Wet-laid technique with Cyperus esculentus: Development, manufacturing and characterization of a new composite

A. Carbonell[1], T. Boronat[1*], E. Fages[2], S. Girones[2], E. Sanchez-Zapata[3], J.A. Perez-Alvarez[3], L. Sanchez-Nacher[1], D. Garcia-Sanoguera[1]

[1] Instituto de Tecnología de Materiales (ITM), Universitat Politècnica de València (UPV), Plaza Ferrándiz y Carbonell 1, 03801 Alcoy (Alicante) Spain
[2] Textile Research Institute (AITEX), Plaza Emilio Sala 1, 03801 Alcoy (Alicante) Spain
[3] IPOA Research Group, Agrofood Technology Department, Orihuela Polytechnical High School, Miguel Hernández University, Ctra. a Beniel Km. 3.2, 03312 Orihuela (Alicante) Spain

* Correspondence to T. Boronat
E-mail: tboronat@dimm.upv.es
Tel.: +34 96 652 84 68

Abstract

Biobased composites were fabricated with waste of tiger nut natural fibers, different binder fibers (lyocell and cotton) and thermo-bonding fibers (PLA, HDPE, PA6-CoPA). These composites were processed by wet-laid process and hot-press molding process. The obtained composites were characterized by flexural, hardness and Charpy impact tests.

The internal structure of the composite was analyzed by SEM observing a significant heterogeneity. The high fiber content (80% wt) and the lack of a continuous matrix phase causes mediocre mechanical response of the material. Thermo-bonding fibers kind are more influential than the binder fibers. The best mechanical responses were obtained with additions of PLA fibers thermo-bonding fibers. Flexural modulus was maximum (865 MPa) for 80%wt tiger nut/10% wt binder fiber / 10% PLA fiber composite.

Keywords: Polymer-matrix composites; Particle-reinforcement; Thermomechanical; Thermal properties; Tiger nut

1. Introduction

The depletion of oil resources and the stricter environmental regulations, are acting synergistically to boost new materials and products which are compatible with the environment and are independent of fossil fuels. In the field of composite materials widely used in engineering, the increasing environmental concerns have encouraged the replacement of synthetic fibers by natural fibers. Natural fibers, as the name suggests, are extracted from natural resources, and may be from mineral, vegetable or animal source. In recent years, there has been substantial growth in the research, development and application of the so-called "biobased composites" or NFRP (Natural Fiber Reinforceed Plastics). Such composites are applied in major industrial application sectors such as construction, automobile, interior design, packaging, toy, etc. These materials show interesting characteristics of sustainability, recyclability and even biodegradability. The NFRP offer additional advantages such as low density, high strength/weight ratio, no brittle fracture, and good acoustic and thermal insulation properties. These characteristics make them interesting materials mainly for the construction industry and the automobile [1-5].

On the Mediterranean coast, due to the special nature of the climate, a native variety called "Cyperus esculentus L." is grown. This herbaceous plant produces an edible tuber known as tiger nut as shown in Figure 1 (a). Its most appreciated use is for the development of a milk-like beverage called “Horchata”. For the production of this beverage, the tuber is ground, and after its processing a wet solid waste is generated without any kind of application, as shown in Figure 1 (b). For the producers, the residue of ground tiger nut in making horchata is a problem because it generates large volumes without any economic value. Also, such residue is capable of rapid fermentation. This involves costs for the producer, and environmental damage.

The tiger nut is also cultivated in areas with temperate climates such as Brazil, Chile, and the states of Louisiana, Florida, Missouri, New Mexico in USA [6-8].

Figure 1. (a) Tiger nut tuber. (b) Waste generated in the manufacture of horchata.

One way to minimize this problem, is the revaluation of this lignocellulosic waste using it as natural fillers on "biobased composites". In order to incorporate very high levels (≥ 50%) of this residue in a polymer matrix it is planned the use of compression molding technique from several layers of nonwoven with high content in tiger nut. Nonwoven materials are characterized by very low density, to be porous and be formed of fibers or filaments which are joined by chemical, thermal or mechanical forming network-like structures. Its production cost is low as well as being very versatile materials, easy to form, recyclable, flexible, etc. They are widely used as filters, hygiene and personal care, automotive components, elements for thermal and acoustic insulation, etc. and new products which use this type of tissue x appear every day.

In this work, wet-laid nonwoven processing technology has been chosen in order to obtain fabric using tiger nut waste. The traditional technique of paper processing has been applied and the tiger nut waste in an aqueous medium became a nonwoven sheet.

The Wet-Laid technology is used in order to develop nonwoven composites of all fibers which have the ability to be dispersed in fluids. The Wet-Laid technology enables the production of products with very good homogeneity, versatility in product finish and high production.

In general, the process follows these steps: Fiber dispersion in water, continuous formation of the nonwoven filtering on a mesh, consolidation and drying of the nonwoven. The dispersion of the fibers in a liquid is extremely important in the production method for the nonwoven Wet-Laid technology. Raw materials must be able to be separated homogeneously in loose fibers to form a slurry and remain evenly distributed during its transport to the formation of nonwoven. The mixture of fibers suspended in an aqueous medium is deposited on a porous conveyor belt which allows the pass of water. The conveyor belt transports the nonwoven to the next stage where the material is consolidated by a thermal process.

As a result, it is obtained a uniform sheet of nonwoven fibers that have not been damaged during processing distribution. In addition this technology is environmentally friendly and enables the production of nonwovens with high volume of fiber and low quantity of matrix, so the final costs are also reduced because the polymeric matrices are expensive [9-14].

These nonwovens are the base of laminated composites by hot pressing of several layers. During the processing, pressure and temperature are applied and the thermo-bonding materials that were added merge in order to unite the composite. Such composites do not have a continuous matrix and are characterized by high amounts of fiber, sometimes they are greater than 90 wt% fiber, but the mechanical behavior is adequate for many technical applications that not require high resistance [15-18].

Thus, the main objective of this study is to obtain and characterize mechanically the composite material obtained by hot-press molding. The material is made of layers of nonwoven which are rich in tiger nut waste and are obtained using a wet laid process. The influence of different binding fibers and thermo-bonding over the properties of the material will also be determined. It is intended to determine whether the biocomposite rich in tiger nut residue is suitable for application as technological material.

2. Experimental

Materials

The tiger nut residue from the production of horchata has been provided by the University Miguel Hernández (Elche, Spain). In order to obtain the nonwoven it is used the wet original waste (without drying) since in the wet laid technology, in the first stage it is mixed with water. The moisture of the tiger nut waste is about 80%, so it is plasticene like. Particles of this residue have an average size of 1.5mm with polygonal shapes, as shown in Figure 2.

Figure 2. Tiger nut particles from the manufacture of "Horchata" (8X).

Cotton fibers and Lyocell fibers have been used as binding materials in order to give cohesion to the tiger nut fiber sheet, both supplied by STW Fibers (Schwarzwälder Textil-Werke GmbH, Schenkenzell, Germany). The cotton fibers are natural fibers with reference FB1 / 150 and 1.3 mm in length. Lyocell fibers are spun cellulose fibers, reference GL1.7 / 4 and a length of 4 mm. It is advisable that the length of the fibers should not exceed 15 mm, since otherwise it is very likely that the dispersion thereof is not adequate and a nonuniform sheet will be obtained with a large number of defects.

Different materials have been used as thermo-bonding component of the composite, such as high density polyethylene (HDPE), polylactic acid fibers (PLA) and bicomponent fibers Polyamide6/Copolyamide.

HDPE has been acquired from the company STW Fibers. The sales reference is FPE 910F. The fiber length is 1.3-2mm and 30μm in diameter, with a melt temperature of 132ºC.

PLA fibers were supplied by the company Trevira GmbH (Germany), and its reference is 260, the fiber length is 6mm and melting temperature is 160ºC.

The bicomponent fibers PA6/CoPA have been provided by the company EMS-GRILTECH GmbH (EMS-CHEMIE, Neumünster, Germany), whose sales reference is BA 140. They have a core/shell structure in which the fiber core is composed by PA6 and sheath or outer layer is formed by CoPA. The shell or sheath has a melting point of 135ºC with 6 mm fiber length.

In addition, flax fiber has been used as reference material in order to compare the characteristics of the obtained composite with tiger nut fiber. STW Fibers also provides the Commercial flax fibers, reference F513, with 6mm in length and with a diameter between 10 and 500μm.

<u>Processing of nonwoven sheets</u>

The formulations proposed for the study of the composite materials with tiger nut are shown in Table 1. Each one of these formulations was processed by wet laid technique following the steps shown in figure 3, obtaining nonwoven sheets rich in natural fiber.

Table I. Materials for manufacturing of the nonwoven sheets.

SAMPLE #	**NATURAL**		**BINDER**		**THERMO-BOUNDING**			**GRAMMAGE g.m^{-2}**
	TIGER NUT FIBER %wt	**FLAX FIBER %wt**	**LYOCELL %wt**	**COTTON %wt**	**PLA %wt**	**PA6/CoPA %wt**	**HDPE %wt**	
1	80	-	10	-	10	-	-	342
2	80	-	10	-	-	10	-	307
3	80	-	10	-	-	-	10	352
4	80	-	-	10	10	-	-	295
5	80	-	-	10	-	10	-	314
6	80	-	-	10	-	-	10	
7	-	80	10	-	10	-	-	422
8	-	80	10	-	-	10	-	428
9	-	80	10	-	-	-	10	409
10	-	80	-	10	10	-	-	348
11	-	80	-	10	-	10	-	336
12	-	80	-	10	-	-	10	360
13	40	40	10	-	10	-	-	345
14	40	40	10	-	-	10	-	395
15	40	40	10	-	-	-	10	361

16	40	40	-	10	10	-	-	336
17	40	40	-	10	-	10	-	324
18	40	40	-	10	-	-	10	

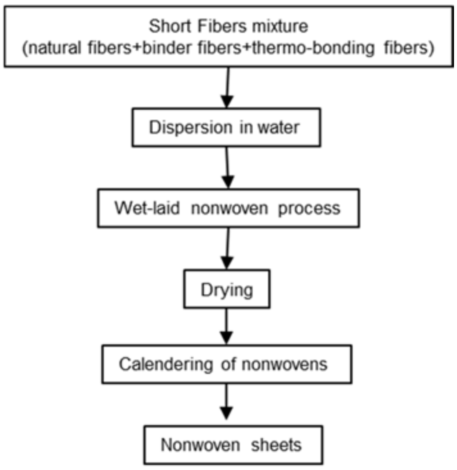


Figure 3. General outline of the process of obtaining nonwovens with wet laid technology.

Each one of the fiber blends proposed in the study, with a dry weight of 800 g, is dispersed in water (1g l^{-1}) in a high shear stirrer in order to separate and uniformly disperse the fibers. The used stirrer was provided by PILL Nassvliestechnik (PILL Nassvliestechnik GmbH, Reutlingen, Germany). It has a capacity of 800 liters, and stirring was performed at 2400 rpm for 10 minutes. Once the fibers are well dispersed in the aqueous medium, the mixture was transported to the area of formation of the nonwoven using of hydraulic pumps.

The fiber/water mixture is deposited on a porous conveyor belt with a feed rate of 1 $m.min^{-1}$ which acts as a filter. The porous belt drains water and the fibers remain deposited over the belt forming the nonwoven sheet. The consolidation of the obtained sheet is done in two steps: The first one through a drying oven, and the second one passing the material between two hot rollers that apply pressure and heat to the nonwoven. The drying oven used was supplied by Tacome SA, mod. SDT-600 (Tacome SA, Valencia, Spain). In this first drying step the nonwoven is maintained at 180°C for the PLA blend, 150ºC for PA6/CoPA, and the HDPE at 152ºC all of them for 10 minutes long. The second process is the calendering of the nonwoven through rollers which are maintained at 200 ºC with a linear fixed pressure on the sheet of 0.124 MPa m. The calendering is performed in an equipment provided by Tacome SA, mod. CL-600 (Tacome SA, Valencia, Spain). After the consolidation phase, the nonwoven sheet is rolled up on a machine supplied by the same company Tacome SA mod. EN-600, as shown in Figure 4. The average thickness of the obtained nonwoven is 1.18 mm, and the grammage for each one of the different formulations is shown in Table I.

Figure 4. Nonwoven sheet rich in natural fiber rolled up, obtained by wet laid technology.

Manufacture of the composite materials

Composite materials are processed by hot pressing. For the hot pressing 8 sheets sized 12.5x12.5 cm^2 are superimposed inside the press in order to obtain laminates with 3 mm thickness. The press is set at different temperatures depending on termoconformante used (165ºC PLA, 135ºC PA6/CoPA and 150ºC HDPE) and it is set at a constant pressure of 8T for 8 min. After that the mold is cooled for 20 min at room temperature before demolding the biocomposite. The hot molding press used was supplied by Robima SA (Valencia, Spain).

Characterization of the biocomposites

The bending tests were performed in a universal testing machine IBERTEST ELIB 30 (SAE Ibertest, Madrid, Spain) according to the UNE EN ISO 178, applying a constant load speed of 2 $mm \cdot min^{-1}$ and a load cell of 5 kN. The specimens used were sized 10x80x3 mm^3. Impact tests were performed using a 6J Charpy pendulum manufactured by Metrotec (Metrotec SA, San Sebastian, Spain) following the ISO 179:1993. Biocomposites hardness is determined by a durometer Shore D scale, mod. 673D (J. Bot Instruments SA, Barcelona, Spain) according to the guidelines of the UNE-EN-ISO 868.

The scanning electron microscope SEM used for the morphological characterization of the fracture of the biocomposites obtained by hot pressing, was supplied by FEI Company, model Phenom (FEI Company, Eindhoven, Netherlands). Before the morphological study, samples are coated with a layer of gold-palladium using a sputter coater Emitech mod. SC7620 (Quorum Technologies, UK)

3. Results and discussion

Nonwoven morphology

The different biocomposites proposed in this work (Table I) have been obtained in order to determine the influence of different binding and thermo-bonding fibers on the properties thereof. A first series with high content in tiger nut waste (80 wt%) has been produced which is the objective of the work. A second series is formulated with 80% wt of flax as reference material. The bio-component rich in flax lets to compare the mechanical properties and also validate if the composite of tiger nut is optimal for technological applications. The third group (40% wt tiger nut + 40 wt% flax) is used as a comparative reference of the type of natural fibers. It should be noticed that two proposed samples have not been analyzed. The sample number 6 (80% wt tiger nut/10% wt cotton/10% wt HDPE) and the sample number 18 (40% wt tiger nut+40% wt flax/10% wt cotton/10% wt HDPE) lacked consistency after the wet laid process. The sheet obtained with these compositions with cotton and HDPE did not consolidated in the drying process and it breaks down. This problem occurs when HDPE is used as thermoplastic fiber. It may be because the HDPE is in the form of very short fiber, almost powder and hinders the bonding entanglement with tiger nut particles. The other two thermomolders, PLA y PA6/CoPA, are in the form of threads and it is produced a better interaction with the different fibers.

The structure in the obtained sheets by wet laid is characterized by a very heterogeneous appearance, without continuity in matrix form. Due to the use of high contents of natural fiber/particle and low amounts of thermoplastic fiber, the nonwoven consolidation occurs mainly by mechanical entanglement of binder fibers-thermo-bonding fibers together with bonding partial melting of the thermo-bonding fibers, as shown in the examples of Figure 5. Micrograph (a) shows the irregular particle shape of the tiger nut residue "hooked" on the network formed by the lyocell fibers and bicomponent in a nonwoven with 80 wt% waste of tiger nut. Micrograph (b) shows in detail the junctions between the fibers by heat fusing thereof. Comparatively, the SEM image (c) of nonwoven with 80% wt of flax fiber, presents fibers of different thicknesses irregularly distributed. Micrograph (d) shows with higher magnification the "links" between fibers, they are caused by thermal fusion of the thermo-bonding fibers during the manufacturing of the nonwoven. From the structural point of view, there is not difference in spatial arrangements of the phases of the composite with tiger nut or with flax. They are structurally equivalent.

This type of irregular structures without internal continuity determine the behavior of the biocomposites obtained with these nonwovens. The few unions by thermal fusion of the thermo-bonding fibers give consistency and consolidate the nonwoven for its processing, but also influence in a poor mechanical response of the biocomposite.

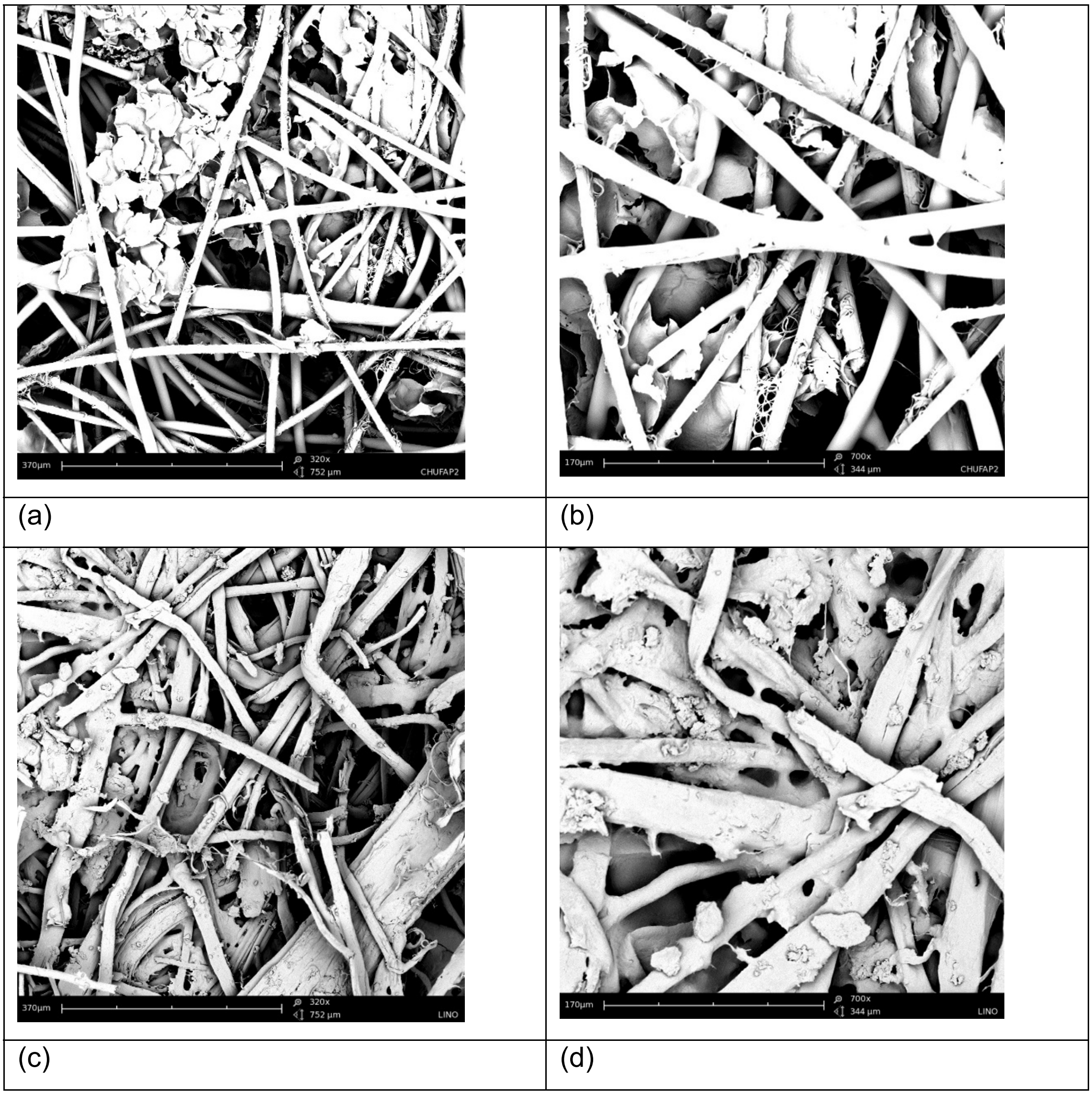


Figure 5. SEM micrographs of the nonwoven surface. (a) 80%wt tiger nut + 10%wt Lyocell+10%wt PA6/CoPA, 320X. (b) 80%wt tiger nut + 10%wt lyocell+10%wt PA6/CoPA, 700X. (c) 80%w flax fiber + 10%wt lyocell +10%wt HDPE, 320X. (d) 80%w flax fiber + 10%wt lyocell +10%wt HDPE, 700X

<u>Biocomposites flexural properties</u>

Figure 6 depicts the flexural modulus of the bicomposites with high contents of natural fibers (80%wt tiger nut, 80%wt flax, 40%wt tiger nut+ 40%wt flax) for different binders and thermo-boundings used. The behavior for each one of the three systems is similar for each mixture binder/thermo-bonding. The higher elastic modulus is obtained using PLA as thermo-bounding, and their values are very similar when binder fiber varies:

Lyocell or cotton. The Lyocell/PLA composites present high values of elastic modulus: 861.8; 615.1 and 677.0 MPa for tiger nut; flax and tiger nut flax respectively.

When PA6/CoPA or HDPE are used as thermo-bounding nonwoven fiber, the flexural modulus values obtained are half or even less of those for the PLA with values around 390 MPa for HDPE and 214 MPa for PA6/CoPA. From the viewpoint of the types of natural fibers, the tiger nut/PLA exhibits the maximum value of flexural modulus, when it reaches 861.8MPa. Its flexural modulus is 28% higher for tiger nut/Lyocell/PLA that obtained in flax/Lyocell/PLA. In the case of using cotton as a binder, this parameter is 18% higher for tiger nut too.

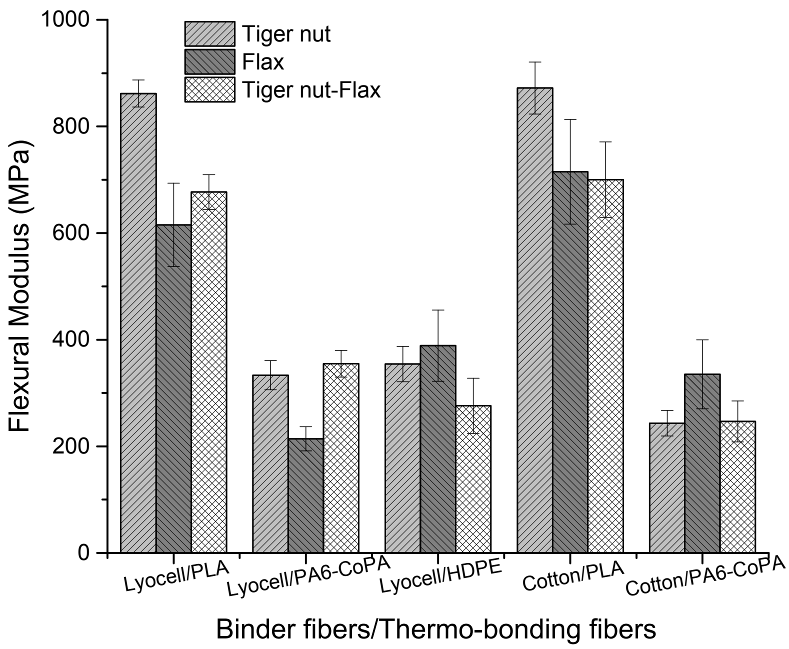


Figure 6. Flexural modulus of the biocomposites with high natural fiber content (80% wt), based on different binders and thermo-boundings.

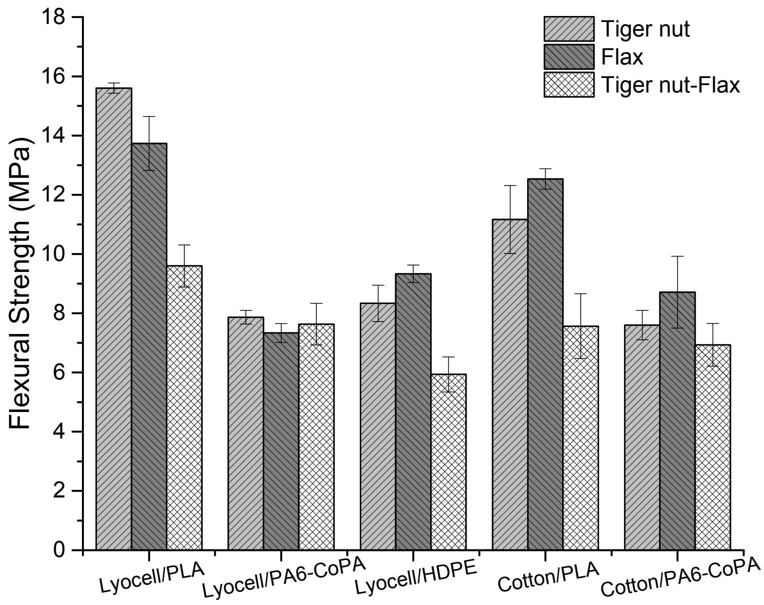


Figure 7. Flexural strength of the biocomposites with high natural fiber content (80% wt), based on different binders and thermo-boundings.

Figure 7 shows the flexural strength of the biocomposites with high natural fiber content in accordance with different binders and thermo-boundings used. The behavior for each of the three systems is similar for each binder/ thermo-bounding mixture. The greatest resistance against bending are obtained using PLA fibers, and the lowest for the bicomponent fibers PA6/CoPA. The Flexural strength presents similar values to the biocomposites of tiger nut and flax, when Lyocell is used as binder it is obtained values of flexural strength of 15.6 and 13.7 MPa respectively. When cotton is used as binder the flexural strength is lower reaching values of 11.2 MPa for tiger nut and 12.5 MPa for flax. . However, for laminated tiger nut+ flax the flexural strength is lower, particularly 9.6 and 7.6 MPa for the Lyocell/PLA and cotton/PLA respectively.

From the viewpoint of the types of natural fibers, the tiger nut/Lyocell/PLA reach the maximum value of flexural strength. Its flexural modulus is 12% higher than that one obtained in flax/Lyocell/PLA (15.6 MPa). When cotton is used as a binder, this parameter is 10% higher for flax/Lyocell/PLA. It can be concluded that the response to bending stresses in the biocomposites studied, the most influential component is the thermo-bonding used. The use of fibers of PLA provides maximum performance against bending. It has to be considered that the flax fiber bicomposites are used as benchmark in this paper. Analyzing the graphs it can be determined that laminates with high contents of tiger nut waste can perfectly replace the rich flax fiber composites since their behavior is very similar.

Table II. Values of Shore D hardness of the biocomposites of high content of natural fiber (80% wt), based on different binders and thermo-bondings..

	Natural Fibers					
	Tiger nut 80% wt		Flax 80% wt		Tiger nut 40% wt + Flax 40% wt	
Binder/thermo-bonding Fibers	**Shore D**	**SD**	**Shore D**	**SD**	**Shore D**	**SD**
Lyocell/PLA	46.3	2.4	45	1.5	47.6	2.3
Lyocell/PA6-CoPA	45.0	0.5	45.3	2.0	44.0	1.7
Lyocell/HDPE	47.0	0.5	38.3	0.8	45.0	2.5
Cotton/PLA	55.3	1.1	55	2.5	45.0	2.1
Cotton/PA6-CoPA	47.3	1.0	39.3	1.7	37.3	0.3

The hardness obtained for the three systems biocomposite tested: 80%wt Tiger nut, 80%wt Flax, 40%wt Tiger nut+40%wt Flax, is shown in Table II. The Shore D hardness obtained are very similar for all tested samples. Only it is observed a slight increase in hardness with the use of PLA as thermo-bonding.

<u>Biocomposites impact properties</u>

The results obtained for the biocomposites for the Charpy impact tests are shown in figure 8. In it is depicted the energy absorbed in the impact test, for the proposed natural fibers: 80%wt Tiger nut, 80%wt Flax, 40%wt Tiger nut+40%wt Flax, based on the various binders and thermo-bonders. Despite the fiber content are very high (80% wt), they are not embedded in a continuous matrix (Figure 9), so the the biocomposites studied absorb low impact energy. Continuous matrices absorb most of the energy before failure, partly because their possibility of deformation when forces are transmitted to the fiber.

The biggest impact energy absorption corresponds to the composites with PA6/CoPA bicomponent as thermo-bonding, which values range between [15.9 and 14.4] KJ m^{-2}. These values are above twice the value presented using PLA. The impact resistance is similar in biocomposites with tiger nut fiber, flax fiber or mixture of the both. HDPE fiber has an intermediate impact behavior regarding the two previous ones, with values of impact energy between [11.6 and 10.5] KJ m^{-2}. The most fragile composites under impact conditions are those with PLA as thermo-bounding. In such cases there is no significant difference between different natural fibers.

Regarding binder fiber used in the nonwoven, Lyocell and cotton, there is no clear influence regarding the impact resistance of the composite. The obtained values are very similar for the two different binders, maintaining the same thermo-bonding.

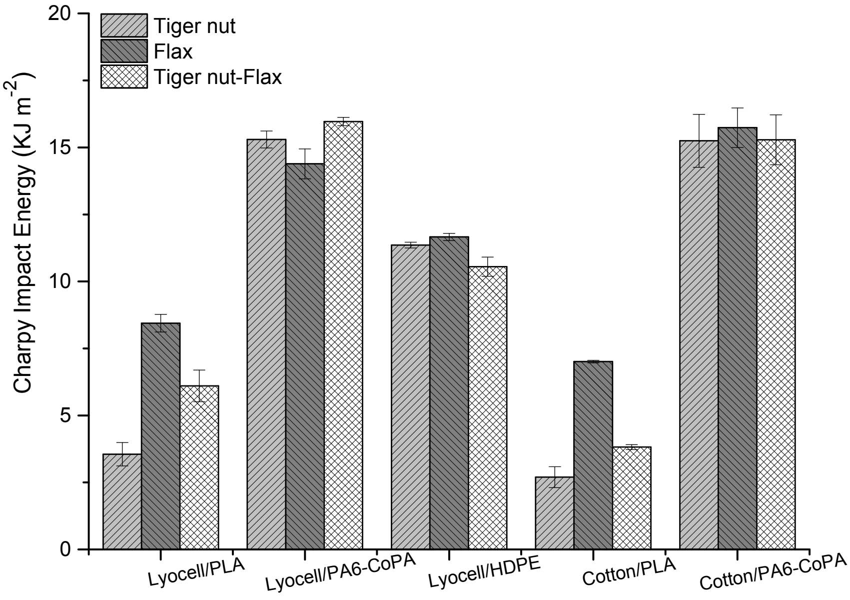
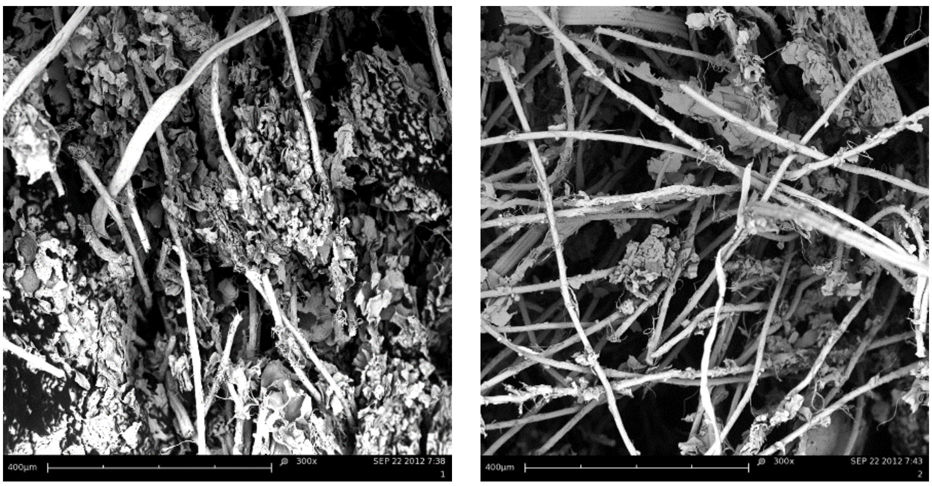
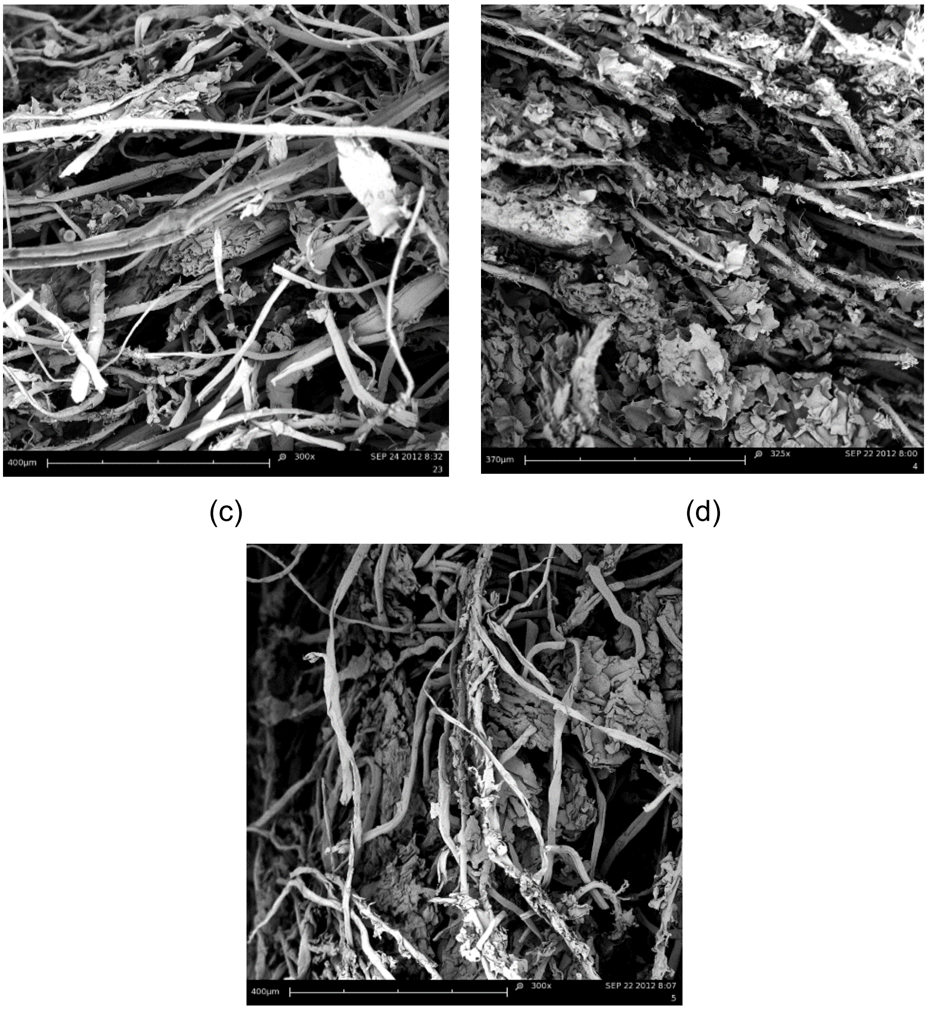


Figure 8. Variation of the energy absorbed in the impact test of the biocomposites with high natural fiber content (80% wt), based on different binders and thermo-boundings.

The morphology study of the fracture surfaces obtained after the impact test (Figure 9) show a lack of continuity in the internal structure of the biocomposites studied.



(a) (b)



(c) (d)

(e)

Figure 9. SEM micrographs of the impact fracture surface of biocomposites with 80% wt waste tiger nut. (a) 80%wt tiger nut + 10%wt lyocell+10%wt PLA, 300x. (b) 80%wt tiger nut + 10%wt lyocell+10%wt PA6/CoPA, 300x. (c) 80%wt tiger nut + 10%wt lyocell +10%wt HDPE, 300x. (d) 80%wt tiger nut + 10%wt Cotton +10%wt PLA, 300X. (e) 80%wt tiger nut + 10%wt Cotton+10%wt PA6/CoPA, 300x.

The morphologies of impact fracture surfaces f biocomposites rich in tiger nut residue, are characterized by a very irregular and heterogeneous appearance. The particles of tiger nut are embedded in the fibers used as a binder and thermo-bonding. The fibers do not have a preferred orientation, are disposed in all directions. Due to the length of fibers and its ripple, tiger nut particles remain "trapped" by the effect of entanglement of the fibers. It is not observed a uniform structure by the absence of matrix phase. This discontinuity causes the existence of many voids in the internal structure of biocomposite. Such structures are responsible for the low impact strength of the analyzed composites. This morphology is similar in all samples although the used fibers as binders and/or thermo-bonding are different. It should be keep in mind that the better impact resistance of the PA6/CoPA composites may be due to the low melting temperature of the bicomponent, 135ºC for the sheath and 220ºC for the core. The outside part of the PA/CoPA termobinder melts during the forming process of the composite, which facilitates the bond points between fibers: tigernut / binder / PA6CoPA. The inside part of the bicomponent fiber does not melt and it facilitates the bonding between fibers by entangling effect between them.

4. Conclusions

A nonwoven material has been obtained applying wet-laid technology with high content of tiger nut waste. This technique of obtaining nonwoven in a wet manner presents the advantage that it is possible to use waste in the form of short fibers or particles and only needs water in order to process the material, so it is an eco-efficient technology. The use of such high contents of tiger nut, 80% wt, permits the revaluation of a major industrial waste.

Obtaining wet laid sheets requires the addition of binder fibers, lyocell and cotton, and thermo-bonding, PLA; HDPE; PA6-CoPA.

The subsequent forming of several layers of nonwoven by termoconpresión lets to obtain biocomposites. Such composites are characterized by not filing a continuous matrix, which has negative effects on the mechanical response of the material. In the response to the bending stresses in the biocomposites studied, the most influential component is the thermo-bonding used. The use of PLA fibers provides maximum performance against bending. Tiger nut/lyocell/PLA exhibit the maximum flexural modulus, 861.78 MPa, and 28% higher than that obtained with flax/lyocell/PLA. In the case of using cotton as a binder, this parameter reaches a value of 872.04 MPa, 18% higher than flax/cotton/PLA.

The results obtained in this study determine that the biocomposites with high contents of tiger nut residue, are suitable materials for many technical applications that not require high strengths. Its manufacturing is optimal using the wet-laid technique and compression molding. This material comes from an agri-food waste and it is very interesting from an environmental and sustainability point of view. It would be suitable for applications such as soundproofing panels or thermal insulation for interior cladding in construction, automotive industry, furniture, etc.

REFERENCES

[1] Akil HM, Omar MF, Mazuki AAM, Safiee S, Ishak ZAM, Abu Bakar A. Kenaf fiber reinforced composites: A review. Materials & Design. 2011;32:4107-21.

[2] Shanmugam D, Thiruchitrambalam M. Static and dynamic mechanical properties of alkali treated unidirectional continuous Palmyra Palm Leaf Stalk Fiber/jute fiber reinforced hybrid polyester composites. Materials & Design. 2013;50:533-42.

[3] Singha AS, Thakur VK. Synthesis and Characterization of Pine Needles Reinforced RF Matrix Based Biocomposites. E-Journal of Chemistry. 2008;5:1055-62.

[4] Thakur VK, Thakur MK. Processing and characterization of natural cellulose fibers/thermoset polymer composites. Carbohydr Polym. 2014;109:102-17.

[5] Thakur VK, Thakur MK, Gupta RK. Review: Raw Natural Fiber-Based Polymer Composites. Int J Polym Anal Charact. 2014;19:256-71.

[6] Sanchez-Zapata E, Fernandez-Lopez J, Angel Perez-Alvarez J. Tiger Nut (Cyperus esculentus) Commercialization: Health Aspects, Composition, Properties, and Food Applications. Comprehensive Reviews in Food Science and Food Safety. 2012;11:366-77.

[7] Sanchez-Zapata E, Fuentes-Zaragoza E, Viuda-Martos M, Fernandez-Lopez J, Sendra E, Sayas E, et al. Reclaim of the By-Products from "Horchata" Elaboration Process. Food and Bioprocess Technology. 2012;5:954-63.

[8] Shklavtsova ES, Ushakova SA, Shikhov VN, Anishchenko OV. Tolerance of chufa (Cyperus esculentus L.) plants, representing the higher plant compartment in bioregenerative life support systems, to super-optimal air temperatures. Advances in Space Research. 2013;51:124-32.

[9] Doh SJ, Lee JY, Lim DY, Im JN. Manufacturing and analyses of wet-laid nonwoven consisting of carboxymethyl cellulose fibers. Fibers and Polymers. 2013;14:2176-84.

[10] Du Y, Wu T, Yan N, Kortschot MT, Farnood R. Fabrication and characterization of fully biodegradable natural fiber-reinforced poly(lactic acid) composites. Composites Part B-Engineering. 2014;56:717-23.

[11] Fages E, Cano MA, Girones S, Boronat T, Fenollar O, Balart R. The use of wet-laid techniques to obtain flax nonwovens with different thermoplastic binding fibers for technical insulation applications. Textile Research Journal. 2013;83:426-37.

[12] Fages E, Girones S, Sanchez-Nacher L, Garcia-Sanoguera D, Balart R. Use of wet-laid techniques to form flax-polypropylene nonwovens as base substrates for eco-friendly composites by using hot-press molding. Polymer Composites. 2012;33:253-61.

[13] Yoon MJ, Doh SJ, Im JN. Preparation and Characterization of Carboxymethyl Cellulose Nonwovens by a Wet-laid Process. Fibers and Polymers. 2011;12:247-51.

[14] Yoon YN, Im JN, Doh SJ. Study on the Effects of Reaction Conditions on Carboxymethyl Cellulose Nonwoven Manufactured by Wet-laid Process. Fibers and Polymers. 2013;14:1012-8.

[15] Cheng Q, Wang S, Rials TG, Kit KM, Hansen M. Fabrication optimization of polypropylene composites reinforced with steam-exploded wood flour by wet process. European Journal of Wood and Wood Products. 2009;67:449-55.

[16] Kim K-Y, Doh SJ, Im JN, Jeong WY, An HJ, Lim DY. Effects of Binder Fibers and Bonding Processes on PET Hollow Fiber Nonwovens for Automotive Cushion Materials. Fibers and Polymers. 2013;14:639-46.

[17] Misnon MI, Islam MM, Epaarachchi JA, Lau K-t. Potentiality of utilising natural textile materials for engineering composites applications. Materials & Design. 2014;59:359-68.

[18] Wang H, Jin X, Mao N, Russell SJ. Differences in the Tensile Properties and Failure Mechanism of PP/PE Core/Sheath Bicomponent and PP Spunbond Fabrics in Uniaxial Conditions. Textile Research Journal. 2010;80:1759-67.