

## Thermo-pressing of cake meal from sunflower whole plant, one only operation for two actions: expression of residual oil and molding of biodegradable agromaterials

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### ABSTRACT

• Sunflower (*Helianthus annuus* L.) is cultivated for its seeds' high oil content. Water is an interesting alternative medium for sunflower oil extraction. Aqueous extraction of oil can be conducted in a twin-screw extruder using the whole seeds (Evon et al., Ind. Crops Prod. 26:351, 2007) or using a press cake (Evon et al., Ind. Crops Prod. 29:455, 2009). When it is conducted using the whole plant, wringing out the resulting mix is favored because of the natural abundance of fibers in stalk (Evon et al., Ol. Corps gras Li. 17:404, 2010). A filtrate and a cake are produced simultaneously, and twin-screw extrusion appears to be a powerful tool for the biorefinery of sunflower whole plant. Oil extraction yield is 57% in the best operating conditions, and residual oil content in cake is 14.3% dry matter. As cake is a mixture of fibers and proteins, it can be considered as a natural composite that is processed successfully into biodegradable agromaterials by thermo-pressing (Evon et al., Ol. Corps gras Li. 17:404, 2010; Evon et al., Adv. Mater. Res. 112:63, 2010). Thermo-pressing is not only a molding operation to manufacture cohesive fiberboards. It also consists in increasing the oil extraction efficiency (Evon et al., Ol. Corps gras Li. 17:404, 2010; Evon et al., Adv. Mater. Res. 112:63, 2010). Indeed, part of residual oil in cake is expressed during molding through the vents of mold, due to the pressure applied. The objective of the study was to evaluate the influence of thermo-pressing conditions on oil expression yield during molding and on flexural properties of fiberboards.

• An experimental design with three variables was realized: from 250 to 500 kgf/cm<sup>2</sup> for pressure applied (in 5 levels), from 60 to 300 s for molding time (in 7 levels), and from 600 to 1200 mg/cm<sup>2</sup> for cake quantity (in 3 levels). Temperature of the aluminium mold positioned between the two plates of the MAPA 50 (PEI, France) heated hydraulic press was 200°C. Cake used for molding was produced in a Clextral BC 45 (France) twin-screw extruder. It was slightly dehulled (17.6% dry matter for residual oil content), leading to an oil extraction yield of 46.1% (yield based on the residual oil content in cake).

• Oil expression yield during molding increases with the increase of pressure applied, and especially with the increase of molding time. At the same time, it is not so much influenced by the modification of cake quantity. Highest oil expression yield is 58.8% in proportion to the oil that the cake contains, leading to a total oil yield (oil extracted by water in twin-screw extruder, and oil expressed during molding) of 77.8% in proportion to the oil that the sunflower whole plant contains. It is associated with the next thermo-pressing conditions: 469 kgf/cm<sup>2</sup> for pressure applied, 300 s for molding time, and 697 mg/cm<sup>2</sup> for cake quantity. Flexural properties of the corresponding fiberboard are 8.1 MPa for flexural strength at break, and 1778 MPa for elastic modulus. Its thickness is 5.40 mm, leading to a mean apparent density of 1.25. Such flexural strength at break is a bit lower (-25%) than the one of the most resistant fiberboard (10.8 MPa), manufactured from the next thermo-pressing conditions: 250 kgf/cm<sup>2</sup> for pressure applied, 300 s for molding time, and 807 mg/cm<sup>2</sup> for cake quantity. For such conditions, oil expression yield is 48.0% in proportion to the oil that the cake contains, leading to a total oil yield close (-8%) to the highest yield obtained (71.9% in proportion to the oil that the sunflower whole plant contains instead of 77.8%).

• Thermo-pressing of cake from sunflower whole plant leads to two actions in a single step: the expression of part of residual oil in cake that contributes to the improvement of the oil extraction efficiency, and the molding of biodegradable fiberboards. Their flexural properties are promising. Moreover, because residual oil content in fiberboards is at least 8.0% dry matter, they are not too water-sensitive (i.e. more durable than other thermo-pressed agromaterials).

• Such fiberboards are value-added agromaterials that may have direct industrial applications. Indeed, they would be potentially usable as inter-layer sheets for pallets, for the manufacturing of biodegradable containers (composters, crates), or for their heat insulation properties in building trade.

**Key words:** Biodegradable agromaterials – oil expression – oil extraction – sunflower whole plant – thermo-pressing – twin-screw extrusion

## INTRODUCTION

Sunflower is cultivated for its seeds' high oil content. Oil represents up to 80% of its economic value. The industrial processes for oil production consist of three successive stages: pressing, extraction of the residual oil using hexane and refining (Rosenthal et al., 1996). The 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. Consequently, numerous solvents have been considered, including water (Rosenthal et al., 1996). Several researchers have studied the aqueous extraction of sunflower oil that is an environment-friendly alternative to the solvent extraction. It can be conducted using the whole seeds (Evon et al., 2007) or using a press cake (Evon et al., 2009) in a twin-screw extruder that enables an efficient mechanical lysis of the cells. Twin-screw extruder is used to carry out continuously three essential unit operations: conditioning and grinding of the starting material, liquid/solid extraction and liquid/solid separation. A filter section is outfitted along the barrel to collect separately an extract (filtrate) and a raffinate (cake). However, the introduction of a lignocellulosic residue upstream from the filtration module is essential to enable the liquid/solid separation.

When it is applied to whole plant, aqueous fractionation in 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, and twin-screw extrusion appears to be a powerful solution for the biorefinery of sunflower whole plant. In best operating conditions, oil extraction yield is 57%, and residual oil content in cake is 14.3% dry matter. These conditions lead to the co-extraction of proteins but also pectins and hemicelluloses. The oil is extracted in the form of two different oil-in-water emulsions. These hydrophobic phases are stabilized by phospholipids and proteins at interface. These emulsions may have direct industrial applications for non food uses (Evon et al., 2010a). An aqueous extract containing part of the water-soluble constituents from whole plant is also generated. It is much diluted, and it would be potentially recyclable for aqueous extraction in the twin-screw extruder.

The cake is relatively moist (at least 62%), and it is first dried to make easier its conservation. It is largely composed of lignocellulosic fibers (around 58% dry matter). Actually, the cake is a lixiviated matter where soluble molecules (proteins, pectins...) and lipids are partly removed. At the same time, molecules from plant skeleton are not extracted. The cake is suitable for use in animal feeds and for energy production in pellets burning furnaces. Nevertheless, new valorisations of the cake as a mixture of lignocellulosic fibers and proteins can be also considered. As a natural composite, it is successfully processed into agromaterials by thermo-pressing (Evon et al., 2010b). Proteins ensure the agromaterial cohesion, and lignocellulosic fibers entanglement also acts like reinforcement. The flexural properties of the panels increase simultaneously with temperature, pressure and time chosen for molding operation. When thermo-pressing is conducted using a cake with a residual oil content of 14.5% dry matter, highest flexural strength at break (11.5 MPa) and highest elastic modulus (2.2 GPa) are obtained from next molding conditions: 200°C for temperature of the two aluminium plates of the heated hydraulic press, 320 kgf/cm<sup>2</sup> for pressure applied, 60 s for molding time, and 500 mg/cm<sup>2</sup> for cake quantity.

This study aimed to evaluate the influence of thermo-pressing conditions (pressure applied during molding, molding time, and cake quantity) on oil expression yield during molding and on flexural properties of fiberboards manufactured from a cake slightly dehulled, and inside a mold equipped with vents to allow the expression of residual oil during molding.

## MATERIALS AND METHODS

Thermo-mechanical fractionation in the twin-screw extruder was carried out using a batch of sunflower whole plant from oleic type (Table 1). Whole plant was previously dried in a ventilation oven (50°C, 48 h) and crushed using a hammer mill (Electra VS 1, France) fitted with a 15 mm screen. Its moisture content was 7.25±0.02% (standard NF V 03-903). The extruder used was a Clextral BC 45 (France) co-rotating twin-screw extruder. It had seven modular barrels, each 200 mm in length. Four modules (modules 3, 4, 5 and 7) were heated to 80°C by thermal induction and cooled by water circulation. A filter section was outfitted 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 ( $\theta_c$ ) were monitored from a control panel. Sunflower whole plant was fed into the extruder inlet port by a volumetric screw feeder located in module 1. Water was injected at the start of module 4. The screw profile chosen was already used for the aqueous extraction of oil from sunflower whole plant (Evon et al., 2010a). The extruder was left to function for 30 minutes before any sampling to ensure the stabilization of the operating conditions. Upon achieving steady operation, the filtrate and the cake were collected during 30 minutes to avoid any variability of the outlet flow rates. Sample collection was carried out once. The filtrate and the cake were weighed.

The oil extraction yield was calculated according to the following formula:

$$R_{L1} = \frac{(Q_S \times L_S) - (Q_C \times L_C)}{Q_S \times L_S} \times 100 \quad (1)$$

$R_{L1}$  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  $L_C$  is the oil content in the cake (%).

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

Material	Method	Sunflower whole plant	Cake
Minerals	NF V 03-322	8.01±0.04	6.97±0.01
Lipids	NF V 03-908	24.11±0.04	17.57±0.06
Proteins	NF V 18-100	11.46±0.20	9.19±0.18
Cellulose	ADF-NDF	24.81±0.59	30.99±0.19
Hemicelluloses	ADF-NDF	8.10±0.30	12.64±0.16
Lignins	ADF-NDF	10.27±0.16	15.47±0.01

The cake was molded by thermo-pressing using a heated hydraulic press (PEI, France) with 400 tons capacity. The aluminium mold used was equipped with vents to allow the expression during molding of residual oil from cake. The fiberboards produced were 150 mm × 150 mm squares. An experimental design with three variables was realized to evaluate the influence of thermo-pressing conditions on oil expression yield during molding and on flexural properties of fiberboards manufactured from this cake (Table 2). Such conditions include pressure applied during molding, molding time, and cake quantity. Temperature of the aluminium mold was 200°C. It was the same as the one that led to the highest flexural properties in a previous study (Evon et al., 2010b), allowing the glass transition of proteins in the cake during molding. The cake moisture at molding was 2.89±0.11% (standard NF V 03-903).

**Table 2.** Thermo-pressing conditions used with the heated hydraulic press for the manufacturing of the sixteen fiberboards (200°C for the temperature of the aluminium mold).

Trial	$X_1$	Pressure applied (kgf/cm <sup>2</sup> )	$X_2$	Molding time (s)	$X_3$	Cake quantity (mg/cm <sup>2</sup> )
1	1.000	500	0.000	180	0.000	900
2	-1.000	250	0.000	180	0.000	900
3	0.500	438	0.866	284	0.000	900
4	-0.500	313	-0.866	76	0.000	900
5	0.500	438	-0.866	76	0.000	900
6	-0.500	313	0.866	284	0.000	900
7	0.500	438	0.289	215	0.816	1145
8	-0.500	313	-0.289	145	-0.816	655
9	0.500	438	-0.289	145	-0.816	655
10	0.000	375	0.577	249	-0.816	655
11	-0.500	313	0.289	215	0.816	1145
12	0.000	375	-0.577	111	0.816	1145
13	0.000	375	0.000	180	0.000	900
14	0.000	375	0.000	180	0.000	900
15	0.000	375	0.000	180	0.000	900
16	0.000	375	0.000	180	0.000	900

The oil expression yield during molding was calculated according to the following formula:

$$R_{L2} = \frac{(m_C \times L_C) - (m_{FB} \times L_{FB})}{m_C \times L_C} \times 100 \quad (2)$$

$R_{L2}$  is the oil expression yield during molding in proportion to the oil that the cake contains (%),  $m_C$  the mass of cake used for thermo-pressing (g),  $m_{FB}$  the mass of fiberboard (g), and  $L_{FB}$  is the oil content in the fiberboard (%). The oil expression yield during molding can be also expressed in proportion to the oil that the sunflower whole plant contains ( $R_{L2}'$ , %).

The total oil yield was calculated according to the following formula:

$$R_{LT} = R_{L1} + R_{L2}' = R_{L1} + \left( R_{L2} \times \frac{100 - R_{L1}}{100} \right) = \left( R_{L1} \times \frac{100 - R_{L2}}{100} \right) + R_{L2} \quad (3)$$

$R_{LT}$  is the total oil yield (oil extracted with water in the twin-screw extruder, and oil expressed during molding) in proportion to the oil that the sunflower whole plant contains (%).

A 5-kN H5KT (JFC, France) universal testing machine fitted with a 500 N load cell was used to assess the flexural properties of the test specimens (French standard NF EN 310), including breaking load (F), flexural strength at break ( $\sigma_f$ ), and elastic modulus ( $E_f$ ). The test specimens were 150 mm long and 30 mm wide. Their thickness was measured at three points with an electronic digital sliding caliper having a 0.01 mm resolution, and the mean value ( $t$ ) was recorded to calculate their volume and section. All specimens were weighed to calculate their mean apparent density ( $d$ ). The test speed was 3 mm/min and the grip separation was 100 mm. Test specimens were cut, and equilibrated in climatic chamber (60% RH, 25°C) during three weeks before being tested. All determinations were carried out four times.

## RESULTS AND DISCUSSION

Thermo-mechanical fractionation of whole plant and aqueous extraction of sunflower oil were conducted simultaneously in the twin-screw extruder. The screw rotation speed, the inlet flow rate of the sunflower whole plant, and the inlet flow rate of the water were 62.5 rpm, 5.7 kg/h, and 20.4 kg/h, respectively. A filtrate and a cake were collected continuously. The flow rate of the filtrate was 13.8 kg/h, and the flow rate of the cake was 12.3 kg/h. Lipids and water-soluble components, mainly proteins, were partly extracted during the process. The cake moisture was  $68.29 \pm 0.35\%$  (standard NF V 03-903). The cake was dried in a ventilation oven (60°C, 24 h) immediately after its production to make easier its conservation. Its chemical composition is mentioned in Table 1. It was slightly dehulled (17.6% dry matter for its residual oil content) compared with other cakes described in previous studies (Evon et al., 2010a, 2010b). This led to an oil extraction yield of only  $46.1 \pm 0.2\%$  (yield based on the residual oil content in the cake). Because lipids and proteins were partly extracted by water, their residual contents in the cake decreased logically: from 24.1 to 17.6% dry matter, and from 11.5 to 9.2% dry matter, respectively. On the contrary, cellulose and lignins were not extracted. Thus, a significant increase of their contents was observed at the same time: from 24.8 to 31.0% dry matter, and from 10.3 to 15.5% dry matter, respectively. Chemical composition of the cake confirmed that it was a mixture of fibers and proteins, meaning that it could be considered as a natural composite to be transformed into fiberboards by thermo-pressing.

The sixteen fiberboards manufactured were cohesive. As previously observed (Evon et al., 2010b), proteins acted as an internal binder inside fiberboards, and they contributed to ensure the agromaterial cohesion. At the same time, lignocellulosic fibers entanglement also acted like reinforcement. For all fiberboards, part of residual oil in the cake was expressed during molding, due to the pressure applied. This led to the decrease of residual oil content in fiberboards (until 8.8% dry matter), and to the increase of total oil yield (at least 56.9% in proportion to the oil that the sunflower whole plant contained instead of 46.1% for oil extraction yield in the twin-screw extruder, and until 75.6%) (Table 3). Logically, oil yields increased with the decrease of the residual oil content in fiberboards, meaning that the poorest fiberboards in lipids were associated with the best oil expression efficiencies. The mean apparent density of fiberboards varied from 1.04 to 1.22 (Table 4). At the same time, their flexural strength at break varied from 1.9 to 9.6 MPa, and their elastic modulus varied from 166 to 1494 MPa (Table 4). The flexural strength at break and the elastic modulus tended to increase with the increase of the mean apparent density, and the highest values of the flexural strength at break were associated with the highest values of the elastic modulus. Thus, the denser fiberboards were the most resistant and the most rigid agromaterials. The flexural properties of fiberboards tended also to increase with the increase of oil expression yield.

The best-fit second-order responses to describe the oil content ( $L_{FB}$ ) in the fiberboard, the corresponding oil expression yield in proportion to the oil that the cake contained ( $R_{L2}$ ), and the corresponding total oil yield ( $R_{LT}$ ) were given in Equations 4 to 6 (0.94, 0.95, and 0.95 for R squared values, respectively):

$$L_{FB} = 10.44 - (0.43 \times X_1) - (3.32 \times X_2) + (0.08 \times X_3) - (0.05 \times X_1 \times X_2) + (1.23 \times X_1 \times X_3) + (0.96 \times X_2 \times X_3) + (0.69 \times X_1 \times X_1) + (1.47 \times X_2 \times X_2) + (1.44 \times X_3 \times X_3) \quad (4)$$

$$R_{L2} = 45.77 + (2.45 \times X_1) + (19.67 \times X_2) - (0.02 \times X_3) - (0.31 \times X_1 \times X_2) - (7.24 \times X_1 \times X_3) - (5.79 \times X_2 \times X_3) - (4.70 \times X_1 \times X_1) - (9.38 \times X_2 \times X_2) - (8.32 \times X_3 \times X_3) \quad (5)$$

$$R_{LT} = 70.75 + (1.32 \times X_1) + (10.61 \times X_2) - (0.01 \times X_3) - (0.17 \times X_1 \times X_2) - (3.91 \times X_1 \times X_3) - (3.12 \times X_2 \times X_3) - (2.53 \times X_1 \times X_1) - (5.06 \times X_2 \times X_2) - (4.49 \times X_3 \times X_3) \quad (6)$$

**Table 3.** Quantification of the oil expressed during molding for the sixteen fiberboards.

Trial	m <sub>FB</sub> (g)	H <sub>FB</sub> <sup>1</sup> (%)	L <sub>FB</sub> (% dry matter)	R <sub>L2</sub> (%)	R <sub>L2</sub> ' (%)	R <sub>LT</sub> (%)
1	197.9	7.01±0.02	11.20±0.08	40.5±0.4	21.8±0.2	67.9±0.4
2	195.6	6.68±0.01	11.07±0.12	41.7±0.6	22.5±0.3	68.5±0.5
3	190.5	6.64±0.02	8.82±0.07	54.7±0.4	29.5±0.2	75.6±0.4
4	204.9	7.35±0.01	14.57±0.14	20.2±0.8	10.9±0.4	56.9±0.6
5	202.3	7.39±0.05	14.27±0.20	22.8±1.1	12.3±0.6	58.4±0.8
6	191.9	7.03±0.09	9.21±0.01	52.5±0.1	28.3±0.0	74.4±0.2
7	246.5	7.07±0.01	10.60±0.02	44.8±0.1	24.2±0.0	70.3±0.2
8	146.5	7.38±0.01	14.24±0.10	23.0±0.6	12.4±0.3	58.5±0.5
9	143.7	7.34±0.03	11.76±0.04	37.6±0.2	20.3±0.1	66.3±0.3
10	139.4	7.42±0.02	9.11±0.03	53.1±0.1	28.7±0.1	74.7±0.2
11	246.8	7.24±0.02	11.10±0.05	42.2±0.2	22.8±0.1	68.9±0.3
12	253.4	7.38±0.08	13.78±0.03	26.5±0.2	14.3±0.1	60.4±0.3
13	195.5	7.33±0.05	11.22±0.04	41.3±0.2	22.3±0.1	68.4±0.3
14	194.3	7.22±0.02	10.52±0.03	45.3±0.2	24.4±0.1	70.5±0.3
15	191.8	7.13±0.07	10.08±0.07	48.2±0.4	26.0±0.2	72.0±0.4
16	192.8	6.83±0.03	9.96±0.06	48.3±0.3	26.1±0.2	72.1±0.4

<sup>1</sup> Fiberboards were equilibrated in a climatic chamber (60% RH, 25°C) during three weeks before moisture measurements.

**Table 4.** Flexural properties of the sixteen fiberboards.

Trial	t (mm)	d	F (N)	σ <sub>f</sub> (MPa)	E <sub>f</sub> (MPa)
1	7.64±0.56	1.16±0.02	87.8±1.3	7.5±0.1	1006±6
2	7.55±0.40	1.15±0.02	107.6±1.6	9.4±0.1	1261±17
3	7.11±0.40	1.22±0.02	93.6±4.2	9.3±0.4	1494±74
4	8.30±0.42	1.08±0.04	46.9±13.2	3.4±1.0	370±140
5	8.48±0.48	1.05±0.04	49.8±16.6	3.5±1.2	378±61
6	7.30±0.38	1.18±0.01	102.6±6.3	9.6±0.6	1294±53
7	9.60±0.57	1.14±0.02	109.3±8.6	5.9±0.5	644±48
8	5.95±0.43	1.13±0.02	33.9±8.4	4.8±1.2	464±120
9	5.66±0.44	1.15±0.03	34.9±4.0	5.4±0.6	783±209
10	5.26±0.44	1.21±0.01	40.5±4.7	7.3±0.9	1437±77
11	9.55±0.39	1.11±0.03	73.1±14.0	4.0±0.8	347±137
12	10.36±0.43	1.04±0.02	40.4±5.8	1.9±0.3	166±24
13	7.56±0.36	1.16±0.01	85.1±12.1	7.5±1.1	1014±125
14	7.58±0.27	1.15±0.02	86.2±2.6	7.5±0.2	1024±82
15	7.22±0.52	1.18±0.01	81.5±7.9	7.8±0.8	1234±22
16	7.29±0.46	1.18±0.01	81.3±6.4	7.6±0.6	1221±96

The best-fit second-order responses to describe the thickness (t), the mean apparent density (d), the flexural strength at break (σ<sub>f</sub>), and the elastic modulus (E<sub>f</sub>) of the fiberboard were given in Equations 7 to 10 (1.00, 0.97, 0.96, and 0.94 for R squared values, respectively):

$$t = 7.41 - (0.01 \times X_1) - (0.70 \times X_2) + (2.58 \times X_3) - (0.22 \times X_1 \times X_2) + (0.29 \times X_1 \times X_3) - (0.08 \times X_2 \times X_3) + (0.18 \times X_1 \times X_1) + (0.45 \times X_2 \times X_2) + (0.32 \times X_3 \times X_3) \quad (7)$$

$$d = 1.17 + (0.01 \times X_1) + (0.08 \times X_2) - (0.04 \times X_3) + (0.04 \times X_1 \times X_2) - (0.01 \times X_1 \times X_3) - (0.01 \times X_1 \times X_1) - (0.04 \times X_2 \times X_2) - (0.04 \times X_3 \times X_3) \quad (8)$$

$$\sigma_f = 7.60 - (0.19 \times X_1) + (3.36 \times X_2) - (1.17 \times X_3) - (0.24 \times X_1 \times X_2) + (0.86 \times X_1 \times X_3) - (0.33 \times X_2 \times X_3) + (0.88 \times X_1 \times X_1) - (1.84 \times X_2 \times X_2) - (3.83 \times X_3 \times X_3) \quad (9)$$

$$E_f = 1123.16 + (39.15 \times X_1) + (606.77 \times X_2) - (312.24 \times X_3) + (110.80 \times X_1 \times X_2) - (52.11 \times X_1 \times X_3) - (460.03 \times X_2 \times X_3) + (10.35 \times X_1 \times X_1) - (322.55 \times X_2 \times X_2) - (647.05 \times X_3 \times X_3) \quad (10)$$

The influence of thermo-pressing conditions on oil content in fiberboard is illustrated by the second-order response mentioned in Equation 4. Its statistical analyze revealed that its most significant coefficients were a<sub>1</sub>, a<sub>13</sub>, a<sub>11</sub>, a<sub>22</sub> and a<sub>33</sub> coefficients, and especially a<sub>2</sub> coefficient. Pressure applied and especially

molding time had an effect on the oil content in fiberboard, and their increase improved the impoverishment in lipids of fiberboard, due notably to  $a_1$  and  $a_2$  coefficients that were negative. Logically, the most significant coefficients in Equations 5 and 6 were the same (i.e.  $a_1$ ,  $a_{13}$ ,  $a_{11}$ ,  $a_{22}$  and  $a_{33}$  coefficients, and especially  $a_2$  coefficient). Because  $a_1$  and  $a_2$  coefficients in Equations 5 and 6 were positive, the oil expression yield in proportion to the oil that the cake contained ( $R_{L2}$ ) and the total oil yield ( $R_{LT}$ ) increased with the increase of pressure applied, and especially with the increase of molding time. At the same time, they were not so much influenced by the modification of cake quantity. The calculation of the optimal thermo-pressing conditions using Equation 5 revealed that the best oil expression yield was obtained under next conditions: 469 kgf/cm<sup>2</sup> for pressure applied (i.e.  $X_1 = 0.75$ ), 300 s for molding time (i.e.  $X_2 = 1.00$ ), and 697 mg/cm<sup>2</sup> for cake quantity (i.e.  $X_3 = -0.68$ ). This led to an oil expression yield of 58.8% in proportion to the oil that the cake contained, calculated according to Equation 5, and to a total oil yield (oil extracted by water in twin-screw extruder, and oil expressed during molding) of 77.8% in proportion to the oil that the sunflower whole plant contained, calculated according to Equation 6. The oil content in fiberboard was then 8.0% dry matter, calculated according to Equation 4. Flexural properties of the resulting fiberboard were calculated according to Equations 9 and 10: 8.1 MPa for flexural strength at break, and 1778 MPa for elastic modulus. Its thickness was 5.40 mm, calculated according to Equation 7, and its mean apparent density was 1.25, calculated according to Equation 8.

The calculation of the optimal thermo-pressing conditions using Equation 9 revealed that the most resistant fiberboard was manufactured under next conditions: 250 kgf/cm<sup>2</sup> for pressure applied (i.e.  $X_1 = -1.00$ ), 300 s for molding time (i.e.  $X_2 = 1.00$ ), and 807 mg/cm<sup>2</sup> for cake quantity (i.e.  $X_3 = -0.31$ ). This led to a flexural strength at break of 10.8 MPa, calculated according to Equation 9. It was a bit higher (+ 33%) than the one of the fiberboard that was associated with the best oil expression yield (8.1 MPa). The elastic modulus of this most resistant fiberboard was 1428 MPa, calculated according to Equation 10. Its thickness was 6.91 mm, calculated according to Equation 7, and its mean apparent density was 1.16, calculated according to Equation 8. Its oil content was 10.0% dry matter, calculated according to Equation 4, and oil expression yield was then 48.0% in proportion to the oil that the cake contained, calculated according to Equation 5. This led to a total oil yield of 71.9 % in proportion to the oil that the sunflower whole plant contained, calculated according to Equation 6, close (-8 %) to the highest value obtained (77.8%).

Oil expressed during molding could be collected. Firstly, its filtration would eliminate small solid particles that were driven through the vents of the mold during thermo-pressing. Then, its refining would make it a vegetable oil usable for human feeding. Two other applications can be also considered for such refined oil: its use as a biolubricant or its transformation into biodiesel after transesterification of triglycerides with methanol to produce fatty acid methyl esters (FAME). Even if the pressure applied during molding led to a partial oil expression, residual oil content in fiberboards was not negligible (Table 3). It was at least 8.0% dry matter (case of the best oil expression yield), and it was more than 10% dry matter in most cases (until 14.6% dry matter). Thus, in spite of their global hydrophilic character, residual oil in fiberboards will contribute to make them less water-sensitive and more durable than dehulled thermo-pressed agromaterials. Such fiberboards would be potentially usable as inter-layer sheets for pallets, for the manufacturing of biodegradable containers (composters, crates for vegetable gardening) by assembly of fiberboards, or as heat insulating materials in building trade.

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