The aqueous extraction of sunflower oil from whole plant in twin-screw extruder, a first step for the manufacturing of biodegradable agromaterials by thermo-pressing

Philippe Evon, Virginie Vandenbossche, Pierre-Yves P.-Y. Pontalier, Luc Rigal

- *Topic:* *"By-products and utilization".*

The aqueous extraction of sunflower oil from whole plant in twin-screw extruder, a first step for the manufacturing of biodegradable agromaterials by thermo-pressing

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Keywords: Sunflower whole plant, twin-screw extruder, aqueous extraction process, oil and extraction, proteins and extraction, thermo-pressing, biodegradable agromaterials.

Twin-screw extrusion is an original solution for the biorefinery of sunflower whole plant according to an aqueous extraction process. In best operating conditions, oil yield is 57% and residual oil content in the cake meal is 14%. Oil is extracted in the form of two oil-in-water emulsions stabilized by phospholipids and proteins at interface.
The cake meal would be suitable for use in animal feeds and for energy production in pellets burning furnaces. As a mixture of fibers and proteins, it is also considered as a natural composite. It can be processed into biodegradable agromaterials by thermo-pressing.
During molding, part of residual oil is expressed (until 41% of oil from whole plant), leading to the increase of the total oil extraction yield (aqueous extraction in twin-screw extruder and expression during thermo-pressing): until 81% of oil from whole plant.
Panels have promising mechanical properties in bending (until 12MPa for stress at break). They are usable as inter-layer sheets for pallets, for their sound and heat insulation properties or for the manufacturing of containers by assembly of panels. Their hydrophobic character (8% for residual oil content in the panels) makes them resistant to water.

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Processing Technologies

The aqueous extraction of sunflower oil from whole plant in twin-screw extruder, a first step for the manufacturing of biodegradable agromaterials by thermo-pressing

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OUTLINE

AQUEOUS EXTRACTION OF SUNFLOWER OIL

- Aqueous extraction of sunflower oil is an environment-friendly alternative to the solvent oil extraction [1,2].
- It enables the simultaneous production of an **oil-in-water emulsion** (hydrophobic phase) and a **protein isolate** (hydrophilic phase) in the same process.
- It can be conducted in a twin-screw extruder starting from :
  - … the seeds [3].
  - … the press cake [4].
  - … the whole plant [5].


Direct extraction of oil from sunflower seeds by twin-screw extruder according to an aqueous extraction process: Feasibility study and influence of operating conditions

[3]


Aqueous extraction of residual oil from sunflower press cake using a twin-screw extruder: Feasibility study

[4]


La technologie d'extrusion bi-vis, une solution originale et performante pour le bioraffinage du tournesol plante entière

[5]

[1] **Rosenthal**, **A.**, **Pyle**, **D.L.** and **Niranjan**, **K.**, Aqueous and enzymatic processes for edible oil extraction. *Enzyme and Microbial Technology*, **19 (6)**, 402-420 (1996).

[2] **Evon**, **Ph.**, **Vandenbossche**, **V.**, **Pontalier**, **P.Y.** and **Rigal**, **L.**, Aqueous extraction of oil from sunflower seeds in batch reactor: reorganization of the mixing in three formulated fractions. *98th AOCS Annual Meeting & Expo*, AOCS, Québec City, QC, Canada (2007).

[3] **Evon**, **Ph.**, **Vandenbossche**, **V.**, **Pontalier**, **P.Y.** and **Rigal**, **L.**, Direct extraction of oil from sunflower seeds by twin-screw extruder according to an aqueous extraction process: feasibility study and influence of operating conditions. *Industrial Crops and Products*, **26 (3)**, 351-359 (2007).

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[5] **Evon**, **Ph.**, **Vandenbossche**, **V.**, **Pontalier**, **P.Y.** and **Rigal**, **L.**, The twin-screw extrusion technology, an original and powerful solution for the biorefinery of sunflower whole plant. *Oléagineux, Corps gras & Lipides*, **17 (6)**, 404-417 (2010).

RESULTS OF THE THERMO-MECHANICAL FRACTIONATION OF SUNFLOWER WHOLE PLANT [1]

- Experiments were conducted with a Clextral BC 45 (France) co-penetrating and co-rotating twin-screw extruder (80°C for the barrel temperature).

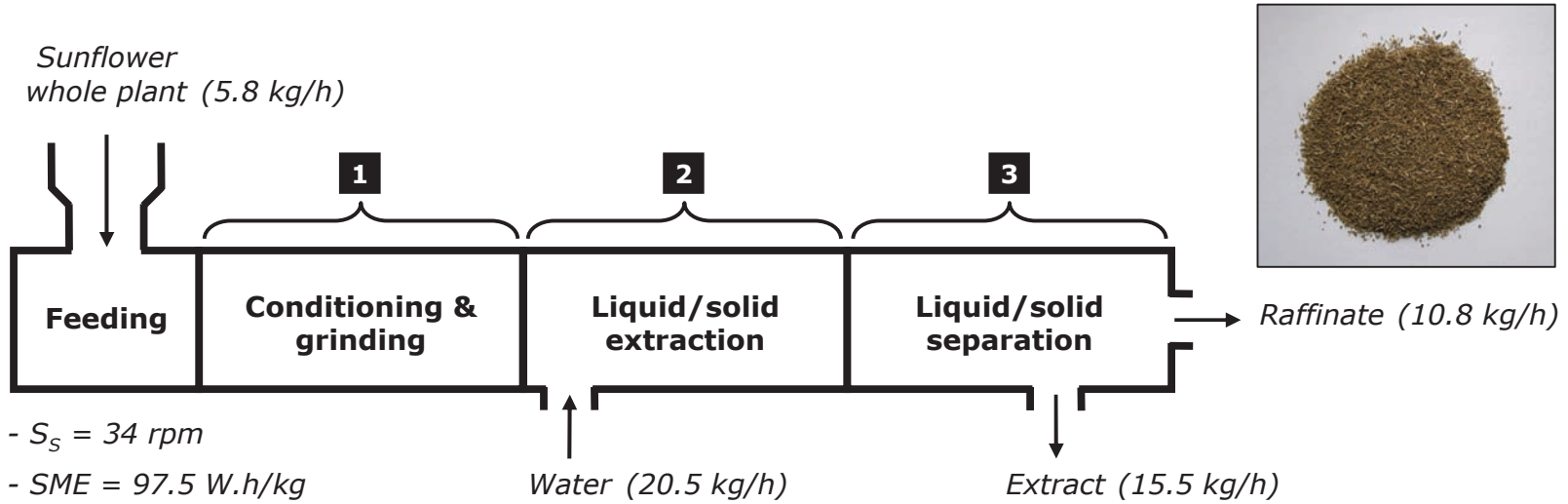
Helianthus annuus L.

- **Oleic sunflower whole plant** (15 mm homogenate) (La Toulousaine de Céréales, France):
  - 8.2% of moisture content.
  - 26.8% dry matter of oil content.
  - 10.7% dry matter of protein content.
  - 40.9% dry matter of fibres content (cellulose, hemicelluloses, and lignins).
  - *33.1% dry matter for cellulose, and lignins.*
  - 7.0% dry matter of pectin content.

- Co-extraction of oil, proteins, and pectins is effective with water starting from whole plant.

RESULTS OF THE THERMO-MECHANICAL FRACTIONATION OF SUNFLOWER WHOLE PLANT [2]

- After feeding, three essential unit operations are carried out in a single step:

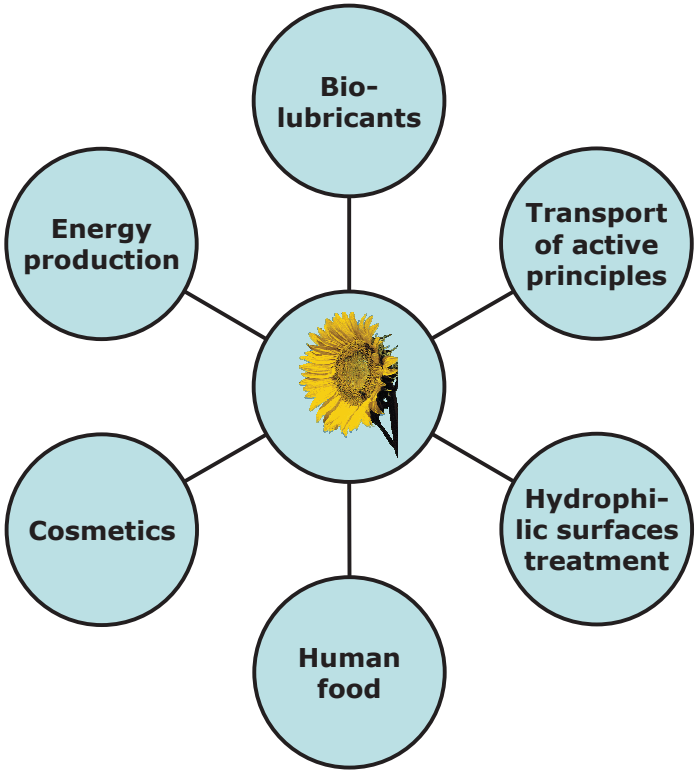


- Wringing out the mixing is favoured because of the natural abundance of fibres in the sunflower stalk (until 80%).
- An extract (filtrate) and a raffinate (cake meal) are produced continuously.

S_S, screw rotation speed; SME, specific mechanical energy.

APPLICATIONS OF THE LIQUID PHASES FOR INDUSTRIAL PRODUCTS

- The process is an original and powerful solution for the fractionation of the sunflower whole plant : ***biorefinery concept***.
- The extracted fractions (oil-in-water emulsions, and hydrophilic phase) may have applications as bases for industrial products:



DESCRIPTION OF THE CAKE MEAL

Cake meal

- In best operating conditions, residual oil content in the cake meal is 14% dry matter.
- The cake meal is first dried (105°C, 24 h) to make easier its conservation (< 5% for moisture content after drying).
- It is a mixture of lignocellulosic fibres and **proteins (globulins) with thermoplastic properties**.

- It would be suitable for:
  - ... use in animal feeds.
  - ... energy production in pellets burning furnaces.

THE CAKE MEAL, A NATURAL COMPOSITE

- As a mixture of fibres and proteins, the cake meal is also considered as a natural composite.

- Because of its thermo-mechanical behaviour, thermo-pressing is a promising molding operation for the manufacturing of **renewable and biodegradable agromaterials** [5,6].


La technologie d'extrusion bi-vis, une solution originale et performante pour le bioraffinage du tournesol plante entière

[5]


Thermo-mechanical behaviour of the raffinate resulting from the aqueous extraction of sunflower whole plant in twin-screw extruder: manufacturing of biodegradable agromaterials by thermo-pressing

[6]

[5] **Evon**, **Ph.**, **Vandenbossche**, **V.**, **Pontalier**, **P.Y.** and **Rigal**, **L.**, The twin-screw extrusion technology, an original and powerful solution for the biorefinery of sunflower whole plant. *Oléagineux, Corps gras & Lipides*, **17 (6)**, 404-417 (2010).

[6] **Evon**, **Ph.**, **Vandenbossche**, **V.**, **Pontalier**, **P.Y.** and **Rigal**, **L.**, Thermo-mechanical behaviour of the raffinate resulting from the aqueous extraction of sunflower whole plant in twin-screw extruder: manufacturing of biodegradable agromaterials by thermopressing. *Advanced Materials Research*, **112**, 63-72 (2010).

THERMO-MECHANICAL TRANSFORMATION OF THE CAKE MEAL DURING MOLDING

- The simultaneous effect of pressure and temperature results in the **glass transition** of proteins.

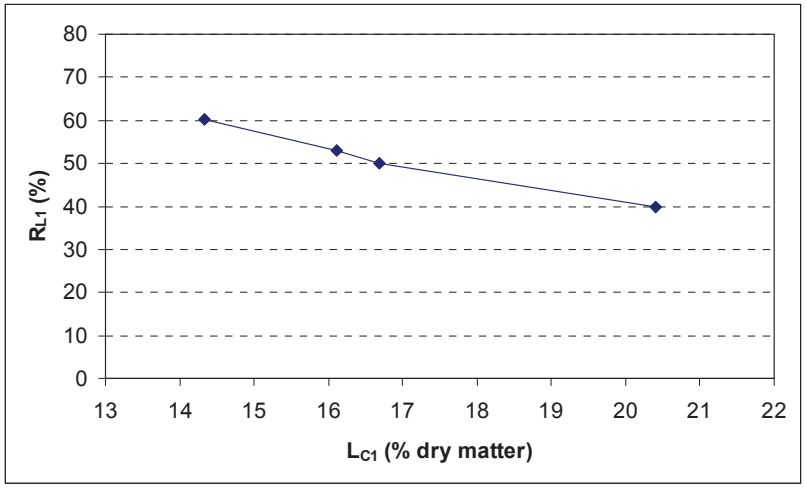
- The reorganization of their structure allows the **mechanical aspect** of the agromaterial; it gives to the agromaterial its **cohesion**, and its **flexibility**.

- The fibres entanglement also acts like **reinforcement**.

OUTLINE

CHEMICAL COMPOSITION OF THE CAKE MEAL

- Four different cakes were chosen, corresponding to different levels of aqueous extraction efficiency in twin-screw extruder.
- R_{L1}, oil extraction yield in twin-screw extruder (%), based on the residual oil content in the cake meal (L_{C1}) (% dry matter).
- In best operating conditions, oil yield is 60% (57% after foot removal in the filtrate) and residual oil content in the cake meal is 14% dry matter.



L_{C1} (% DM)	R_{L1} (%)
14.3 ± 0.1	60.2
16.1 ± 0.0	53.0
16.7 ± 0.1	50.0
20.4 ± 0.1	40.0

- 6.8-8.7% DM of protein content.
- 40-46% DM of fibres content (cellulose and lignins).

DM, dry matter.

OUTLINE

THERMO-PRESSING OF THE CAKE MEAL

- Heated hydraulic press used for thermo-pressing:
  - Model: MAPA 50 (PEI, France).
  - Force: 500 kN (50 tonnes).
  - Maximum pressure: 297 bars.

- Thermo-pressing conditions:
  - Mass of the cake meal: 125 g.
  - Temperature of the two plates: 200°C.
  - Pressure applied: 240 kgf/cm^2 during 3 min.

- Panels produced: 150 mm × 150 mm.

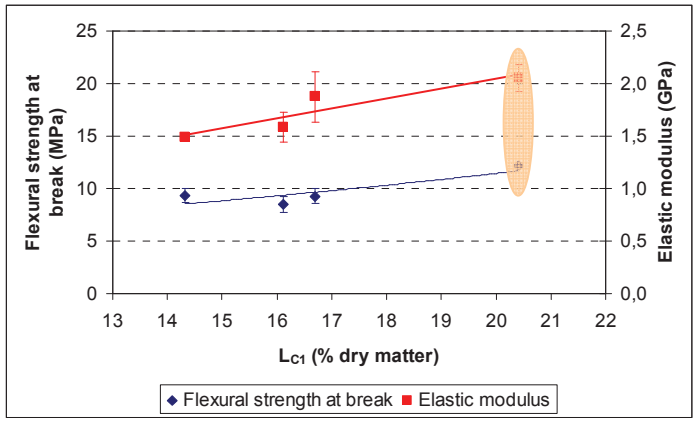
- Conditioning of specimens in a climatic chamber (60% RH, 25°C) during three weeks before bending tests.

MAPA 50 heated hydraulic press (laboratory equipment)

OUTLINE

INFLUENCE OF AQUEOUS EXTRACTION EFFICIENCY ON MECHANICAL PROPERTIES IN BENDING OF THE AGROMATERIAL

- Agromaterials have promising mechanical properties in bending (standard NF EN 310): until 12 MPa for stress at break.



- Best stress at break (12 MPa) is obtained with the less dehulled cake meal, due to a most important content of proteins inside it (10% DM after oil expression).
- It is the most rigid agromaterial (2 GPa for elastic modulus).

L_{C1} (% dry matter)	14.3 ± 0.1	16.1 ± 0.0	16.7 ± 0.1	**20.4 ± 0.1**
F (N)	37.5 ± 2.1	42.0 ± 3.2	37.7 ± 3.2	**47.9 ± 2.6**
σ_f (MPa)	9.4 ± 0.7	8.5 ± 0.8	9.3 ± 0.7	**12.2 ± 0.2**
E_f (GPa)	1.5 ± 0.0	1.6 ± 0.1	1.9 ± 0.2	**2.1 ± 0.1**

F, breaking load (30 mm for specimen width & 100 mm for grip separation); σ_f, flexural strength at break; E_f, elastic modulus.

FORMULAS FOR OIL YIELDS CALCULATION

- R_{L2}, oil expression yield during molding (in proportion to the oil that the cake meal contains) (%).

- R_{L2}', oil expression yield during molding (in proportion to the oil that the sunflower whole plant contains) (%).

- R_{LT}, total oil yield (oil extracted in twin-screw extruder, and oil expressed during molding) (%).

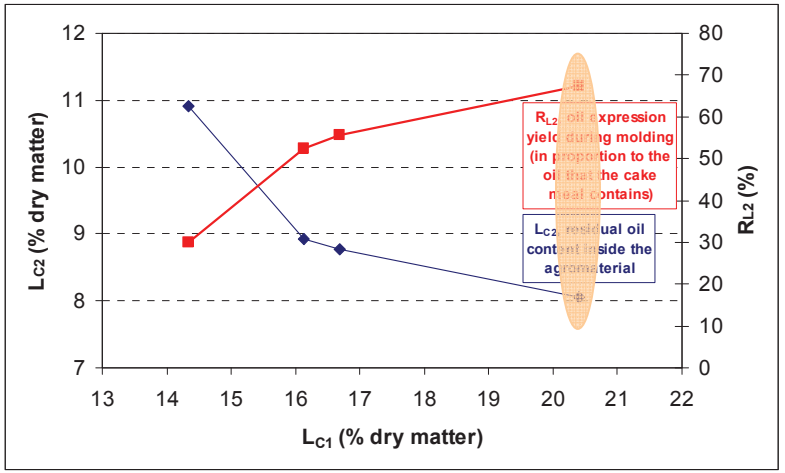
$$R_{L2}' = R_{L2} \times \frac{100 - R_{L1}}{100}$$

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

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

INFLUENCE OF AQUEOUS EXTRACTION EFFICIENCY ON TOTAL OIL YIELD [1]

- During molding, until 67% of residual oil from cake meal is expressed (i.e. until 40% of oil from whole plant).
- Lowest residual oil content in the agromaterial (8% dry matter) is obtained with the less dehulled cake meal.

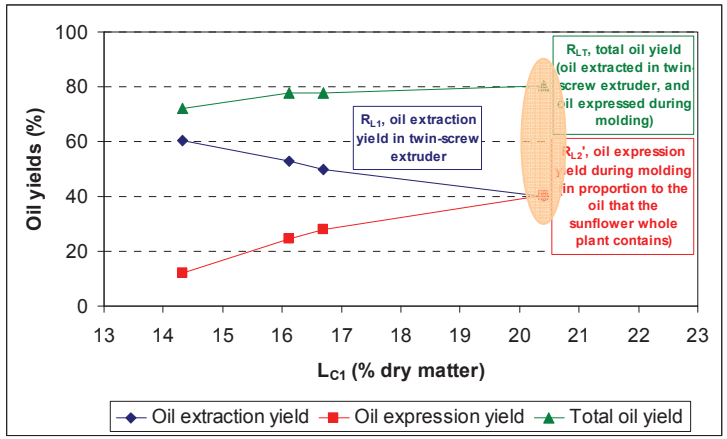


L_{C1} (% dry matter)	14.3 ± 0.1	16.1 ± 0.0	16.7 ± 0.1	**20.4 ± 0.1**
L_{C2} (% dry matter)	10.9 ± 0.0	8.9 ± 0.0	8.8 ± 0.0	**8.1 ± 0.0**
R_{L2} (%)	29.9	52.4	55.5	**67.3**

INFLUENCE OF AQUEOUS EXTRACTION EFFICIENCY ON TOTAL OIL YIELD [2]

- Oil expression during molding leads to the increase of the total oil yield (R_{LT}): aqueous extraction in twin-screw extruder (R_{L1}), and expression during thermo-pressing (R_{L2}').

- Until 80% of oil from whole plant for R_{LT} (case of the less de-hulled cake meal that gives also the most resistant agromaterial).



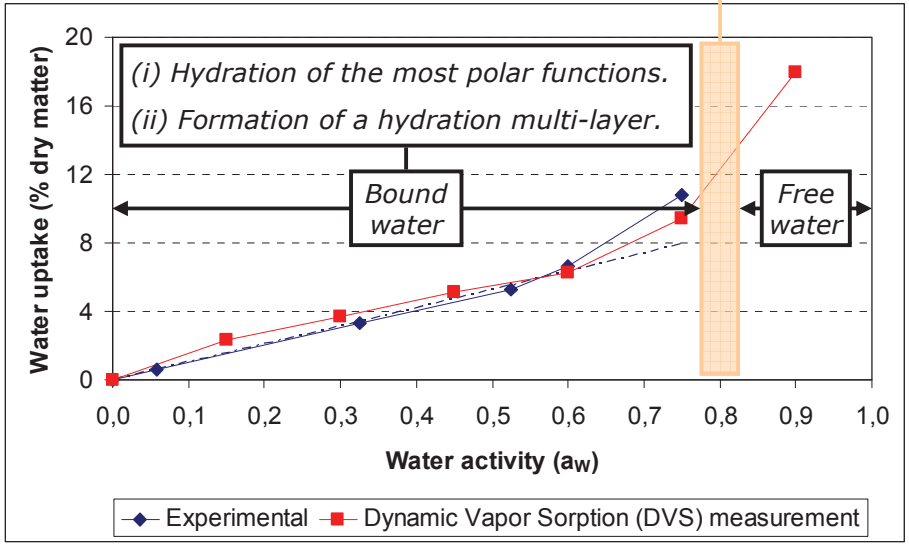
L_{C1} (% dry matter)	R_{L1} (%)	R_{L2}' (%)	R_{LT} (%)
14.3 ± 0.1	60.2	11.9	72.1
16.1 ± 0.0	53.0	24.6	77.6
16.7 ± 0.1	50.0	27.8	77.8
20.4 ± 0.1	**40.0**	**40.4**	**80.4**

OUTLINE

INFLUENCE OF THE AGROMATERIAL CONDITIONING ON ITS MECHANICAL PROPERTIES IN BENDING [1]

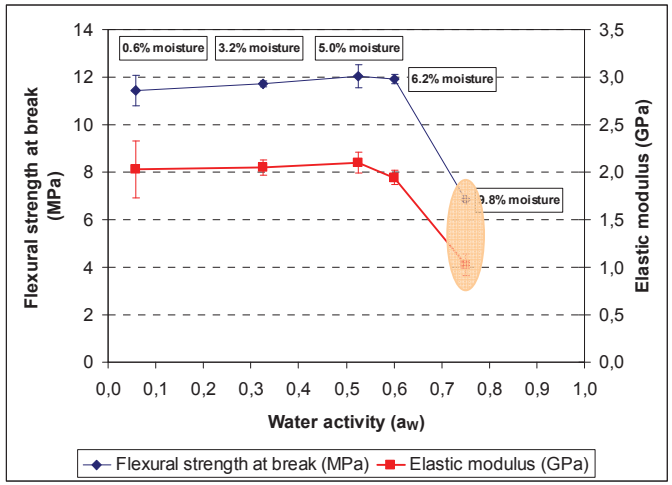
- Thermo-pressing conditions:
  - Cake meal used for study: cake number 4 (i.e. the less dehulled).
  - Mass of the cake meal: 125 g.
  - Temperature of the two plates: 200°C.
  - Pressure applied: 240 kgf/cm^2 during 4 min.
- Panel produced: 150 mm × 150 mm.
- Conditioning (25°C) during three weeks in five different atmospheres:
  - 5.0% RH.
  - 32.5% RH.
  - 52.5% RH.
  - 60.0% RH.
  - 75.0% RH.
- Changes in water uptake.

Glass transition of sunflower proteins (globulins) at room temperature



INFLUENCE OF THE AGROMATERIAL CONDITIONING ON ITS MECHANICAL PROPERTIES IN BENDING [2]

- Mechanical properties in bending of the agromaterial are decreasing drastically when conditioning is realized at 75.0% RH, due to water that acts as a plasticizer for globulins [7,8].



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[8] **Rouilly**, **A.**, **Orliac**, **O.**, **Silvestre**, **F.** and **Rigal**, **L.**, Thermal denaturation of sunflower globulins in low moisture conditions. *Thermochimica Acta*, **398 (1-2)**, 195-201 (2003).

- Stress at break is only 7 MPa at 75.0% RH instead of 11-12 MPa for the other conditionings.
- Same tendency is also observed with the elastic modulus.

Conditioning	5.0% RH	32.5% RH	52.5% RH	60.0% RH	**75.0% RH**
F (N)	39.5 ± 3.8	44.3 ± 0.8	46.2 ± 1.3	44.1 ± 2.4	**27.6 ± 0.3**
σ_f (MPa)	11.4 ± 0.6	11.7 ± 0.1	12.0 ± 0.5	11.9 ± 0.2	**6.9 ± 0.0**
E_f (GPa)	2.0 ± 0.3	2.0 ± 0.1	2.1 ± 0.1	1.9 ± 0.1	**1.0 ± 0.1**

F, breaking load (30 mm for specimen width & 100 mm for grip separation); σ_f, flexural strength at break; E_f, elastic modulus.

OUTLINE

CONCLUSION [1]

- **Twin-screw extrusion** technology is an original solution for the **biorefinery** of sunflower whole plant.
- Such process is a first step for the manufacturing of **agro-materials**.
- As a mixture of fibres and proteins, the cake meal generated can be considered as a **natural composite**.
- It can be processed into **biodegradable agromaterials** by **thermo-pressing**.
- During molding, part of residual oil is expressed, leading to the increase of the total oil yield: until 80% of oil from whole plant (corresponding also to the most resistant agromaterial).
- The hydrophobic character of the agromaterials (at least 8% dry matter for residual oil content inside them) makes them resistant to water.

CONCLUSION [2]

- Agromaterials have promising mechanical properties in bending compared with those of other industrial (Isorel®, laminated board…) and experimental (vegetable fiberboards…) materials:
  - Until 12.2 MPa for stress at break.
  - Until 2.1 GPa for elastic modulus.
  - 1.03 for the corresponding density.

- The manufacturing of agromaterials (i) with higher dimensions and (ii) with same mechanical properties in bending is still possible with an industrial heated hydraulic press:
  - Model: MAPA 400 (PEI, France).
  - Force: 4000 kN (400 tonnes).
  - Maximum pressure: 270 bars.
  - Panels produced: until 800 mm × 800 mm.

MAPA 400 heated hydraulic press (industrial equipment)

POTENTIAL USES OF THE BIODEGRADABLE AGROMATERIALS

- Panels:
  - Inter-layer sheets for pallets in handling and storage industry.
  - Sound and heat insulation panels (between 90 and 140 mW/m K for thermal conductivity at 25°C) in building trade…
- Conic bowls for the feeding of pets (cats, dogs).
- Containers (assembly of panels):
  - Composters.
  - Crates for vegetable gardening…

(i) Panel
(thermo-pressing)

(ii) Conic bowl
(thermo-molding)

(iii) Composter
(assembly of panels)

Thank you for your attention.

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