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Twin-Screw Extrusion Process to Produce Renewable Fiberboards

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Abstract

A versatile twin-screw extrusion process to provide an efficient thermo-mechano-chemical pre-treatment on lignocellulosic biomass before using it as source of mechanical reinforcement in fully bio-based fiberboards was developed. Various lignocellulosic crop by-products have already been successfully pre-treated through this process, e.g., cereal straws (especially rice), coriander straw, shives from oleaginous flax straw, and bark of both amaranth and sunflower stems.

The extrusion process results in a marked increase in the average fiber aspect ratio, leading to improved mechanical properties of fiberboards. The twin-screw extruder can also be fitted with a filtration module at the end of the barrel. The continuous extraction of various chemicals (e.g., free sugars, hemicelluloses, volatiles from essential oil fractions, etc.) from the lignocellulosic substrate, and the fiber refining can, therefore, be performed simultaneously.

The extruder can also be used for its mixing ability: a natural binder (e.g., Organosolv lignins, protein-based oilcakes, starch, etc.) can be added to the refined fibers at the end of the screw profile. The obtained premix is ready to be molded through hot pressing, with the natural binder contributing to fiberboard cohesion. Such a combined process in a single extruder pass improves the production time, production cost, and may lead to reduction in plant production size. Because all the operations are performed in a single step, fiber morphology is better preserved, thanks to a reduced residence time of the material inside the extruder, resulting in enhanced material performances. Such one-step extrusion operation may be at the origin of a valuable industrial process intensification.

Compared to commercial wood-based materials, these fully bio-based fiberboards do not emit any formaldehyde, and they could find various applications, e.g., intermediate containers, furniture, domestic flooring, shelving, general construction, etc.

Introduction

Extrusion is a process during which a flowing material is forced through a hot die. Extrusion, therefore, permits the forming of preheated products under pressure. The first industrial single-screw extruder appeared in 1873. It was used for the manufacture of metallic continuous cables. From 1930 onwards, single-screw extrusion was adapted to the food industry to produce sausages and past. Conversely, the first twin-screw extruder has first been used for developments in the food industry. It did not appear in the field of synthetic polymers until the 1940s. For this purpose, new machines were designed, and their operation was also modeled¹. A system with co-penetrating and co-rotating screws was developed, allowing mixing and extrusion to be carried out simultaneously. Since then, the extrusion technology has developed continuously via the design of new types of screws. Today, the food industry makes extensive use of twin-screw extrusion although it is more expensive than single-screw extrusion as twin-screw extrusion permits access to more elaborated material processing and final products. It is particularly used for extrusion-cooking of starchy products but also the texturing of proteins and the manufacture of pet food and fish feed.

More recently, twin-screw extrusion has seen its field of application extended to the thermo-mechano-chemical fractionation of plant matter^{2,3}. This new concept has led to the development of real reactors capable of transforming or fractionating plant matters in a single step, up to the separate production of an extract and a raffinate by liquid/

solid separation^{2,3,4}. Work carried out at the Laboratory of Agro-industrial Chemistry (LCA) has highlighted the multiple possibilities of the twin-screw technology for the fractionation and valorization of agroresources^{2,3}. Some of the examples are: 1) The mechanical pressing and/or "green" solvent extraction of vegetable oil^{5,6,7,8,9,10}. 2) The extraction of hemicelluloses^{11,12}, pectins¹³, proteins^{14,15}, and polyphenolic extracts¹⁶. 3) The enzymatic degradation of plant cell walls for producing second-generation bioethanol¹⁷. 4) The production of biocomposite materials with protein¹⁸ or polysaccharide¹⁹ matrices. 5) The production of thermoplastic materials by mixing cereals, and bio-based polyesters^{20,21}. 6) The production of biocomposites by compounding a thermoplastic polymer, bio-based or not, and plant fillers^{22,23}. 7) The defibration of lignocellulosic materials for producing paper pulp^{13,24}, and fiberboards^{25,26,27,28,29,30,31,32}.

The twin-screw extruder is often considered as a continuous thermo-mechano-chemical (TMC) reactor. Indeed, it combines in a single step chemical, thermal, and, also, mechanical actions. The chemical one results in the possibility to inject liquid reagents in various points along the barrel. The thermal one is possible due to the thermal regulation of the barrel. Lastly, the mechanical one depends on the choice of the screw elements along the screw profile.

For the defibration of lignocellulosic materials to produce fiberboards, the most recent works have used rice straw^{25,28}, coriander straw^{26,29}, oleaginous flax shives²⁷ as well as

sunflower^{30,32} and amaranth³¹ barks. The current interest of lignocellulosic biomasses for such an application (i.e., mechanical reinforcement) is explained by the regular depletion of forest resources used for producing wood-based materials. Crop residues are inexpensive and may be widely available. In addition, current wood particles are mixed with petrochemical resins which can be toxic. Often accounting for more than 30% of the total cost of current commercial materials³³, some resins contribute to formaldehyde emissions and reduce indoor air quality³⁴. Research interest has shifted to the use of natural binders.

Lignocellulosic biomass is mainly composed of cellulose and hemicelluloses, forming a heterogeneous complex. Hemicelluloses are impregnated with layers of lignins that form a three-dimensional network around these complexes. The use of lignocellulosic biomass for the manufacture of fiberboards generally requires a defibration pre-treatment. For this, it is necessary to break down the lignins that protect cellulose and hemicelluloses. Mechanical, thermal, and chemical³⁵ or even enzymatic^{36,37,38} pre-treatments must be applied. These steps also increase self-adhesion of fibers, which can promote the production of binderless boards²⁷ even if an exogenous binder is most often added.

The primary purpose of pre-treatments is to improve the particle size profile of micrometric fibers. A simple grinding offers the possibility to reduce the fiber size^{27,39,40}. Inexpensive, it contributes to increase the fiber specific surface. The components of the inner cell wall become more accessible and the mechanical properties of the obtained panels are improved. The efficiency of defibration is significantly increased when a thermo-mechanical pulp is produced, e.g., by digestion plus defibration⁴¹, from different pulping processes⁴² or by

steam explosion^{43,44,45,46,47}. More recently, LCA has developed an original pre-treatment of lignocellulosic fibers using twin-screw extrusion^{25,26,27,28,29,30,31,32}. After TMC defibration, the extruder also enables the homogeneous dispersion of a natural binder inside fibers. The resulting premix is ready to be hot pressed into fiberboards.

During the defibration of rice straw, twin-screw extrusion was compared to a digestion plus defibration process²⁵. The extrusion method revealed a significantly reduced cost, i.e., nine times lower than the pulping one. Furthermore, the amount of added water is reduced (1.0 max liquid/solid ratio instead of 4.0 min with the pulping method), and a clear increase in the average aspect ratio of refined fibers (21.2-22.6 instead of 16.3-17.9) is observed as well. These fibers present highly improved mechanical strengthening capability. This was demonstrated for rice straw-based fiberboards, in which pure non deteriorated lignin (e.g., Biolignin) was used as a binder (up to 50 MPa for bending strength and 24% for thickness swelling after 24 h immersion in water)²⁸.

The interest of TMC defibration in twin-screw extruder has also been confirmed with coriander straw²⁶. The aspect ratio of refined fibers varies from 22.9-26.5 instead of only 4.5 for simply ground fibers. 100% coriander-based fiberboards were obtained by adding to the extrusion-refined straws a cake from the seed as protein binder (40% in mass). Their flexural strength (up to 29 MPa) and especially their resistance to water (up to 24% thickness swelling) were significantly improved compared to panels made from simply crushed straw. Moreover, these panels do not emit formaldehyde and, as a consequence, they are more environmentally and human-health friendly than medium-density fiberboard (MDF) and chipboard²⁹ classically found in the market.

Similarly, panels entirely based on amaranth³¹ and sunflower³², combining extrusion-refined fibers from bark as reinforcement and seed cake as a protein binder, were successfully produced. They showed flexural strengths of 35 MPa and 36 MPa, respectively. However, their water resistance was found to be lower: 71% and 87%, respectively, for thickness swelling. Self-bonded panels based on extrusion-refined shives from oleaginous flax straw can also be obtained²⁷. In this case, it is the ligneous fraction, released during the twin-screw TMC defibration, that contributes to the self-bonding. However, hardboards obtained show a lower mechanical strength (only 12 MPa flexural strength), and very high thickness swelling (127%).

All the extruded fiber-based panels presented above can find industrial applications and are, therefore, sustainable alternatives to current commercial wood-based materials. According to the International Organization for Standardization (ISO) requirements^{48,49,50}, their specific applications will depend on their mechanical and water sensitivity characteristics.

In this paper, the procedure to extrude and refine lignocellulosic fibers before using them as mechanical reinforcement in renewable boards is described in detail. As a reminder, this process reduces the amount of water to be added in comparison to traditional pulping methodologies, and it is also less energy consuming²⁵. The same twin-screw machine can also be used for adding a natural binder to fibers.

More specifically, a detailed outline for conducting the twin-screw extrusion-refining of shives from oleaginous flax (*Linum usitatissimum* L.) straw is presented. The straw used in this study was commercially obtained. It was from the Everest variety, and the plants were cultivated in the South West part of France in 2018. In the same extruder pass, a plasticized

linseed cake (used as exogenous binder) can also be added in the middle of the barrel, and then mixed intimately to the refined shives along the second half of the screw profile. A homogeneous mixture having the form of a fluffy material is collected at the machine outlet. The one-step TMC operation is conducted using a pilot scale machine. Our goal is to provide a detailed procedure for the operators to conduct properly the extrusion-refining of shives, and then the cake addition. Following this operation, the obtained premix is ready for subsequent manufacture of 100% oleaginous flax-based hardboards using hot pressing.

Protocol

1. Prepare the raw materials

1. Use oleaginous flax shives, which are the result of a preliminary stage of mechanical extraction of the bast fibers from straw in an "all fiber" extraction device⁵¹. Use a vibrating sieve to remove short textile fibers that they may still contain.

NOTE: As the removal of these short textile fibers may be difficult, do not hesitate to repeat this sieving operation as many times as necessary. Here, the objective is to improve the flow of the oleaginous flax shives in the hopper of the weight feeder, and, therefore, facilitate their dosing before their introduction into the twin-screw extruder.

2. Use a plasticized linseed cake, obtained by destructuring/plasticizing of the proteins according to the methodology described by Rouilly et al.¹⁸.

NOTE: By doing so, the proteins show better thermoplastic and adhesive aptitudes.

3. Grind the agro-granulates of plasticized linseed cake using a hammer mill fitted with a 1 mm grid, and then sieve the grinded material obtained to retain only the particles smaller than 500 μm .

2. Check the proper functioning of the constant weight feeders and the piston pump

1. For the flow rates at which the operator works during production, chosen to avoid the clogging of the machine (15 kg/h for oleaginous flax shives (OFS), and from 1.50 kg/h to 3.75 kg/h for plasticized linseed cake), check the correspondence between the set value entered to the two constant weight feeders and the solid flow rates really distributed by these dosing devices.

NOTE: The actual solid flow rate is determined experimentally by weighing the mass of the solid distributed by the constant weight feeder for a known period of time (5 min). If there is a significant deviation between the set value and the actual measured flow rate, this may indicate a malfunction of the weigh feeder. To prevent this, the entire dosing unit should be thoroughly cleaned, with particular emphasis on the area where the weighing device is located. In fact, the cause of this type of malfunction is very often a poor cleaning of the device, as traces of previously used solids can be found in the smallest corners of the dosing unit. If the problem persists, it will then be necessary to check the correct gauging of the balance itself and, if necessary, recalibrate it.

2. Calibrate the piston pump to establish a relationship between the electrical power of the motor and the actual water flow rate distributed by the pump.

NOTE: For each tested electrical power, the actual water flow rate is determined experimentally by weighing the

mass of the water distributed by the piston pump for a known period of time (5 min). Five different electrical powers are tested to draw the calibration curve. The highest electrical power tested is chosen so that it delivers a higher water flow rate than that chosen during production.

3. Once the calibration of the pump has been carried out, check for the water flow rate at which the operator works during production (15 kg/h to avoid the clogging of the machine while preserving the length of the extrusion-refined fibers) the correspondence between the set value given to the piston pump for the engine power and the water flow rate actually distributed.

3. Prepare the twin-screw extruder

1. Correctly arrange the twin-screw extruder modules (AB1-GG-8D, FER, and ABF types) by connecting them one after the other (by means of two half clamps) in the correct order according to the machine configuration to be used:

1. Set up the configuration for which only the fiber defibration takes place (**Figure 1A**).
2. Alternatively, set up the configuration which is completed with the addition of the natural binder (**Figure 1B**).

NOTE: For both configurations, the first module is used for the introduction of oleaginous flax shives. This is a type AB1-GG-8D module, which has an 8D length, D corresponding to the screw diameter (i.e., 53 mm). The large upper opening of this module is primarily intended to facilitate the introduction of the shives. Modules 2 to 8 are temperature controlled. They are closed modules (type FER), except module 5 in the case of configuration (step 3.1.2), which is of

ABF type (i.e., module equipped with a side opening to ensure the connection of the side feeder used to force the introduction of the plasticized linseed cake inside the main barrel). The side feeder consists of two co-rotating and co-penetrating Archimedean screws of constant pitch and conjugated profile.

2. Position the water inlet pipe laterally at the end of module 2 to connect the piston pump to the machine.
3. Set aside the screw elements (**Figure 2**) that will be needed to set up the screw profile, either the one used for configuration (step 3.1.1) or the one used for configuration (step 3.1.2) (**Figure 3**).

NOTE: Make sure that these are the correct screw elements by carefully checking their type (T2F, C2F, C1F, CF1C, BB, or INO0), length, pitch (for the conveying and reverse screw elements), and their staggering angle (for the BB mixing blocks).

4. Set up the screw profile (**Figure 3**) by inserting the screw elements along the two splined shafts, from the first pair to the last one.

NOTE: The screw profiles used for the two configurations tested, are different and both result from prior optimization^{25,26,27}.

5. When assembling the screw profile, make sure that the threads of the screw elements just inserted on the splined shafts are always perfectly aligned with the previously assembled elements.
6. Once the entire screw profile is assembled, screw by hand the screw points at the end of the two shafts, completely close the barrel of the machine and then tighten the two screw points to the tightening torque recommended by the manufacturer (30 daN m for the

twin-screw extruder used in this study) using a torque wrench.

7. With the barrel of the machine partially reopened, i.e., with the shafts retracted into the barrel over a distance of approximately 1D, turn the screws at low speed (25 rpm max) to ensure that the entire screw profile is correctly fitted.

NOTE: In the case of incorrect installation of the screw elements (e.g., the misalignment for one of them), accelerated wear of the screw elements will be inevitably observed. When testing the rotation of both shafts with the barrel of the machine almost fully open, this results in the shafts touching each other at the point of the incorrectly positioned screw element.

8. Completely close the barrel of the machine so that both shafts are entirely trapped inside the barrel.
9. Once the barrel is closed, clamp it to the machine with half clamps, and make sure with the help of a level tester that the barrel is perfectly horizontal.

NOTE: If the barrel of the twin-screw extruder is not perfectly horizontal, this may lead to premature wear by abrasion of the screw elements and/or the inner walls of the barrel.

10. Position the peripherals (the weight feeders for the two solids to be introduced, and the piston pump for the water to be injected) at the required places along the barrel: above module 1 for the feeder used for the oleaginous flax shives, above the hopper of the side feeder (itself connected laterally to module 5) for the one used for the plasticized linseed cake (case of configuration (step 3.1.2) only), and at the end of module 2 for the water injection.

4. Carry out the twin-screw extrusion treatment according to configuration (step 3.1.1) or configuration (step 3.1.2)

1. From the supervision of the machine, enter the set temperatures of each of the modules and start the temperature control of the barrel: for configuration (step 3.1.1), 25 °C for the feeding module (module 1) and 110 °C for the following ones; for configuration (step 3.1.2), 25 °C for module 1, 110 °C for the refining zone (modules 2 to 4), and 80 °C for the premixing one (modules 5 to 8).

NOTE: The temperature control of the barrel is carried out separately from one module to another by (i) heating with two resistive half clamps fixed around each module, and (ii) cooling by circulating cold water inside the module. A 25 °C is privileged for the feeding module. For an efficient refining of the fibers, a 110 °C temperature is preferred. An 80 °C temperature is sufficient for the premixing operation. Since the refining and premixing zones are both located along several modules, all modules in the same zone are assigned the same set temperature.

2. Wait for the stability of the measured temperatures and make sure that these temperatures are equal to the set points.

NOTE: The measured temperatures are given on the control panel of the machine. In order to ensure a second control of these temperatures, it is also possible to measure them with an infrared thermometer at the level of each module along the barrel.

3. Slowly turn the screws (i.e., 50 rpm max).

NOTE: Premature abrasive wear of the screw elements and the inner walls of the barrel can occur if the screws turn too quickly while the machine is empty.

4. Gently feed the twin-screw extruder with water (5 kg/h flow rate).
5. Wait for around 30 s until water comes out at the end of the barrel.
6. Then, start to introduce the oleaginous flax shives in module 1 at a 3 kg/h flow rate, and wait (for around 1 min) for the solid to start coming out of the extruder.
7. Gradually increase (at least in three successive steps) the speed of the screws, then the water flow rate and finally the shives flow rate until the desired set points are reached: 150 rpm, 15 kg/h and 15 kg/h, respectively (**Table 1**).

NOTE: These set points were determined in previous studies and result from the optimization of the process^{25,26,27}.

8. Wait for the machine stabilization by following the evolution of the electrical current consumed by the engine over time (variation of the electrical current no more than 5% from the 125 A average value).
- NOTE:** The stabilization time is usually in the range of 10 to 15 min.
9. For configuration (step 3.1.2) only, start introducing the plasticized linseed cake at 0.50 kg/h once the machine has stabilized in amperage after the shives and water addition to the desired set values. Then, increase the flow rate of the plasticized linseed cake in at least three successive steps up to the desired set point (from 1.50 kg/h to 3.75 kg/h, which corresponds to values between 10% and 25% by mass in relation to the shives) (**Table 1**).
10. Once the electrical current consumed by the twin-screw extruder motor is perfectly stable, make sure that the temperature profile measured along the barrel is conform to the set values given by the operator, and then start

sampling the extruded shives for configuration (step 3.1.1) or the premix for configuration (step 3.1.2) at the outlet.

NOTE: In order to not clog the unit, the current drawn by the motor must always remain below its limit value (i.e., 400 A for the pilot scale twin-screw extruder used in this study). It should, therefore, be checked that this limit value is not reached during the entire flow ramp-up phase as well as during sampling. During production, if the cooling system of the machine is not able to maintain the temperature of at least one module at its set value, this may be the consequence of an inappropriate screw profile (i.e., too restrictive screw elements at this location), which causes a local self-heating of the treated material. It is then necessary to make sure, e.g., by means of a thermogravimetric analysis (TGA) of the solid being processed, that this temperature does not cause any fiber degradation.

11. During the entire sampling process, make sure that the machine feed is trouble-free by regularly checking the effective entry of solids and water into the barrel of the machine.

NOTE: A stable amperage of the current drawn by the motor of the twin-screw extruder during the entire sampling time is a confirmation of a stable feeding of the machine.

12. At the end of production, switch off the two solid dosing units and the piston pump.
13. Empty the machine while gradually reducing the speed of rotation of the screws to 50 rpm.
14. When nothing comes out of the barrel end, clean the inside of the barrel of the twin-screw extruder with plenty of water, introduced in large excess from module 1, while

the screws are still rotating at 50 rpm. Add water until the solid residues disappear completely at the outlet of the barrel. Then, stop the rotation of the screws and switch off the heating control of the machine.

5. Dry and condition the resulting extrudates (i.e., extrusion-refined shives or premix)

1. When the extrudates are not to be molded into fiberboards immediately after the twin-screw extrusion process, dry them with a hot air stream to a humidity between 8% and 12% before their conditioning. For this purpose, use a simple ventilated oven or, in the case of large quantities of extrudate to be dried, a continuous belt dryer.

NOTE: With such humidity, the extrudates can be conditioned without the risk of fungus or mold growth over time. Packaging should be carried out in perfectly sealed plastic bags, which should be stored in a dry place.

2. Dry the extrudates with hot air flow to a humidity between 3% and 4% when fiberboard molding takes place immediately after the twin-screw extrusion process.

NOTE: Previous studies showed that a moisture content of 3% to 4% of the solid to be hot pressed is ideal to limit degassing phenomena at the end of molding. When it occurs and it is not controlled, degassing can generate defects (e.g., blisters or cracks) inside the fiberboard, and these defects have a negative impact on its mechanical resistance^{26,27,31,32}. When hot pressing is carried out after the extrudates have been stored in airtight plastic bags at a moisture content of 8% to 12%, they should be dried further, i.e., up to 3%-4%, before molding.

6. Mold the fiberboards by hot pressing

NOTE: The operating conditions for hot pressing have been chosen on the basis of previous studies^{26,27,31,32}.

1. Pre-heat the mold. Then, position the solid material to be hot pressed inside the mold. Lastly, pre-heat this solid material for 3 min before applying the pressure.

NOTE: For all the fiberboards produced, the proportion of shives in the mix to be molded represents a mass of 100 g when the mold used is square in shape and with 15 cm sides.

2. Apply a pressure of 30 MPa with the raw shives, and 10 MPa, 20 MPa, or 30 MPa with the extruded ones (**Table 2**).

3. Set the mold temperature to 200 °C.

NOTE: Because the temperature greatly influences the quality (especially the bending properties) of the boards obtained^{9,26,27,28,31,32}, it is important to check the mold temperature with an infrared thermometer on both its male and female parts.

4. Set the molding time to 150 s.

5. Manufacture different fiberboards with different contents of plasticized linseed cake (from 0% to 25%) using the extrusion-refined fibers obtained through twin-screw extrusion via configuration (step 3.1.1) or one of the three premixes obtained via configuration (step 3.1.2) (**Table 1** and **Table 2**).

6. As references, also manufacture two additional fiberboards based on the raw OFS, one without the addition of exogenous binder (board number 11) and the other with the addition of 25% (w/w) of plasticized linseed cake (board number 12) (**Table 2**).

NOTE: For these two boards, the molding conditions are the same, i.e., 200 °C for the mold temperature, 150 s for the molding time, and 30 MPa for the applied pressure.

7. Condition and characterize the fiberboards

1. Once the fiberboards have been produced, place them in a climatic chamber at 60% relative humidity and 25 °C until a constant weight is achieved.

NOTE: The fiberboards will then be conditioned and stabilized in terms of humidity.

2. Once equilibrated, cut the fiberboards into test specimens.

NOTE: The most suitable tool for cutting fiberboards is a vertical band saw.

3. From the test specimens, proceed with the characterization of the fiberboards using standardized tests for bending properties (ISO 16978:2003 standard), Shore D surface hardness (ISO 868:2003 standard), internal bond strength (ISO 16260:2016 standard), and water sensitivity after immersion in water for 24 h (ISO 16983:2003 standard).

4. Compare the properties measured for the fiberboards with the recommendations of the French standard dedicated to the specifications for particleboards (NF EN 312) in order to determine their possible uses.

Representative Results

During the fiber refining of oleaginous flax shives using configuration (step 3.1.1), water was deliberately added at a liquid/solid ratio equal to 1.0. According to previous works^{25,26,27}, such a liquid/solid ratio better preserves the length of the refined fibers at the twin-screw extruder outlet than lower ratios, which simultaneously contributes to an increase in their average aspect ratio. Furthermore, the

amount of water added is low enough to eliminate any risk of machine clogging. In the absence of "free" water (i.e., water that would have been added in excess, and part of which would not have been absorbed by the fibers), it was, therefore, not necessary to position a filtration module at the end of the defibration zone. Following the extrusion-refining pre-treatment, the chemical composition of the extrusion-refined fibers was determined (**Table 3**). Logically, in the absence of liquid extract generation during the extrusion-refining pre-treatment, no significant difference in chemical composition was observed between the raw shives and the extruded ones. In terms of appearance, the extrusion-refined fibers have the form of a fluffy material (**Figure 4**, bottom left). This means that the extrusion process, in particular the high shear rate applied, contributes to a modification of the flax shives structure. This was first confirmed by the lower apparent and tapped densities of the extruded shives compared to the values obtained with the raw shives (**Table 4**). The morphological analysis of the fibers also confirmed this first observation as a very significant increase in their aspect ratio is also observed using a fiber morphology analysis device (**Table 5**).

When considering binderless boards from oleaginous flax shives molded using hot pressing, the TMC defibrating pre-treatment using twin-screw extrusion according to configuration (step 3.1.1) is of obvious interest. Indeed, a separation of lignins from cellulose and hemicelluloses inside extruded shives takes place. During hot pressing, lignins can thus be easily mobilized and used as a natural binder. In addition, with a higher average fiber aspect ratio than for raw shives, the particle size profile of the extrusion-refined fibers is more favorable in terms of their performance for mechanical reinforcement. This means that boards made from extruded fibers alone (board numbers 1, 3, and 7), i.e.,

without the addition of plasticized linseed cake as an external binder, are not only all three cohesive, but above all present significantly improved usage properties in comparison to the board obtained by hot pressing of the raw shives (board number 11) (**Table 6**). Although the board number 1 from the extruded shives is hot pressed at a pressure of only 10 MPa, it is even significantly better from the point of view of its mechanical performance than board number 11, which is molded from the raw shives, but at a pressure value three times higher (30 MPa). The advantages of the pre-treatment in the twin-screw extruder for the subsequent mobilization of the lignins as internal binder on the one hand, and for increasing the average fiber aspect ratio on the other hand, are thus clearly demonstrated. A comparison of the usage properties of board numbers 1, 3, and 7 also shows the beneficial effects of higher applied pressure during molding on these properties, whether it is the flexural strength, the Shore D surface hardness, or the water resistance of the material after immersion. As the pressure increases, the mobilization of the lignin-based binder is promoted²⁷. In the molten phase, its viscosity is reduced, and wetting of the fibers is optimized.

Using configuration (step 3.1.2), once the shives were defibrated, the plasticized linseed cake was also added directly into the twin-screw extruder and intimately mixed with the refined fibers in the second half of the screw profile. The plasticized linseed cake was added at contents between 10% and 25% (**Table 1**). Intimate mixing was obtained thanks to the use of two successive series of bilobe paddles (BB elements), mounted in staggered rows (90°). These are positioned at the level of modules 7 and 8 (**Figure 3**). When the plasticized linseed cake is added, the observed increase of total specific energy consumption is very small despite a higher filling of the machine: 1.35 ± 0.04 kW h/kg of dry

matter max instead of 1.28 ± 0.05 kW h/kg of dry matter in the case of configuration (step 3.1.1) for which the shives are defibrated but without the addition of exogenous binder. The CF1C reverse screw elements used for shives defibration are, therefore, the most restrictive elements of the screw profile. The mixing zone of the refined fibers and linseed cake, therefore, contributes in a small extent to the increase in the overall energy consumption of the machine.

The addition of the plasticized linseed cake to the extrusion-refined fibers results in a premix enriched with natural binder, which must be dried to a moisture content of between 3% and 4% before molding. Overall, this addition increases the flexural properties of the fiberboards obtained (**Table 6**). For an applied pressure of 10 MPa, the addition of 25% linseed cake leads to a 15% increase in the flexural strength of the material (comparison of board numbers 1 and 2). For a doubled pressure (20 MPa), an increase of 25% is observed when 10% flax-based binder is added (board number 4) and it rises to 53% when 17.5% of this binder is added (board number 5). Finally, for the highest forming pressure (30 MPa), the relative increase in bending strength is maximum (+12%) when 10% linseed cake is added (comparison of board numbers 7 and 8).

At the same time, the Shore D surface hardness and the water resistance of the fiberboards after immersion are largely independent of the plasticized linseed cake content in the premix. The application of a pressure of at least 20 MPa during hot pressing is still accompanied by a reduction in thickness swelling, regardless of the exogenous binder content. Under such forming conditions, the density of hardboards increases. Their internal porosity is then reduced, and the diffusion of water inside the material during immersion is thus reduced.

The role of exogenous binder played by the linseed cake in the premix is thus confirmed and explained by the presence of a significant content (estimated at 40.5% of its dry mass⁵²) of proteins with plastic and adhesive behavior. This role is also confirmed when the oleaginous flax protein-based binder is added to the raw shives. Indeed, with 25% of this binder (case of board number 12), the board obtained (**Figure 4**, top right) has a flexural strength of 10.6 MPa instead of only 3.6 MPa without binder (board number 11). However, this panel has a lower bending strength than all those based on the extrusion-refined fibers, illustrating the essential role played by the TMC pre-treatment of the shives.

Thanks to the combined action of defibration of the shives and the addition of an exogenous binder within the same twin-screw device, fiberboards with a bending strength of around 23 to 25 MPa are obtained. As an example, with the addition of 25% plasticized linseed cake to the premix and hot pressing of the latter by applying a 30 MPa pressure, the corresponding fiberboard (board number 10) shows a bending strength of 24.1 MPa, a flexural modulus of 4.0 GPa and an internal bond strength of 0.70 MPa (**Figure 4**, bottom right). Based on the recommendations of the French standard (NF) EN 312 (standard dedicated to the specifications for particleboards)⁵³, this board already meets the mechanical requirements of type P6 boards, i.e., boards working under high stress and used in dry environments. Only its thickness swelling after immersion in water for 24 h does not meet the requirements of this standard (78% instead of 16% max). A post-curing treatment (60 °C for 30 min, then 80 °C for 30 min, then 100 °C for 45 min, then 125 °C for 60 min, and finally 150 °C for 90 min before returning to room temperature for 225 min) of this material leads to a reduction in thickness swelling of up to 49%, simultaneously with an increase in flexural strength (25.8 ± 1.0 MPa). However, this

reduction in thickness swelling remains insufficient. For future work, other additional processes, e.g., coating, chemical, or steam treatment, after hot pressing should be tested to improve this dimensional stability parameter²⁷ to a greater extent. Another original solution could be the addition of hydrophobing agent(s), e.g., vegetable oil derivatives, to the premix directly in the twin-screw extruder. In addition,

as this optimal board may be used inside houses, its fire resistance will need to be evaluated before it is proposed to the market. Indeed, this characteristic is of key importance. If the fire resistance of this material proves to be insufficient, the addition of a fireproofing product to the premix directly in the twin-screw extruder should be considered before the panel is molded by hot pressing.

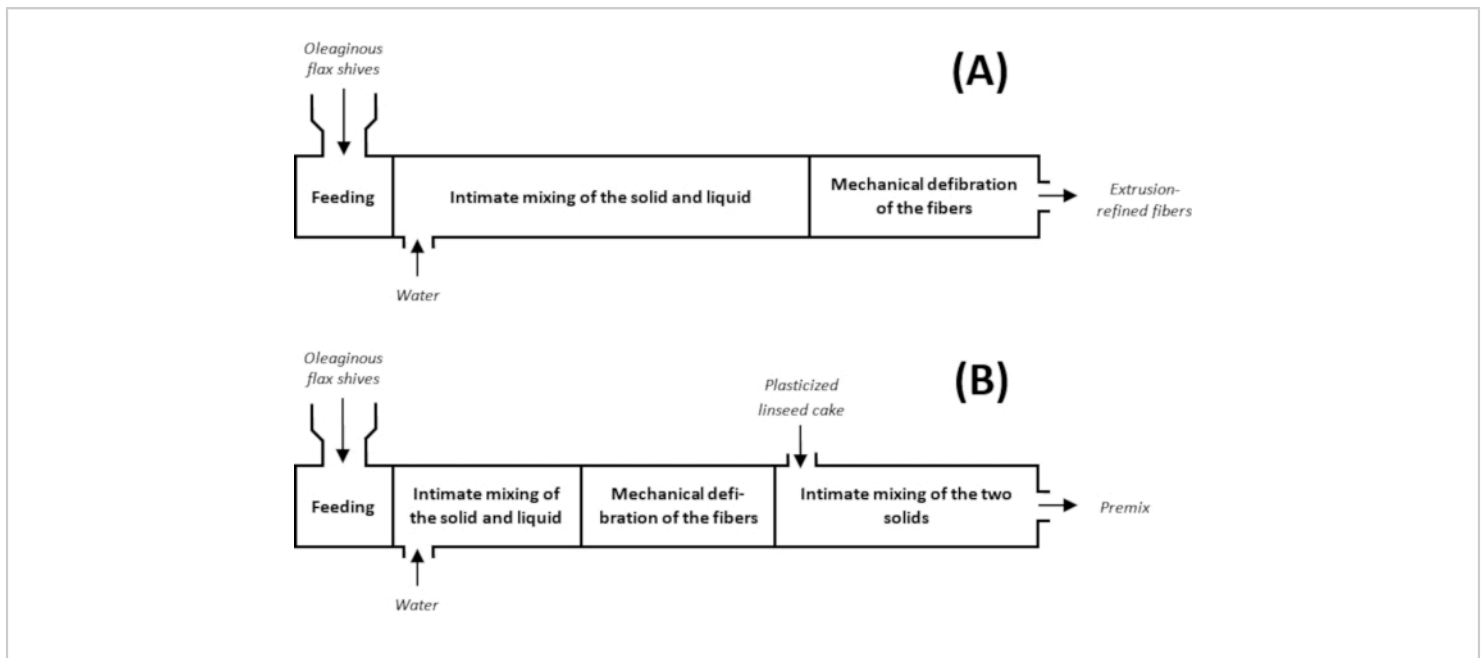


Figure 1: Simplified configurations of the twin-screw extruder used (A) for the only fiber refining of oleaginous flax shives, and (B) for the combined process in a single extruder pass, including the fiber refining of oleaginous flax shives, the addition of plasticized linseed cake, and then the intimate mixing of the two solids. For each of the two tested configurations, the successive unit operations are mentioned. [Please click here to view a larger version of this figure.](#)

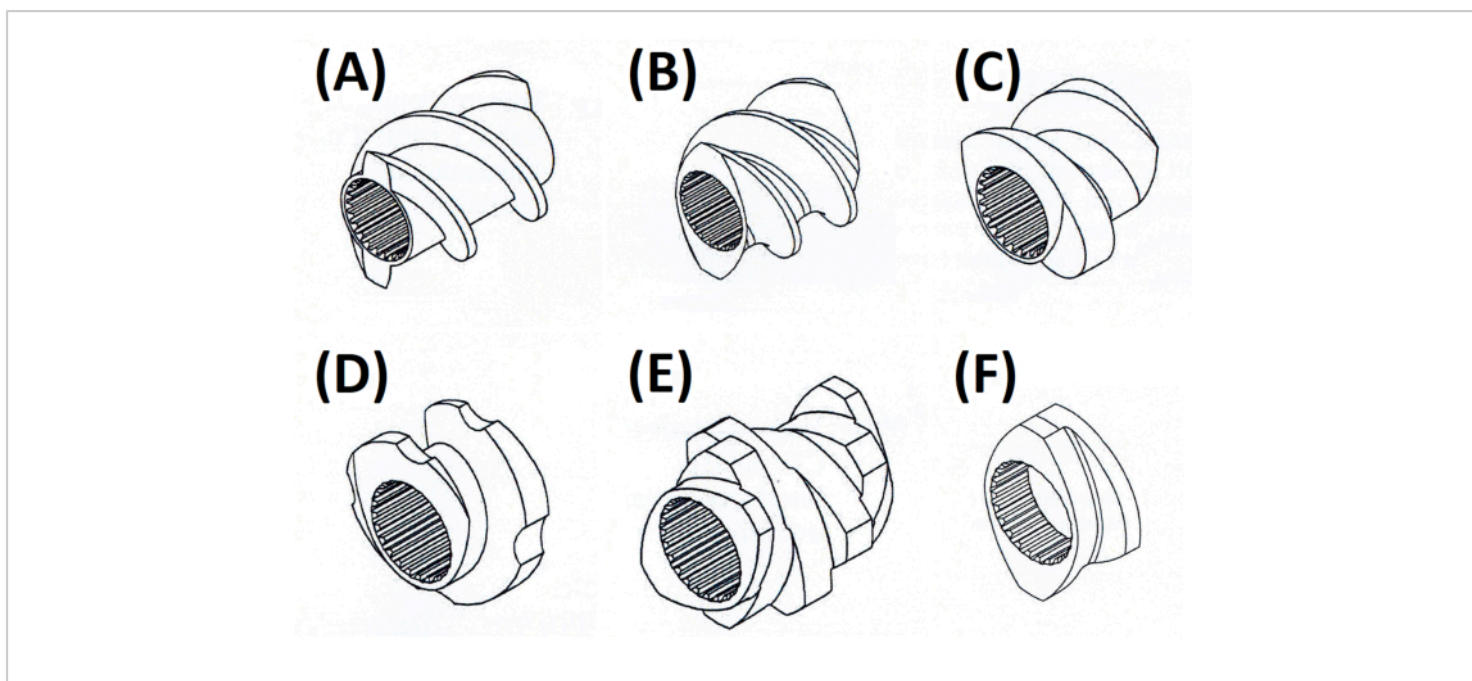


Figure 2: Type of screw elements used along the screw profiles: (A) T2F, (B) C2F, (C) C1F, (D) CF1C, (E) BB, and (F) INO0 screw elements. (A) T2F elements are trapezoidal double-flight screws used for their conveying action. Due to the trapezoidal shape of their threads, T2F elements are non-self-cleaning screws but have very good conveying and swallowing characteristics. They are, therefore, positioned in the feeding areas of the two solids used (i.e., oleaginous flax shives, and plasticized linseed cake). (B) C2F elements are conjugated double-flight screws also used for their conveying action. The shape of their threads is conjugated, which makes the C2F elements self-cleaning screws. They are positioned where the solid and liquid coexist. (C) C1F elements are single-flight screws. In comparison to C2F elements, these conveying screws have a wider thread crest. Therefore, they have a better thrust and a higher shear effect than C2F elements. (D) CF1C elements are conjugated cut-flight, single-flight screws with left-handed pitch. These reverse screw elements are the most restrictive and most important elements of the screw profile. They allow an intense mixing and mechanical shearing of the material as well as an increase of its residence time. CF1C screws are the place where the defibration of the fibers takes place. (E) BB elements are bilobed paddles. They allow a strong mixing effect on the material. They, therefore, promote an intimate mixing action that is particularly important for homogeneously impregnating the oleaginous flax shives with the added water on the one hand, and intimately mixing the extrusion-refined fibers and plasticized linseed cake on the other. (F) INO0 elements are linking elements between double- and single-flight screws. [Please click here to view a larger version of this figure.](#)

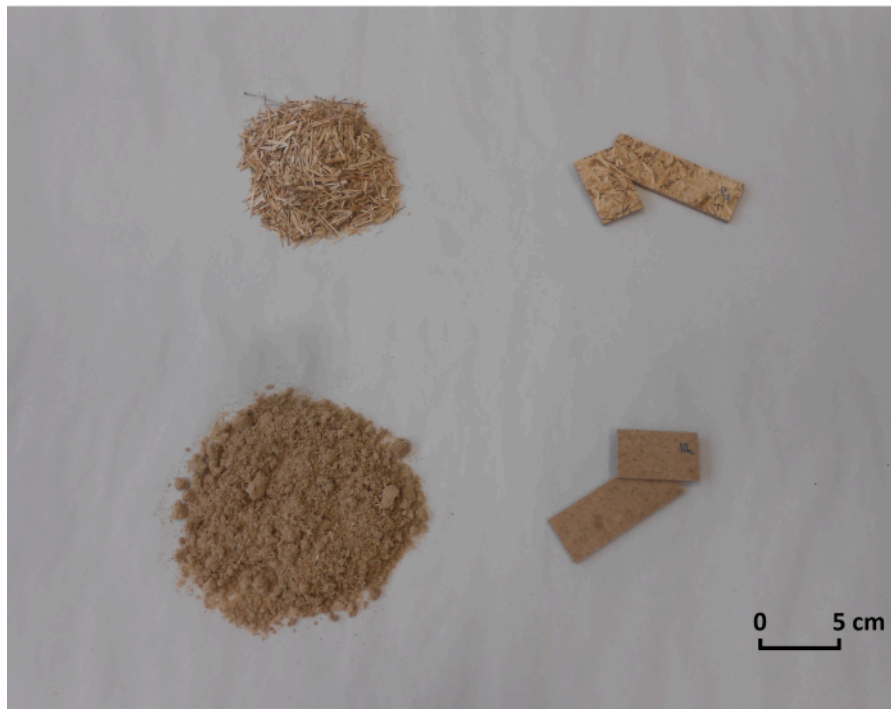


Figure 4: Photograph of OFS (top left) and ERF (bottom left) oleaginous flax shives, and board numbers 12 (top right) and 10 (bottom right). Board numbers 12 and 10 both contain 25% plasticized linseed cake. Board number 12 is made of the OFS raw shives whereas board number 10 originates from the P3 premix (i.e., contains the extrusion-refined fibers). [Please click here to view a larger version of this figure.](#)

Extrudate denomination	ERF	P1	P2	P3
Configuration	(3.1.1.)	(3.1.2.)	(3.1.2.)	(3.1.2.)
Twin-screw extrusion conditions				
Screw rotation speed (rpm)	150	150	150	150
Inlet flow rate of oleaginous flax shives (kg/h)	15.00	15.00	15.00	15.00
Inlet flow rate of plasticized linseed cake (kg/h)	0.00	1.50	2.63	3.75
Inlet flow rate of injected water (kg/h)	15.00	15.00	15.00	15.00

Table 1: Twin-screw extrusion conditions used for configurations (A) and (B). ERF, extrusion-refined fibers originating from configuration (step 3.1.1); P1, premix number 1 originating from configuration (step 3.1.2) and with 10% content (in proportion to the weight of shives) of plasticized linseed cake; P2, premix number 2 originating from configuration (step 3.1.2) and with 17.5% content (in proportion to the weight of shives) of plasticized linseed cake; P3, premix number 3 originating from configuration (step 3.1.2) and with 25% content (in proportion to the weight of shives) of plasticized linseed cake.

Fiberboard number	1	2	3	4	5	6	7	8	9	10	11	12
Raw material	ERF	P3	ERF	P1	P2	P3	ERF	P1	P2	P3	OFS	OFS plus 25% (w/w) of plasticized linseed cake
Mold temperature (°C)	200	200	200	200	200	200	200	200	200	200	200	200
Molding time (s)	150	150	150	150	150	150	150	150	150	150	150	150
Applied pressure (MPa)	10	10	20	20	20	20	30	30	30	30	30	30

Table 2: Molding parameters used for the manufacture of the fiberboards. OFS, oleaginous flax shives (i.e., raw shives not previously treated through twin-screw extrusion). Made of OFS and plasticized linseed cake, the solid mixture used for producing board number 12 was obtained mechanically using a double-helix mixer.

Material	OFS ²⁷	ERF
Moisture (%)	8.4 ± 0.2	8.3 ± 0.2
Minerals (% of the dry matter)	2.0 ± 0.1	2.0 ± 0.1
Cellulose (% of the dry matter)	45.6 ± 0.4	44.3 ± 0.4
Hemicelluloses (% of the dry matter)	22.4 ± 0.1	22.8 ± 0.1
Lignins (% of the dry matter)	25.1 ± 0.6	23.7 ± 0.5
Water-soluble components (% of the dry matter)	4.1 ± 0.1	4.3 ± 0.1

Table 3: Chemical composition of oleaginous flax shives before and after the extrusion-refining pre-treatment. The contents in moisture were determined according to the ISO 665:2000 standard⁵⁴. They were measured from equilibrated materials, i.e., after conditioning in a climatic chamber (60% relative humidity, 25 °C). The contents in minerals were determined according to the ISO 749:1977 standard⁵⁵. The contents in cellulose, hemicelluloses, and lignins were determined using the Acid Detergent Fiber (ADF) - Neutral Detergent Fiber (NDF) method of Van Soest and Wine^{56,57}. The contents in water-soluble compounds were determined by measuring the mass loss of the test sample after 1 h in boiling water. All measurements were conducted in duplicate. Results in the table correspond to the mean values ± standard deviations.

Material	Apparent density (kg/m ³)	Tapped density (kg/m ³)
OFS ²⁷	117 ± 5	131 ± 4
ERF	71 ± 1	90 ± 1

Table 4: Apparent and tapped densities of oleaginous flax shives before and after the extrusion-refining pre-treatment. The tapped density of oleaginous flax shives was measured in triplicate using a densitometer. The apparent density was obtained before compaction. Results in the table correspond to the mean values ± standard deviations. n.d., non-determined.

Material	Fibre length (μm)	Fiber diameter (μm)	Aspect ratio	Fines (%)
OFS ²⁷	5804 ± 4013	1107 ± 669	6 ± 6	n.d.
ERF	559 ± 27	20.9 ± 0.2	27 ± 2	56 ± 2

Table 5: Morphological characteristics of oleaginous flax shives before and after the extrusion-refining pre-treatment. The morphological analysis of raw shives (i.e., before the extrusion-refining pre-treatment) was performed by image analysis using a software from a scan of about 3,000 particles²⁷. That of the extrusion-refined shives was conducted using an analyzer for fiber morphology measurement and characterization. For these measurements, determinations were carried out in triplicate and, for each experiment, about 15,000 particles were analyzed. Results in the table correspond to the mean values ± standard deviations.

Fiberboard number	1	2	3	4	5	6	7	8	9	10	11	12
Bending properties												
Thickness (mm)	4.18 ± 0.07	5.03 ± 0.14	3.73 ± 0.11	3.88 ± 0.01	4.12 ± 0.02	4.56 ± 0.06	3.62 ± 0.12	3.81 ± 0.09	4.06 ± 0.12	4.37 ± 0.12	3.99 ± 0.07	4.69 ± 0.25
Density (kg/m ³)	1051 ± 16	1165 ± 78	1191 ± 59	1241 ± 34	1256 ± 41	1248 ± 37	1213 ± 54	1268 ± 17	1274 ± 23	1253 ± 32	1069 ± 19	1181 ± 40
Flexural strength (MPa)	11.6 ± 1.0	13.3 ± 1.4	16.6 ± 1.4	20.9 ± 2.2	25.5 ± 1.9	22.6 ± 2.1	21.7 ± 1.9	24.4 ± 1.8	23.5 ± 2.1	24.1 ± 2.5	3.6 ± 0.4	10.7 ± 0.9
Elastic modulus (MPa)	2474 ± 138	2039 ± 227	2851 ± 295	3827 ± 303	4272 ± 396	3806 ± 260	3781 ± 375	4612 ± 285	3947 ± 378	4014 ± 409	1071 ± 98	2695 ± 370
Shore D surface hardness (°)	70.7 ± 2.2	69.0 ± 3.0	70.6 ± 1.9	70.5 ± 2.2	70.3 ± 2.0	71.1 ± 1.8	69.0 ± 2.7	70.8 ± 2.0	70.0 ± 2.2	71.0 ± 1.7	61.4 ± 4.8	61.8 ± 3.6
Internal bond strength (MPa)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.70 ± 0.05	n.d.	n.d.
Water sensitivity after immersion in water during 24 h												
Thickness swelling (%)	139.5 ± 14.3	135.4 ± 10.9	76.1 ± 6.8	73.1 ± 1.8	82.3 ± 5.6	90.5 ± 3.9	64.0 ± 4.2	87.1 ± 5.6	100.1 ± 4.4	77.7 ± 2.2	159.9 ± 11.1	179.8 ± 16.3
Water absorption (%)	145.4 ± 10.0	143.1 ± 16.2	66.5 ± 6.3	65.2 ± 3.5	69.1 ± 2.2	83.0 ± 5.0	54.4 ± 1.6	59.8 ± 1.1	86.3 ± 6.7	63.3 ± 1.7	156.8 ± 5.9	150.1 ± 7.0

Table 6: Mechanical properties, thickness swelling, and water absorption of the fiberboards manufactured by hot pressing. The thickness and the density were determined by weighing the test specimens, and measuring their dimensions

using an electronic caliper. The bending properties were determined according to the ISO 16978:2003 standard⁵⁸. The Shore D surface hardness was determined according to the ISO 868:2003 standard⁵⁹. The internal bond strength was determined according to the ISO 16260:2016 standard⁶⁰. The water sensitivity after immersion in water (i.e., thickness swelling and water absorption) was determined according to the ISO 16983:2003 standard⁶¹. All determinations were carried out four times. Results in the table correspond to the mean values \pm standard deviations. n.d., non-determined.

Discussion

The protocol outlined here describes how to process the extrusion-refining of lignocellulosic fibers before using them as mechanical reinforcement in renewable boards. Here, the twin-screw extruder used is a pilot scale machine. With screws of 53 mm in diameter (D), it is equipped with eight modules, each 4D in length, except for module 1 that has an 8D length, corresponding to a 36D total length (i.e., 1,908 mm) for the barrel. Its length is long enough to apply to the processed material the succession of several elementary operations in a single pass, i.e., feeding, compression, intimate mixing between the fibrous solid and the added water, expansion, compression, intense shearing, and then expansion. Here, the extrusion-refining pre-treatment was successfully applied to shives from oleaginous flax straw. They constitute the residue collected after the mechanical extraction of technical fibers from oleaginous flax straw using an "all fiber" extraction device⁵¹. In the same twin-screw machine, it is also possible to add an exogenous binder to the defibrated lignocellulosic biomass immediately after the extrusion-refining step. The second half of the screw profile is thus devoted to the intimate blending of the refined fibers and this external binder. Here, this is a previously plasticized linseed cake that was used as additional binder. It has been added to the refined fibers using various rates (from 10% to 25% in proportion to shives). The resulting 100% oleaginous flax-based premixes were subsequently transformed into hardboards through hot pressing.

Due to the large number of elementary operations to be applied for configuration (step 3.1.2), which allows not only the refining of the fibers but also the addition of an external binder, the length of the barrel of the machine to be used is decisive for the success of the treatment. A barrel length of at least 32D is required, although lengths of 36D or even 40D are more appropriate. The expansion of the mixture transported between two successive zones of restrictive elements is then better and this favors exchanges between the constituents of the solid mixture and the water.

In addition, the screw profile is of key importance for the twin-screw processes^{2,3,4}. In particular, the restrictive areas (i.e., areas of intense mechanical work) must be chosen with the utmost care. Here, this leads to concerns with the reverse screw elements used for the defibration of lignocellulosic biomass, and the mixing elements needed for the impregnation of this biomass with water prior to defibration and subsequent intimate mixing of the refined fibers with natural binder. The typology of these elements (i.e., pitch of reverse screw elements, and width and stagger angle of mixing blocks), their respective lengths, and their positioning along the screw profile can be adapted to the formulation to be produced.

Similarly, the optimization of the operating conditions (i.e., inlet flow rates of solids, inlet flow rate of water, screw rotation speed, and temperature profile) will be necessary for any new formulation to be produced^{2,3,4}. In fact,

just like the screw profile, the operating conditions to be implemented will have to be adapted to the nature of each lignocellulosic biomass treated (e.g., distribution between cellulose, hemicelluloses and lignins, possible presence of other constituents, morphology, and hardness of the solid particles at the inlet, etc.). The filling rate of the twin-screw extruder can thus be adjusted to each new formulation with the aim of optimizing its residence time and increasing the productivity of the machine, while avoiding clogging.

It is, therefore, the filling rate of the twin-screw device that is the main limitation of the defibrating pre-treatment presented here. Depending on the nature of the raw material to be processed, the screw profile used, and the extrusion conditions applied (i.e., input flow rates of solids, liquid/solid ratio, and screw rotation speed), the mean residence time of the mixture inside the twin-screw tool is not the same. In order to increase the productivity of the machine, the objective is always to increase the flow of treated plant material as much as possible while preserving a sufficient quality of the TMC work carried out on it.

At the screw rotation speed used during the production and chosen as close as possible to the maximum rotation speed of the twin-screw machine used to increase its productivity, the machine can be overfilled if the incoming flows of solid material(s) and water become too high. It is, therefore, important for the operators to choose the optimum filling rate to ensure that the machine is not overfilled. To avoid such clogging, the twin-screw tool should be used for a sufficiently long time, i.e., at least half an hour. The stability of the electric current consumed by its motor during the production will be the confirmation of a machine that does not overfeed. Its control panel makes it easy to follow the evolution of the electric current over time. To conclude, the

twin-screw extrusion technology is, therefore, a versatile and high-performance tool to produce renewable fiberboards, free of synthetic resins. First of all, the continuous TMC defibration of lignocellulosic fibers, leading to an increase in their aptitude for mechanical reinforcement through an increase in the mean aspect ratio of the refined fibers, can be performed. The twin-screw tool can be considered as a credible alternative to other defibration methods classically used, i.e., a simple grinding, pulping processes, and steam explosion.

A recent study carried out on rice straw showed that this tool offers the possibility to better preserve the length of the fibers during their defibration than a method resulting from paper processes and involving a digestion stage followed by a defibration one²⁵. The same study also showed that the defibration conducted in a twin-screw extruder was less water consuming and can be performed at a lower cost. During twin-screw defibration, the release of lignins also contributes in part to the cohesion (by self-bonding) of the obtained fiberboards²⁷. These are called "self-bonded boards".

In the same twin-screw extruder and for greater compactness, it is also possible to continuously add an external binder to the previously refined fibers in variable proportions. This reduces production time and cost, as well as the dimensioning of the premix preparation unit. The overall process of pre-treatment of the fibers and preparation of the premix is thus greatly intensified before fiberboards hot-pressing. The addition of an exogenous binder also contributes to a substantial improvement in the usage properties of the materials obtained. This innovative process is, therefore, particularly versatile as it can be adapted to different lignocellulosic biomasses and different natural binders.

In the future, the excellent mixing capability of the twin-screw tool should be further exploited. For example, it could be used

to complement the premix of various functional additives, e.g., hydrophobing agents to improve the water resistance of fiberboards, antifungal agents, fire retardants, colors, etc., so as to provide fully functionalized premix ready for the final molding process.

Disclosures

The authors have nothing to disclose.

Acknowledgments

None

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Twin-Screw Extrusion Process to Produce Renewable Fiberboards

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Materials

Name	Company	Catalog Number	Comments
Analogue durometer	Bareiss	HP Shore	Device used for determining the Shore D surface hardness of fiberboards
Ash furnace	Nabetherm	Controller B 180	Furnace used for the mineral content determinations
Belt dryer	Clextral	Evolum 600	Belt dryer used for the continuous drying of extrudates at the exit of the twin-screw extruder
Cold extraction unit	FOSS	FT 121 Fibertec	Cold extractor used for determining the fiber content inside solid materials
Densitometer	MA.TEC	Densi-Tap IG/4	Device used for determining apparent and tapped densities of extrudates once dried
Double-helix mixer	Electra	MH 400	Mixer used for preparing the solid mixture made of the raw shives and the plasticized linseed cake for producing board number 12
Fiber morphology analyzer	Techpap	MorFi Compact	Analyzer used for determining the morphological characteristics of extrusion-refined shives
Gravimetric belt feeder	Coperion K-Tron	SWB-300-N	Feeder used for the quantification of the oleaginous flax shives
Gravimetric screw feeder	Coperion K-Tron	K-ML-KT20	Feeder used for the quantification of the plasticized linseed cake
Hammer mill	Electra	BC P	Crusher used for the grinding of granules made of plasticized linseed cake
Heated hydraulic press	Pinette Emidecau Industries	PEI 400-t	Hydraulic press used for molding the fiberboards through hot pressing
Hot extraction unit	FOSS	FT 122 Fibertec	Hot extractor used for determining the water-soluble and fiber contents inside solid materials

Image analysis software	National Institutes of Health	ImageJ	Software used for determining the morphological characteristics of raw shives
Oleaginous flax straw	Ovalie Innovation	N/A	Raw material supplied for the experimental work
Piston pump	Clextal DKM	Super MD-PP-63	Pump used for the water quantification and injection
Scanner	Toshiba	e-Studio 257	Scanner used for taking an image of raw shives in gray level
Side feeder	Clextal	E36	Feeder used to force the introduction of the plasticized linseed cake inside the barrel (at the level of module 5) for configuration (b)
Thermogravimetric analyzer	Shimadzu	TGA-50	Analyzer used for conducting the thermogravimetric analysis of the solids being processed
Twin-screw extruder	Clextal	Evolum HT 53	Co-rotating and co-penetrating pilot scale twin-screw extruder having a 36D total length (D is the screw diameter, i.e., 53 mm)
Universal oven	Memmert	UN30	Oven used for the moisture content determinations
Universal testing machine	Instron	33R4204	Testing machine used for determining the bending properties of fiberboards
Ventilated oven	France Etuves	XL2520	Oven used for the discontinuous drying of extrudates at the exit of the twin-screw extruder
Vibrating sieve shaker	RITEC	RITEC 600	Sieve shaker used for the sieving of the plasticized linseed cake
Vibrating sieve shaker	RITEC	RITEC 1800	Sieve shaker used for removing short bast fibers entrapped inside the oleaginous flax shives