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Production of binderless fiberboards from *Calophyllum inophyllum* twin-screw extrusion cakes through thermopressing

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Vegetable oil from *Calophyllum inophyllum* kernels appears as a promising raw material for biodiesel production in Indonesia. For this, the oil must first be extracted and this is the twin-screw extrusion technology that has been used in that case to conduct such extraction. A two-step process was developed consisting in a mechanical pressing and then in the aqueous extraction of residual oil in the press cake. And, it should be noted that a fibrous residue (i.e. sunflower hull) was added in the second twin-screw reactor to facilitate the liquid/solid separation during the aqueous extraction stage. In the best condition tested, 63% of the oil was extracted. In addition, depending on the extrusion conditions used, residual oil content in the final cake varied from 16.7 to 19.1% of its dry matter instead of 74.0% inside the starting material (i.e. the kernel).

In this study, the use of the final cakes as sources for fiberboard manufacture through thermopressing was investigated. These cakes can be considered as natural composites with lignocellulosic fibers from the fibrous residue and proteins from the *Calophyllum inophyllum* kernel breakdown process acting respectively as reinforcing fillers and a natural binder [1-2]. Cohesive self-bonded boards were thus obtained through thermopressing using the next molding conditions: 200 °C temperature, 20 MPa pressure and 300 s molding time.

For the three cakes tested, part of their residual oil was expressed through the sidewall vents of the mold during molding, which was due to the applied pressure, and this contributed to the increase in the total vegetable oil recovery. In addition, the more the residual oil content in the cake, the more the oil expressed. However, oil expression during molding reduced the mechanical resistance of fiberboards, due to defects appearing within the material as the oil escaped. This contributed to a slight increase in the bending properties (from 12.1 to 15.4 MPa for flexural strength at break and from 1.9 to 2.0 GPa for elastic modulus) with the cake's residual oil content decrease (from 19.1 to 16.7%).

A significant improvement in the molding process consisted in conducting the cake's deoiling prior thermopressing using a Soxhlet extraction apparatus and cyclohexane as extracting solvent. From this, the properties of the corresponding board (4.3 mm thickness and 1185 kg/m³ density) were much improved: 79° Shore D surface hardness (instead of 70° without deoiling), 23.7 MPa flexural strength at break and 3.1 GPa elastic modulus. In parallel, thickness swelling after 24 h immersion in water was 43.9% and it has decreased up to 27.6% after the board's heat treatment (200 °C during 10 min).

This optimal fiberboard complied with the French standard NF EN 312 (standard dedicated to the specifications for particleboards), type P7 (i.e. load bearing boards for use under high stress and in wet conditions) for flexural properties (recommendations of 21 MPa and 3.1 GPa for flexural strength at break and elastic modulus, respectively, for boards with a 4 to 6 mm thickness). However, thickness swelling after immersion in water will need to be significantly reduced to achieve the 10% recommended standard value.

[1] P. Evon, J. Vinet, L. Labonne & L. Rigal. *Ind. Crops Prod.*, **65** (2015) 117-126.

[2] E. Uitterhaegen, L. Labonne, O. Merah, T. Talou, S. Ballas, T. Véronèse & P. Evon. *J. Appl. Polym. Sci.*, doi: 10.1002/app.44650 (2017).

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Introduction

- ▶ Vegetable oil from *Calophyllum inophyllum* kernels appears as a promising raw material for biodiesel production in Indonesia.
- ▶ For this, the oil must first be extracted and this is the twin-screw extrusion technology that has been used in that case to conduct such extraction.
- ▶ A two-step process was developed consisting in a mechanical pressing and then in the aqueous extraction of residual oil in the press cake (Fig. 1).
- ▶ A fibrous residue (i.e. sunflower hull) was added in the second twin-screw reactor to facilitate the liquid/solid separation during the aqueous extraction stage.
- ▶ In the best condition tested, 63% of the oil was extracted. Depending on the extrusion conditions used, residual oil content in the final cake varied from 16.7 to 19.1% (Table 1) of its dry matter instead of 74.0% inside the starting material (i.e. the kernel).
- ▶ In this study, the use of the final cakes as sources for fiberboard manufacture through thermopressing is investigated.

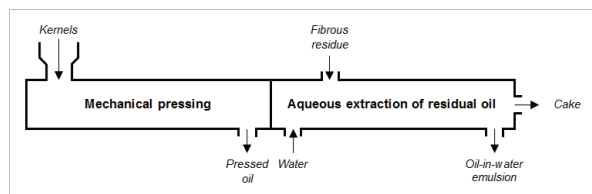


Figure 1. Scheme of the extraction of vegetable oil from *Calophyllum inophyllum* kernels using twin-screw extrusion.

Table 1. Oil yields and residual oil content in cakes number 1 to 3.

Cake number	1	2	3
Oil extraction yield for step 1 (%)	29.0	29.0	29.0
Oil extraction yield for step 2 (%)	34.3	22.9	21.2
Total oil extraction yield (%)	63.3	51.9	50.2
R _{TC} (%)	91.4	90.2	89.7
Residual oil in cake (% of dry matter)	19.1±0.0	16.8±0.3	16.7±0.1

R_{TC}, total oil extraction yield based on the residual oil content of the cake (%).

Results and discussion

- ▶ Cakes can be considered as natural composites with lignocellulosic fibers from the fibrous residue and proteins from the *Calophyllum inophyllum* kernel breakdown process acting respectively as reinforcing fillers and a natural binder [1-2].
- ▶ Cohesive self-bonded boards were obtained through thermopressing (200 °C temperature, 20 MPa pressure and 300 s molding time). ⇒ **No risk of thermal degradation (Fig. 2).**
- ▶ Part of the cake's residual oil was expressed through the sidewall vents of the mold during molding, which was due to the applied pressure. ⇒ **Increase in the total vegetable oil recovery (until 87%).**
⇒ **The more the residual oil content in the cake, the more the oil expressed.**
- ▶ Oil expression during molding reduced the mechanical resistance of fiberboards, due to defects appearing within the material as the oil escaped.
⇒ **Increase in the bending properties with the cake's residual oil content decrease (Table 2).**
- ▶ A significant improvement in the molding process consisted in conducting the cake's deoiling prior thermopressing. ⇒ **Properties of the associated board much improved (Tables 2 and 3, Fig. 3).**

Table 2. Characteristics of binderless fiberboards obtained.

Panel	1	2	3	3d (deoiled)
t (mm)	5.0±0.3	5.1±0.2	5.0±0.1	4.3±0.2
d (kg/m ³)	1148±46	1166±29	1175±35	1185±36
σ _f (MPa)	12.1±2.7	15.1±2.3	15.4±1.3	23.7±2.0
E _f (GPa)	1.89±0.28	1.95±0.27	1.97±0.19	3.07±0.38
IB (MPa)	n.d.	n.d.	n.d.	0.71±0.04
Shore D (°)	69.9±6.2	69.2±2.5	69.5±1.6	78.8±2.3
TS (%)	76±6	88±5	85±8	44±5
After heat treatment	n.d.	n.d.	n.d.	28±2
WA (%)	67±3	80±2	76±3	43±2
After heat treatment	n.d.	n.d.	n.d.	36±3

t, thickness; d, density; σ_f, flexural strength at break; E_f, elastic modulus; IB, internal bond strength; Shore D, surface hardness; TS, thickness swelling; WA, water absorption. – n.d., non determined.

Table 3. Chemical composition of cake number 3 after deoiling.

Compounds	Content (% of dry matter)
Minerals	3.2±0.1
Lipids	0.9±0.3
Proteins	12.9±0.2
Cellulose	43.5±0.1
Hemicelluloses	17.0±0.4
Lignins	17.3±0.1
Water-solubles	10.3±0.0

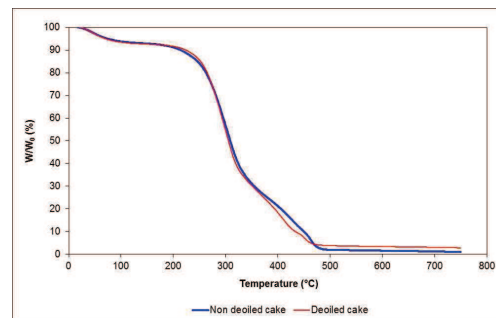


Figure 2. TGA degradation curve of cake number 3 before and after deoiling using a Soxhlet extraction apparatus and cyclohexane as extracting solvent.

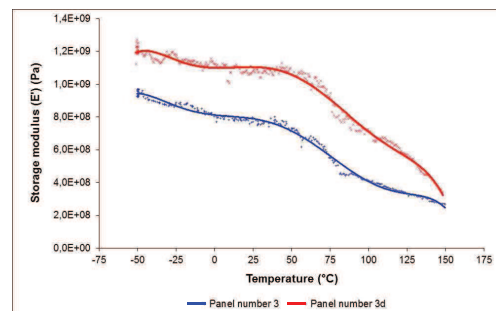


Figure 3. DMTA curves of panels made from cake number 3 before and after deoiling.

Conclusion

- ▶ Optimal fiberboard complies with the French standard NF EN 312 (standard dedicated to the specifications for particleboards), type P7 (i.e. load bearing boards for use under high stress and in wet conditions) for flexural properties (recommendations of 21 MPa and 3.1 GPa for flexural strength at break and elastic modulus, respectively, for boards with a 4 to 6 mm thickness) and almost for internal bond strength (recommendation of 0.75 MPa).
- ▶ However, thickness swelling after immersion in water will need to be significantly reduced to achieve the 10% recommended standard value.

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REFERENCES

- [1] P. Evon, J. Vinet, L. Labonne & L. Rigal, Influence of thermo-pressing conditions on the mechanical properties of biodegradable fiberboards made from a deoiled sunflower cake. *Industrial Crops and Products*, 65 (2015) 117-126.
[2] E. Uitterhaegen, L. Labonne, O. Merah, T. Talou, S. Ballas, T. Véroñese & P. Evon, Optimization of thermopressing conditions for the production of binderless boards from a coriander twin-screw extrusion cake. *Journal of Applied Polymer Science*, doi: 10.1002/app.44650 (2017).

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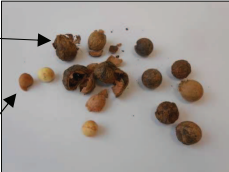
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INP ENSIACET INRA Université de Toulouse AM AGROMAT R&D à l'échelle industrielle

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Calophyllum inophyllum L.


- A tropical tree largely present in Indonesia.
- Possible uses of vegetable oil from fruit:
 - **Drugs:**
cicatrizing properties, cure for rheumatism, ulcers, burns, skin diseases but also on infected wounds.
 - **Formulation of cosmetics.**
 - **Antibacterial properties.**
 - **Promising raw material for biodiesel production.**




Hull

Kernel

Dried fruits



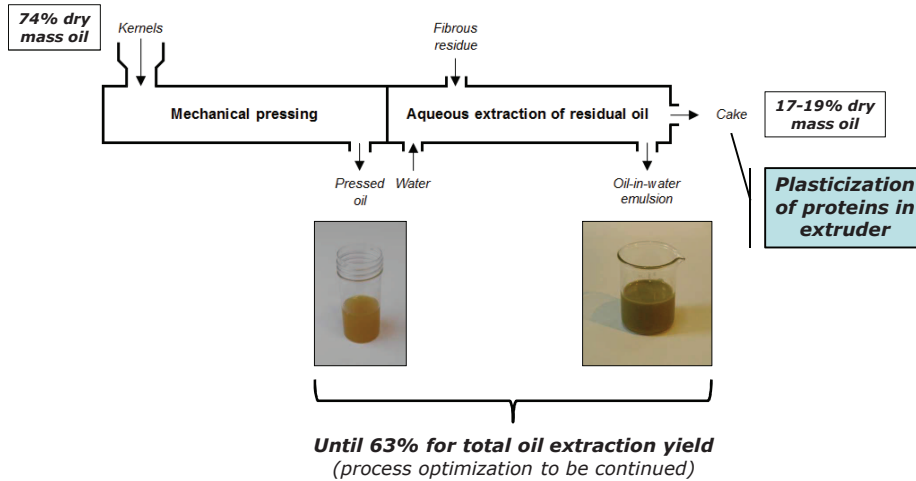
Calophyllum inophyllum L. fruits



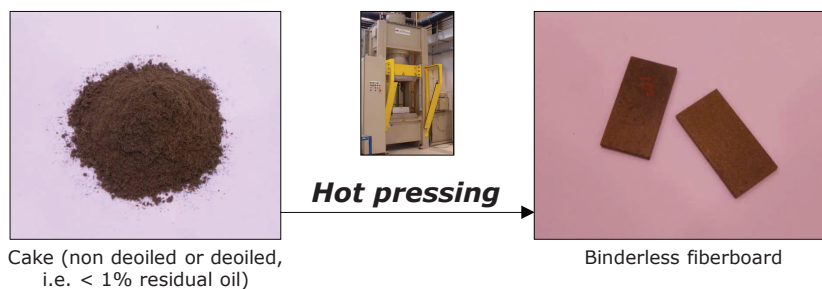
Calophyllum inophyllum L. tree

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Twin-screw extrusion process for oil extraction



Molding of binderless fiberboards from cakes



- Plasticized proteins (12.9%)
- Water-soluble compounds (10.3%)
- Lignocellulosic fibers (77.8%)

Natural binders

Mechanical reinforcement

Characteristics of optimal binderless fiberboard

Board type	σ_f (MPa)	E_f (GPa)	IB (MPa)	TS (%)
P5 <i>(load bearing boards for use in wet conditions)</i>	19	2.45	0.45	14
P6 <i>(load bearing boards for use under high stress and in dry conditions)</i>	20	2.90	0.65	16
P7 <i>(load bearing boards for use under high stress and in wet conditions)</i>	21	3.10	0.75	10
Optimal binderless fiberboard	23.7±2.0	3.1±0.4	0.71±0.04	44±5 <i>(28±2 after heat treatment)</i>

- Optimal binderless fiberboard complies with the French standard NF EN 312, type P7 for flexural properties and almost for internal bond strength.
- Thickness swelling after immersion in water will need to be reduced.