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- *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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World Conference on Oilseed Processing, Fats & Oils Processing, Biofuels & Applications

21-23 June 2011 ♦ Hilton Izmir ♦ Izmir, Turkey

Where the Global Fats and Oils Community Will Discuss Critical Scientific Issues and Technologies Affecting the Future of the Industry.

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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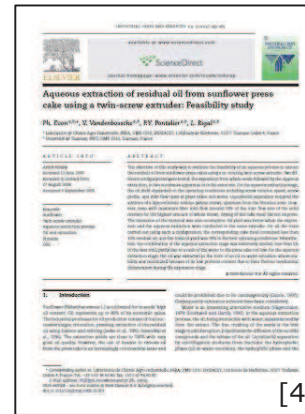
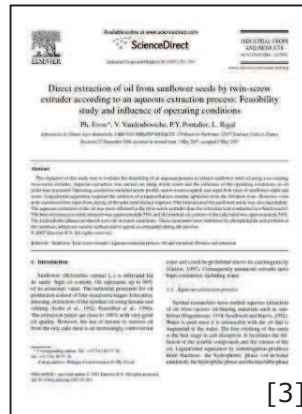
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4. Conclusion.

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].



[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., Vandebossche, 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., Vandebossche, 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).

[4] **Evon, Ph., Vandebossche, V., Pontalier, P.Y. and Rigal, L.**, Aqueous extraction of residual oil from sunflower press cake using a twin-screw extruder: feasibility study. *Industrial Crops and Products*, **29 (2-3)**, 455-465 (2009).

[5] **Evon, Ph., Vandebossche, 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.

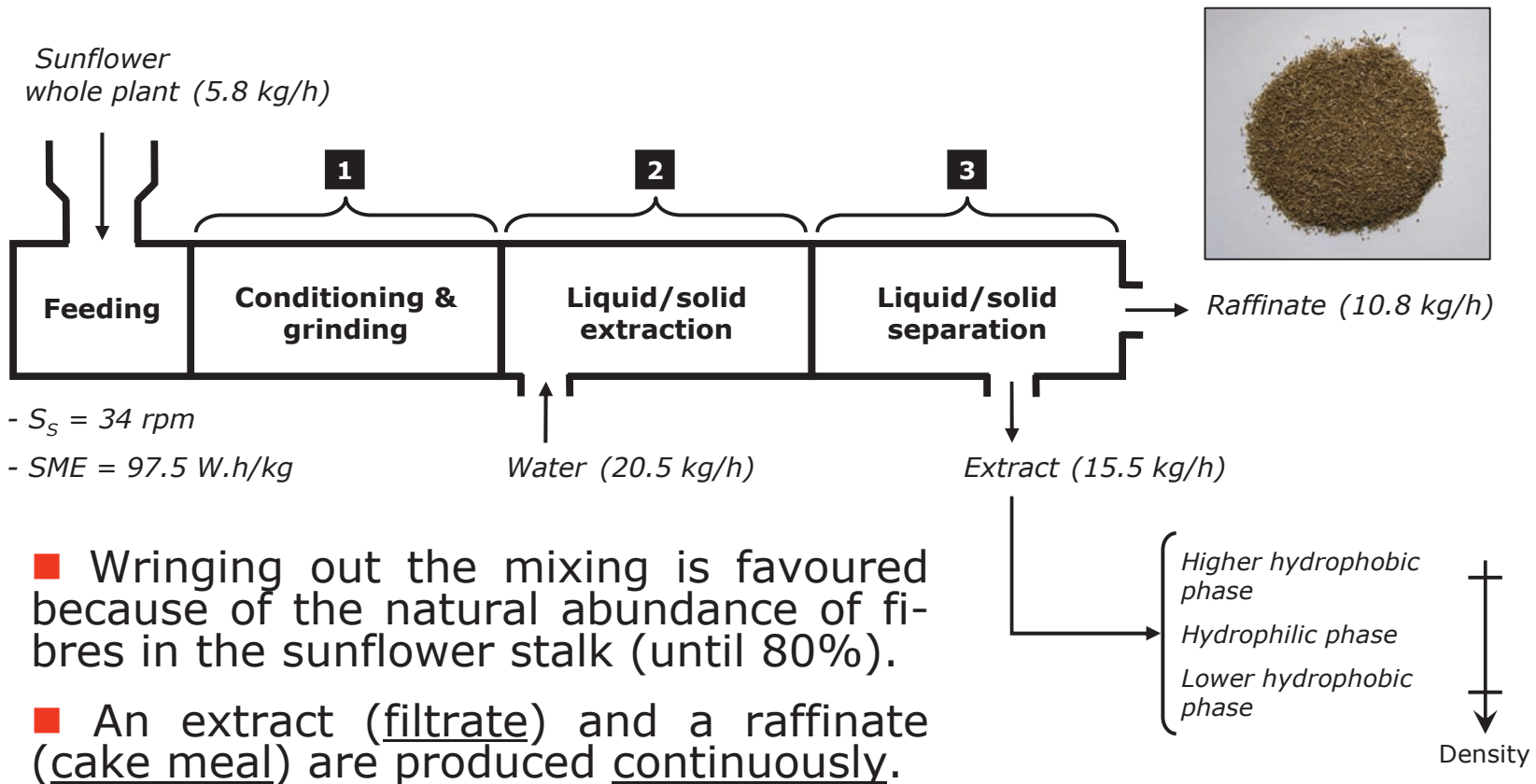
■ **Oleic sunflower whole plant** (15 mm homogenate) (La Toulousaine de Céréales, France):

- 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:

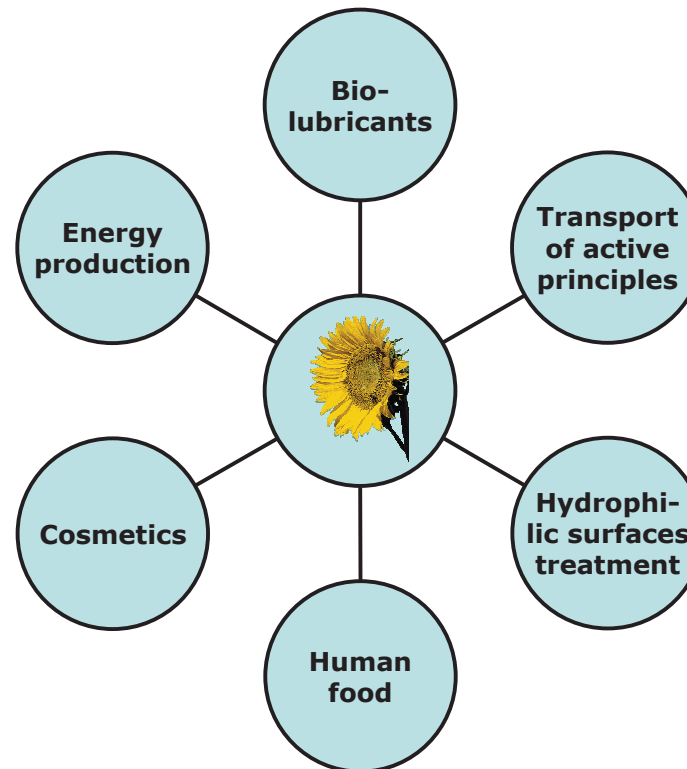


- Wringing out the mixing is favoured because of the natural abundance of fibres in the sunflower stalk (until 80%).
- An extract (filtrate) and a raffinate (cake meal) are produced continuously.

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].



[5]



[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**.

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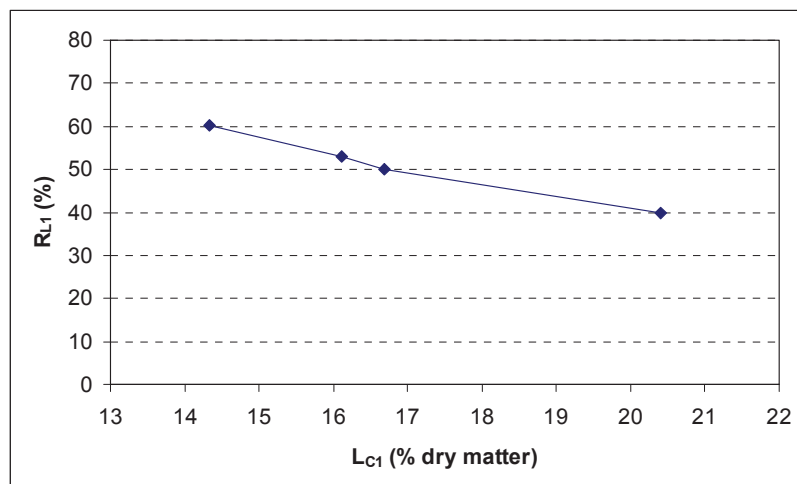
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CHEMICAL COMPOSITION OF THE CAKE MEAL

- 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.

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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/cm² 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)

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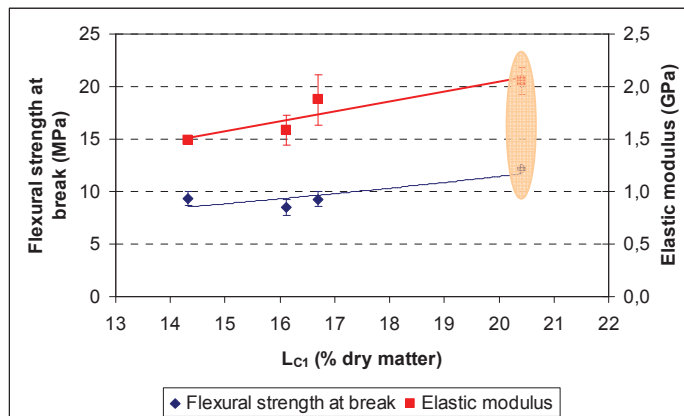
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INFLUENCE OF AQUEOUS EXTRACTION EFFICIENCY ON MECHANICAL PROPERTIES IN BENDING OF THE AGROMATERIAL

■ 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) (%).

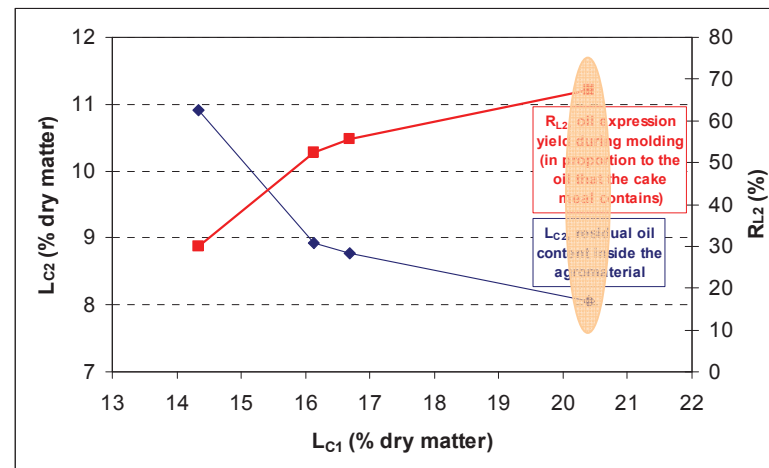
$$R_{L2}' = R_{L2} \times \frac{100 - R_{L1}}{100}$$

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

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

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

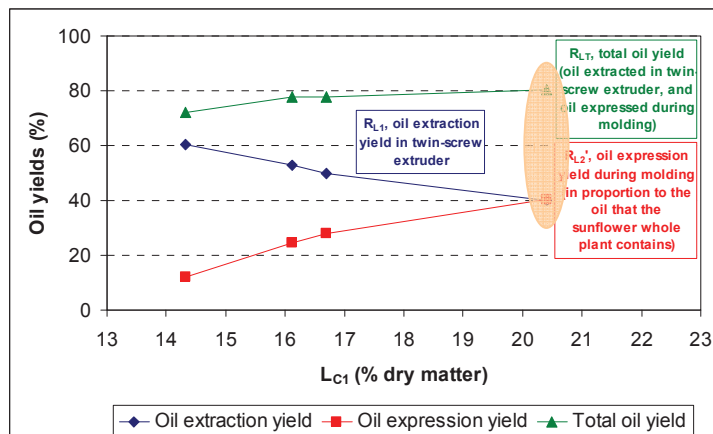
- During molding, until 67% of residual oil from cake meal is expressed (i.e. until 40% of oil from whole plant).
- Lowest residual oil content in the agromaterial (8% dry matter) is obtained with the less dehulled cake meal.



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

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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/cm² during 4 min.

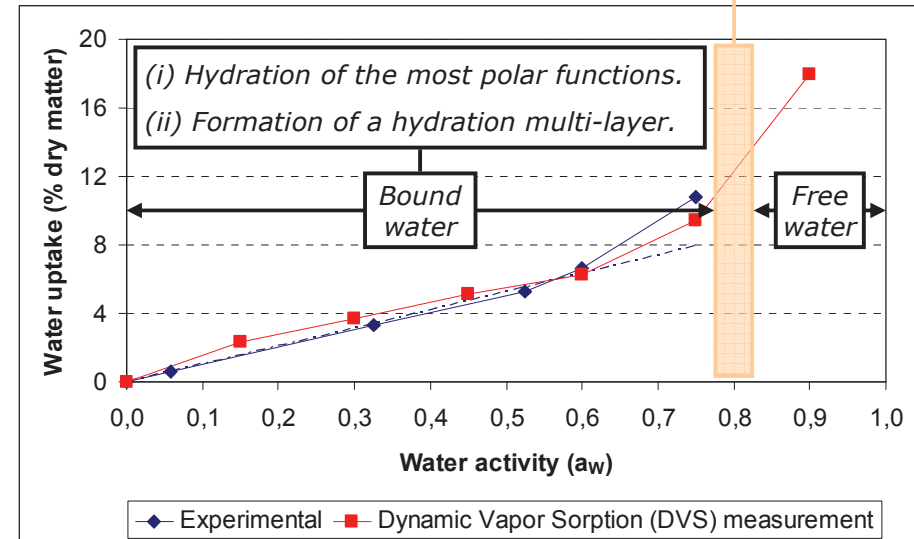
■ Panel produced: 150 mm × 150 mm.

■ 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.

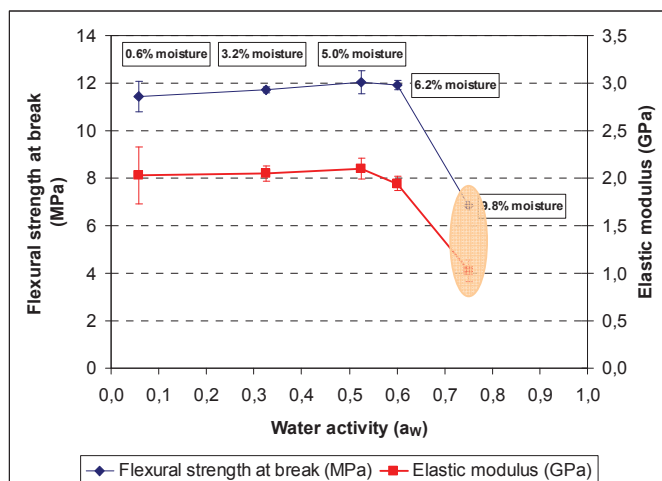
■ Changes in water uptake.

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].



[7] Rouilly, A., Orliac, O., Silvestre, F. and Rigal, L., DSC study on the thermal properties of sunflower proteins according to their water content. *Polymer*, **42 (26)**, 10111-10117 (2001).

[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.

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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*)



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Thank you for your attention.

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