions used, increasing with increasing conditions (Table 4). It varied from 500 to 858 kg/m^3, and the fiberboard with the lowest density (500 kg/m^3) was logically that from trial 1 with the lowest values for mold temperature, pressure applied, and molding time: 140 °C, 150 kgf/cm^2, and 40 s, respectively. This meant that a decrease in the fiberboard density favored water sorption during climatic chamber conditioning, due to its higher porosity. The mechanical properties of fiberboards were also clearly influenced by the thermo-pressing conditions, and could be correlated to their densities. Indeed, the higher the fiberboard density, the higher its mechanical properties (Table 5). Regarding flexural properties, the energy-to-break, the breaking load, the flexural strength at break and the elastic modulus increased progressively from 3.2 to 44.4 mJ, from 3.5 to 18.7 N, from 51.8 to 659.9 kPa, and from 5.9 to 49.4 MPa, respectively, with increasing thermo-pressing temperature, pressure, and molding time. Similarly, for Charpy impact strength, the absorbed energy and the resilience increased from 0.15 to 1.51 J, and from 0.5 to 7.7 kJ/m^2, respectively. Finally, the Shore D surface hardness increased from 19.2 to 47.1°. Thus, the most fragile fiberboard was also the least dense board and was from trial 1.

Since the temperature of the aluminium mold was at least equal to the proteins' glass transition temperature (around 140 °C), this change occurred systematically during molding and they were thus always in a rubbery state. However, because the protein-based resin became more and more viscous with decreasing mold temperature, this meant that the fiber wetting became progressively worse, which could explain why the mechanical properties

Table 4
Swelling, water sorption and density of fiberboards after conditioning in the climatic chamber (60% RH, 25 °C) for three weeks.

Trial	1	2	3	4	5
Dimensions of fiberboards immediately after thermo-pressing					
t_0 (mm)	15.23 (±0.17)	12.77 (±0.12)	12.25 (±0.28)	10.77 (±0.12)	10.26 (±0.11)
l_0 (mm)[a]	150	150	150	150	150
Dimensions and water sorption of fiberboards after conditioning in climatic chamber					
t (mm)[b]	20.18 (±0.61) (+32.5%)	17.09 (±0.76) (+33.8%)	16.00 (±0.56) (+30.5%)	13.85 (±0.46) (+28.6%)	13.03 (±0.46) (+27.0%)
l (mm)[b]	156.33 (±0.61) (+4.2%)	155.75 (±0.42) (+3.8%)	154.08 (±0.20) (+2.7%)	153.75 (±0.42) (+2.5%)	153.92 (±0.20) (+2.6%)
d (kg/m^3)[c]	500 (±23)	625 (±11)	687 (±13)	797 (±27)	858 (±20)
Moisture content (%)[d]	8.33 (±0.17)	7.70 (±0.10)	7.55 (±0.09)	7.03 (±0.14)	6.82 (±0.12)
Water absorbed (% dry matter)	9.08 (±0.20)	8.34 (±0.11)	8.16 (±0.11)	7.56 (±0.16)	7.31 (±0.13)

The percentages in parentheses refer to the relative increase in the fiberboard dimensions (thickness and length) after conditioning in climatic chamber.
[a] l_0 is the length of fiberboards immediately after thermo-pressing.
[b] Measurements were made on specimens prepared for bending tests.
[c] Mean apparent density of fiberboards was calculated after weighing of specimens prepared for bending tests.
[d] Moisture contents were determined after crushing of the equilibrated fiberboards using a Foss Cyclotec 1093 (Denmark) mill fitted with a 1 mm screen.

Table 5
Mechanical properties of the five fiberboards manufactured by thermo-pressing.

Trial	1	2	3	4	5
Flexural properties					
E (mJ)	3.2 (±0.4)	6.7 (±0.6)	15.3 (±1.4)	22.5 (±1.5)	44.4 (±3.9)
F (N)	3.5 (±0.4)	7.7 (±0.3)	11.6 (±0.9)	14.1 (±0.6)	18.7 (±1.0)
σ_f (kPa)	51.8 (±6.1)	157.5 (±5.3)	272.0 (±21.0)	441.7 (±19.5)	659.9 (±36.1)
E_f (MPa)	5.9 (±0.5)	22.1 (±2.4)	26.3 (±2.2)	44.5 (±3.7)	49.4 (±4.9)
Charpy impact strength					
W (J)	0.15 (±0.02)	0.30 (±0.04)	0.78 (±0.09)	0.96 (±0.10)	1.51 (±0.13)
K (kJ/m^2)	0.5 (±0.1)	1.2 (±0.1)	3.2 (±0.4)	4.6 (±0.5)	7.7 (±0.7)
Surface hardness					
Shore D (°)	19.2 (±2.5)	29.6 (±3.0)	37.3 (±3.2)	45.8 (±3.7)	47.1 (±4.4)

Table 6
Thermal conductivity (λ) and thermal resistance (R) at three temperatures (10 °C, 25 °C, and 40 °C) of the bulk cake and of the five fiberboards manufactured by thermo-pressing.

Trial (°C)	Bulk cake[a]	1	2	3	4	5
λ (mW/m K)						
10	60.9	84.4	91.6	94.9	102.3	106.0
25	65.6	88.5	96.4	99.5	106.8	110.5
40	68.3	95.0	104.1	106.6	115.8	120.0
R (m^2 K/W)						
10	0.852	0.239	0.187	0.168	0.135	0.123
25	0.792	0.228	0.177	0.161	0.130	0.118
40	0.760	0.212	0.164	0.150	0.120	0.109

[a] 204 kg/m^3 for bulk density.

of fiberboards diminished as the mold temperature decreased. Thus, when the mold temperature was only 140 °C (trials 1 and 2), proteins became a less effective binder within the material due to insufficient wetting of the fibers, logically leading to the most fragile fiberboards of the study (Table 5). Conversely, fiber wetting was much improved at a 200 °C molding temperature (trial 5). Such thermo-pressing conditions were also associated with the highest values for pressure applied and molding time (250 kgf/cm^2 and 76 s, respectively), leading logically to the densest fiberboard of the study and so to the most mechanically resistant insulation board.

3.3. *Influence of thermo-pressing conditions on heat insulation properties of fiberboards*

The heat insulation properties of the five fiberboards were measured at three temperatures: 10 °C, 25 °C, and 40 °C. As generally observed for thermal insulation solids, the thermal conductivity of the five fiberboards tested increased with increasing temperature, with the thermal resistance logically and simultaneously decreasing (Table 6). The same tendency was also observed for measurements made on the bulk cake. Consequently, the capacity for thermal insulation of all the materials tested, decreased noticeably with increasing temperature. The thermal conductivity of the bulk cake and of the five fiberboards was also clearly influenced by their density, and it increased linearly with increasing density (Fig. 5a). Similar characteristics have been observed on several occasions, in particular for insulation boards made from durian peel and coconut coir (Khedari et al., 2003, 2004), wastes from tissue paper manufacturing and corn peel (Lertsutthiwong et al., 2008), cotton stalk fibers (Zhou et al., 2010), coconut husk and bagasse (Panyakaew and Fotios, 2011), sunflower pith (Vandenbossche et al., 2012), hemp fibers (Benfratello et al., 2013), and date palm fibers (Chikhi et al., 2013). At the same time, the thermal resistance logically decreased (Fig. 5b), and these trends were observed at all three temperatures. However, here we will focus on the 25 °C results, considered as ambient temperature.

Thermal conductivity was rather low for the five fiberboards (Table 6), due to their medium densities (from 500 to 858 kg/m^3) contributing to better heat insulation properties compared with previous results obtained with denser materials (Evon et al., 2012b). Moreover, as already mentioned, it decreased with the decrease in board density. It was 110.5 mW/m K for the most dense (858 kg/m^3) fiberboard and only 88.5 mW/m K for the least dense one (500 kg/m^3). The increase in porosity within the boards thus improved their thermal insulation capacity, and such values were in line with the thermal conductivities of other experimental medium-density insulation boards, made from cotton stalk fibers (Zhou et al., 2010), hemp fibers (Benfratello et al., 2013) or coconut coir (Khedari et al., 2003), and considered as viable options for use in building insulation: 81.5 mW/m K (450 kg/m^3 for board density), 89.9–107.9 mW/m K (369–475 kg/m^3 for board densities) and 103.6 mW/m K (540 kg/m^3 for board density), respectively. At the same time, the thermal resistance varied from 0.118 to 0.228 m^2 K/W (Table 6). In addition, the adsorption of water during climatic chamber conditioning was greater for the less dense fiberboards (Table 4) due to their higher porosity, and thus the heat insulation properties also depended on the moisture content of the equilibrated boards. Indeed, the thermal conductivity decreased with increasing moisture content (Fig. 6a), and the thermal resistance rose at the same time (Fig. 6b).

Because the heat insulation properties of the fiberboards improved with decreasing density (Fig. 5), the fiberboard with the weakest thermal conductivity (trial 1) also corresponded to the most fragile insulation board (Table 5). Conversely, the most conductive fiberboard (trial 5) was also the most mechanically resistant insulation board. Consequently, the medium density (687 kg/m^3) fiberboard from trial 3 (160 °C for mold temperature, 250 kgf/cm^2 for pressure applied, and 76 s for molding time) was a good compromise between mechanical properties (272 kPa for flexural strength at break, 26.3 MPa for elastic modulus, 3.2 kJ/m^2 for Charpy impact strength, and 37.3° for Shore D surface hardness) and those for heat insulation (99.5 mW/m K for thermal conductivity at 25 °C, and 0.161 m^2 K/W for the corresponding thermal resistance). Fitted on walls and ceilings, it could be used for the thermal insulation of buildings. Measurements made on the bulk cake indicated that it was an even better insulation material (only 65.6 mW/m K for thermal conductivity at 25 °C, and 0.792 m^2 K/W for thermal resistance), which was certainly due to its very low bulk density (204 kg/m^3) and to its porous structure (Table 6). It also would be suitable for the thermal insulation of houses as loose fill in attic spaces.

To conclude, the originality of this study derives from two distinct points. Firstly, the optimal fiberboard (trial 3) was a self-bonded composite material like the four others, meaning that there was no necessity to add any external binder to obtain a panel with good cohesion. Secondly, the reinforcing fibers inside these panels originated essentially from sunflower stalks, and these fibers

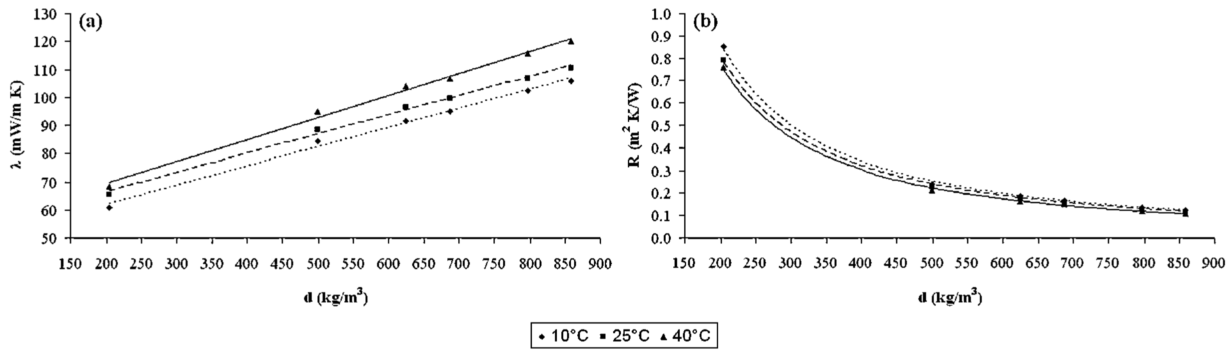

Fig. 5. Thermal conductivity (a) and thermal resistance (b) of the bulk cake and of the five fiberboards manufactured by thermo-pressing, as a function of their density at three temperatures (10 °C, 25 °C, and 40 °C).

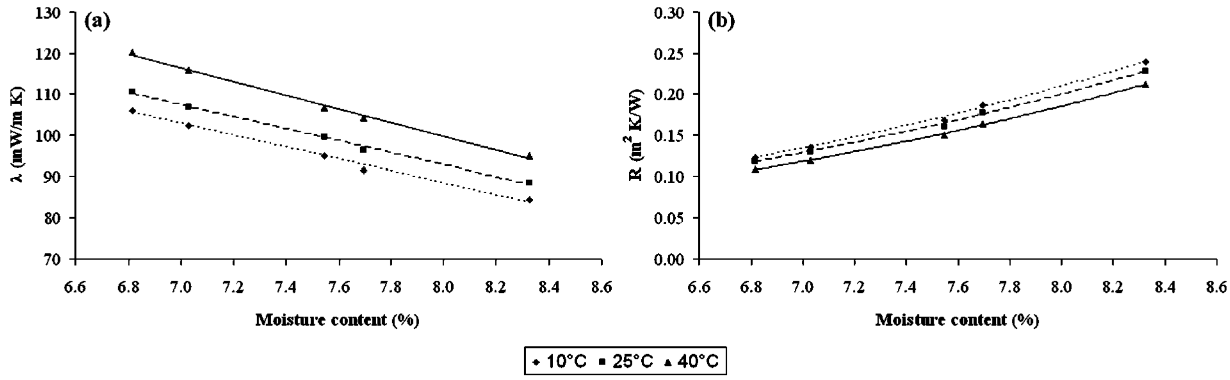

Fig. 6. Thermal conductivity (a) and thermal resistance (b) of the five fiberboards manufactured by thermo-pressing, as a function of their moisture content at three temperatures (10 °C, 25 °C, and 40 °C).

are not now commercially available. Indeed, the sunflower harvest concerns only the seeds and not the by-products of its culture like the stalks and heads. However, their promising ability for both reinforcement of composite materials and thermal insulation could, in the future, justify their harvest in the field at the same time as the seeds. Which in turn could generate an additional source of income for farmers.

4. Conclusion

New thermal insulation fiberboards were manufactured using a heated hydraulic press, from a cake generated during the biorefinery of sunflower whole plant in a twin-screw extruder. All fiberboards were cohesive. Proteins acted as an internal binder, and entanglement of lignocellulosic fibers also acted as reinforcement. The thermo-pressing conditions had an important influence on board density and on mechanical and heat insulation properties. The density of the insulation materials varied from 500 to 858 kg/m^3, and the least dense fiberboard was produced under the lowest values of mold temperature (140 °C), pressure applied (150 kgf/cm^2), and molding time (40 s). The heat insulation properties improved with decreasing fiberboard density, and thermal conductivity of the least dense fiberboard was only 88.5 mW/m K at 25 °C. However, such a board was also the most fragile. A 160 °C mold temperature gave a good compromise between mechanical properties (272 kPa for flexural strength at break, 26.3 MPa for elastic modulus, 3.2 kJ/m^2 for Charpy impact strength, and 37.3° for Shore D surface hardness) and heat insulation properties (99.5 mW/m K for thermal conductivity). Positioned on walls and ceilings, it could be used for the thermal insulation of buildings. The heat insulation capacity of the bulk cake was even better (only 65.6 mW/m K). It also would be suitable for the thermal insulation of houses when used as loose fill in attic spaces.

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