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## Manufacturing and testing composites based on natural materials

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### Abstract

This work focused on the development of composite structures fully based on natural materials. The developed sandwich structures are based on natural fibers and cork acting as a core. The natural fibers used resulted from compounding flax fibers with bio-resin. The core material is agglomerated cork. The aim of this study is to compare these natural structures against similar synthetic sandwiches based on fiberglass and epoxy resin. Several laminates were produced and then subjected to static and dynamic mechanical testing, carried out via bending and impacts tests respectively. After carefully analyzing the results, it was possible to conclude that the proposed materials show compatible mechanical properties and can even compete against synthetic materials. On top of that, natural materials keep the obvious advantage of eco-friendliness.

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### 1. Introduction

Currently, society is aware of environmental problems. Sustainability and recyclability are commonly discussed themes in academia and industry. The feeling of self-responsibility and awareness about the environmental problems led society to change habits, reusing resources and using renewable products. There is a clear trend of substituting synthetic and non-renewable materials by natural and renewable materials with similar or even better properties.

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In this work, sandwich structures made from natural materials are developed. The core consists on agglomerated cork and the skins on flax fibers and bio-resin. This new compound was developed by keeping a balance between sustainability and mechanical properties, proposing a green structure without compromising the mechanical performance. Standard sandwich structures are mainly used due to their strength and weight, being constituted by two outer thin layers responsible for the structure strength and by a softer core responsible for absorbing most of the energy and also for its low weight.

The skins are usually made from fibers such as carbon, glass or Kevlar® or even aluminum sheets. On the other hand, the core is usually made of low density materials such as polymeric or metal foams. These structures can be employed in very demanding applications, as in aerospace, civil construction, automotive, sports, etc [1]. Basically, the surface where the force is applied is under compression and the outer one under tension. On the other hand, the sandwich core supports the shear loads.

Metals have good advantages such as high rigidity, good impact resistance and low cost. However, these are relatively denser, relatively harder and can oxidize. Composite materials are usually made of a polymeric matrix and fibers of same nature. The matrix is responsible for the material adhesion, being responsible for a correct loading distribution. It also allows obtaining complex geometries. Currently, the more common are polyester resins, vinyl and epoxy. The fibers orientation is very important for the overall structure strength.

The core material of a sandwich structure is also very important, since it greatly influences the structure performance. Thus, the final application deeply influences the choice of the core material. The core structure can be a solid block or even a honeycomb. Nevertheless, researchers are looking for new natural and recyclable materials, which can be a better option if these structures can have a similar mechanical behavior to those typically used.

In the literature, there are recent works where cork was used as an energy absorbing material. Alcântara et al. [2] compressed agglomerated cork and concluded that it has a great capacity for energy absorption. Castro et al. [3] concluded the same by testing cork as a core material in lightweight sandwich structures with carbon fiber face sheets. According to Castro et al. [3], in the three point bending tests, parameters such as cork granules size, density and the bonding process greatly influenced the sandwich performance. Nevertheless, in the core, shear stresses were not affected by cork granules size. In addition, cork–epoxy agglomerates presented a significantly better core shear stress limit, which reduces the crack propagation region, placing cork–epoxy agglomerates in the leading edge of currently available materials used within sandwich structures [3]. In the same work, during the impact tests, all cork-based sandwich presented considerably higher load values than those obtained for other type of high performance core synthetic materials. In these impacts was also verified an intrinsic characteristic of cork, the excellent recovery capacity. This characteristic makes it an optimal material to use in multi-impact applications, continuously and effectively absorbing energy after impact, as concluded by Fernandes et al. [4,5], Jardin et al. [6], Ptak et al. [7] and Tchepel et al. [8]

Hachemane et al. [9] performed static and dynamic tests on cork core sandwiches with jute/epoxy face sheets. These structures could be considered natural if not made with epoxy resin. Nevertheless, the initial force, the maximum force and the extent of damage are influenced by cork's density and by the impact energy. In other study, Sousa-Martins et al. [10] used agglomerated cork as core material in sandwich structures with aluminum face sheets. These structures were subjected to shock waves from explosions with success from a protective perspective.

Recently, there is a growing interest by researchers in natural fibers, studying their properties and comparing their performance against synthetic or non-natural fibers. Zhu et al. [11] investigated the effect of fiber configuration on the mechanical properties flax/tannin composites and Petrucci et al. [12] performed three point bending tests on hemp and flax fiber laminates. Fernandes et al. [13] tested high density polyethylene filled with cork powder and coconut short fibers, concluding that the addition of coconut fibers increased the elastic modulus by 27% and the tensile strength by 47% when compared to the samples without coconut fibers.

Kabir et al. [14] used hemp fabrics as reinforcements with polyester resin to form composite skins and carried out alkalization, silane and acetylation treatments on the fibers surface. Kabir et al. [14] suggested that a suitable chemical treatment of the fiber surface can increase the mechanical strength by 30%.

Duigou et al. [15] tested bio-sandwiches and concluded these are very attractive in terms of environmental impact. However, Duigou et al. [15] suggested that further improvements in bio-sandwich mechanical strength are necessary in order to be a valid option for demanding applications, substituting the currently used and non-environmental friendly sandwiches.

In this work, it is proposed a bio-sandwich, made from natural materials. The core is made from agglomerated cork and the composite face sheets are made from a compound of flax fibers and bio-resin. In order to evaluate the mechanical strength of these structures, three point bending tests and Charpy impact tests were performed. The bio solution was compared against typically used compounds made from fiber glass and a non-bio-resin, maintaining the same core material. Thus, it is possible to establish a feasible comparison between the bio-compound and non-bio-compound.

## 2. Materials and methods

In this work, natural fibers are used in order to create an environmental friendly solution. Natural fibers may have different origins: mineral, animal or even vegetal. Fig. 1 illustrates some examples of natural fibers. In this study, flax fibers (Easy composites, UK) were used, mainly due to their good cost/properties ratio. This type of fiber is heavier than fiber glass but is also tougher. Fig. 1 shows the configuration used for flax fibers. Flax fibers with four different surface densities were used in this work: 500 g/m<sup>2</sup>, 400 g/m<sup>2</sup>, 200 g/m<sup>2</sup> and 100 g/m<sup>2</sup>. Regarding fiber glass, two densities were tested: 500 g/m<sup>2</sup> and 200 g/m<sup>2</sup>.

Since one of the objectives of this work is testing a green sandwich, bio-resins were also used. The resins used were the epoxy Sikadur 330 resin and the Super Sap CLR bio-resin (Entropy Resins). The last has a biological origin of 25%.

Regarding the core material, the CoreCork NL 10 (Amorim, Portugal) was chosen. It is a cork granulate specifically designed to be used as a core material in sandwich structures. It is a flexible material, capable of absorbing considerable amounts of energy and can be used to manufacture products with complex shapes. Another advantage of this material is its low capacity to absorb fluids, which greatly reduces the amount of resin used in its manufacturing process. Some of the characteristics of CoreCork NL10 samples are presented in Table 1.

Table 1. CoreCork NL10 characteristics.

Density [kg/m <sup>3</sup> ]	Thickness [mm]	Absorption [%]
120	3	< 4



Fig. 1. Example of flax fiber configuration and cork agglomerate used.

Sandwich samples were manufactured by manual molding and vacuum pressing (Fig. 2). Table 2 shows all the sandwich samples analyzed in this work. The samples were cut using water jet to avoid damaging the samples.

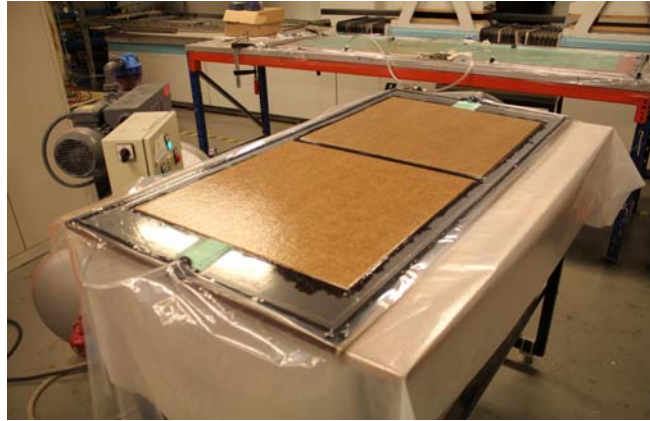


Fig. 2. Sandwich samples manufacturing: manual molding and vacuum pressing.

Table 2. Sandwich samples.

Sample	Top face	Core	Bottom face	Thickness [mm]	Resin
1	Fiber glass 500 g/m <sup>3</sup>	CoreCork NL10	Fiber glass 200 g/m <sup>3</sup> (double layer)	4	Epoxy
2	Flax fiber 500 g/m <sup>3</sup>		Flax fiber 400 g/m <sup>3</sup>	5	Bio
3			Flax fiber 200 g/m <sup>3</sup>	4	Epoxy
4	Flax fiber 100 g/m <sup>3</sup>		Flax fiber 200 g/m <sup>3</sup>	4	Bio
5	Flax fiber 200 g/m <sup>3</sup>		Flax fiber 100 g/m <sup>3</sup>		
6	Flax fiber 100 g/m <sup>3</sup>				

### 3. Experimental tests

Bending tests were carried out according to ASTM C 393 standard [16]. Fig. 3 shows sample 1 (top image) and sample 2 (bottom image) being tested at the moment of maximum load.

A Shimadzu universal testing machine, equipped with a 10 kN load cell was used. The tests were carried out with a velocity of 6 mm/min. The supports and the loading tool have a diameter of 8 mm and 10 mm respectively. For each type of sample, 5 tests were performed (5 samples). In addition, due to the difference between the top and bottom faces of samples 1 to 4, these were tested on both faces, which means that for each type (between 1 and 4 inclusive) 10 tests (10 samples) were performed. In total, 50 samples were tested.



Fig. 3. Three point bending test.

All the samples were visually analyzed, checking the types and regions of failure. Although the ASTM C 393 standard determines the mechanical properties such as shear stress in the core and the stress at the compression faces, in this work the objective is just to study the behavior of the samples regarding bending strength. Thus, it was analyzed the relation between load and displacement during the tests. This allows the calculation of the maximum deflection without failure and the load necessary to create failure at the faces. Observing the samples, two types of failure modes were observed:

- Indentation (crushing) on the loading surface, probably due to the penetration of the loading tool on the samples' surface, (Fig. 4). This behavior was only observed on fiberglass samples;
- Rupture by tensile stress on the opposite face where the load was applied (Fig. 5). This kind of more expectable failure was observed on the majority of flax-fiber composites.

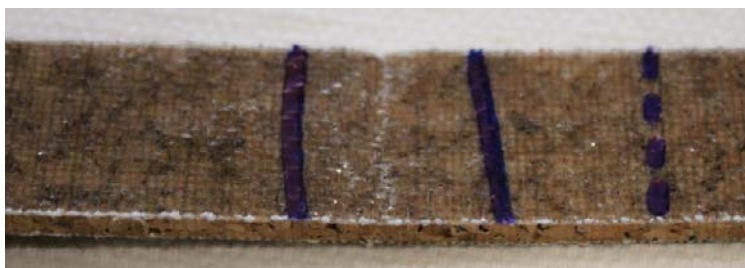


Fig. 4. Indentation on the loading surface.



Fig. 5. Rupture on the opposite face by tensile loads.

Fig. 6 shows the force-displacement curves measured during the three-point bending tests while Figs. 7 and 8 the maximum value for force and displacement on each case study. Analyzing these results, it is possible to conclude that there is a relationship between the load and the fibers surface density. The greater the density, the greater the load needs to be to cause failure.

Regarding fibers' performance, sample 1 (fiberglass+epoxy) was more resistant when loaded at the bottom surface (the less dense one). Nevertheless, the results are very similar not only during the loading but also at failure. The failure between the two types of samples happened for a force value difference of 5 N and 1 mm regarding the displacement.

On the other hand, there is a great difference on the behavior of sample 2 (flaxfiber+bioresin) if loaded on different surfaces. Higher forces are needed when the less dense surface (bottom) is loaded and when failure does not happen immediately, contrarily to the top, denser surface. The failure values are separated by 10 N of force and 10 mm of displacement.

In the case of sample 3 (flaxfiber+epoxy), a similar behavior, compared to samples 1 and 2, is observed regarding the densities of the fibers on each surface. Nevertheless, the failure is more immediate as in the case of sample 1. Sample 4 presents a higher strength when loaded on the top face. Depending on the loading face, there is a difference of 6 N and 10 mm of the failure values.

Regarding the samples 5 and 6, these have the same type of material with same thickness on both surfaces, thus only one side was tested. As expected, due to the samples density difference, sample 5 showed a higher strength than samples 4 and 6. The sample 6, being the sample with less dense fibers, was the one that showed lower strength in the three point bending test.

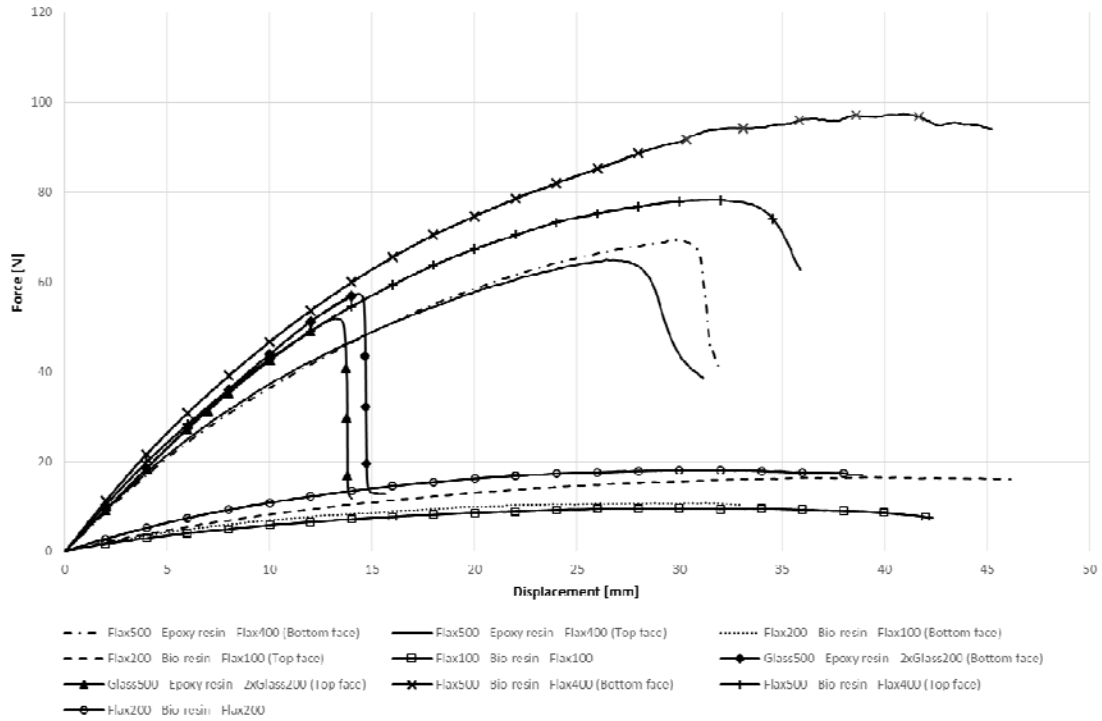


Fig. 6. Force-displacement curves.

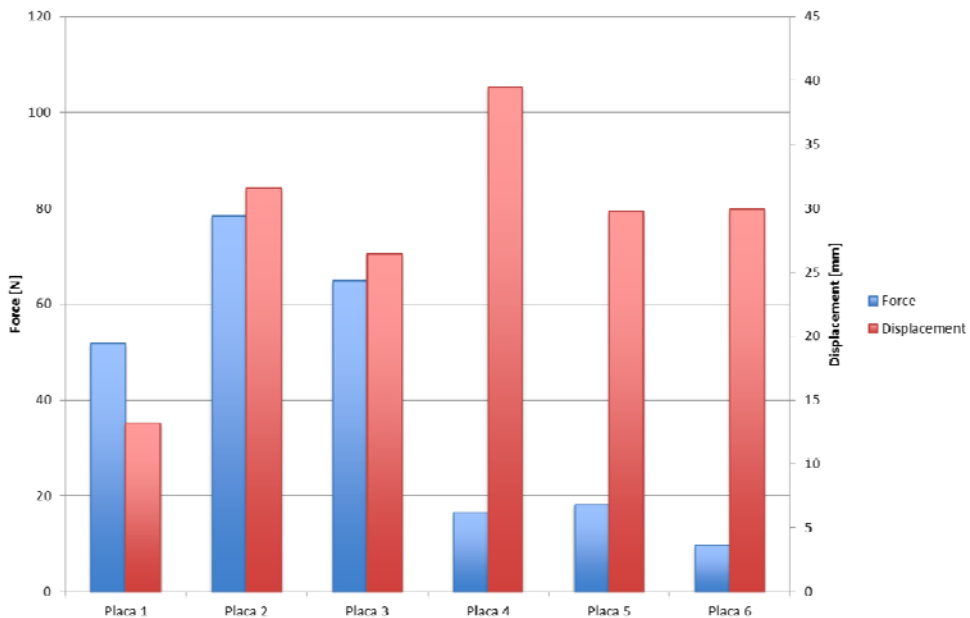


Fig. 7. Maximum load and displacement of the samples loaded on the top surface.

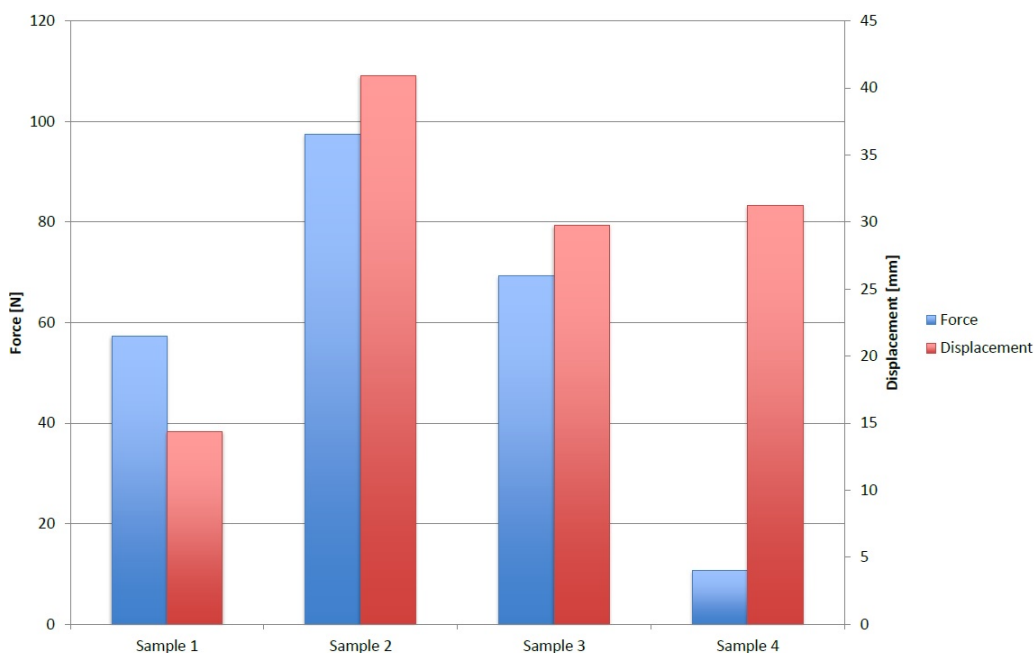


Fig. 8. Maximum load and displacement of the samples loaded on the bottom surface.

Sandwiches 2 and 3 only differ on the type of resin used in their construction. Thus, by comparing the performance of these two, it is possible to evaluate the influence of the resin in the structure.

Comparing the green sandwiches (samples 2, 4, 5 and 6), sample 2 performed better in the three point bending test (Fig. 9). This can be justified by the denser fibers present in sample 2, the sandwiches with denser fibers absorbed more energy than the remaining natural ones. Thus, sample 2 performed better.

In this work, it was also performed a cost analysis. The values presented in Table 3 are the average weight of each type of sample per square meter. Since the objective is to compare the sandwiches and all have the same core material in the same amount, the core material was not considered in this analysis. Additionally, Table 3 shows the average cost of each type of sandwich for a sample with a top/bottom surface area of 0.5 m<sup>2</sup>. In this prevision, 1/3 liters of resin and two fiber mats of 0.5 m<sup>2</sup> were considered.

Table 3. Average cost of each sample per 0.5 m<sup>2</sup>.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Average weight [kg/m <sup>2</sup> ]	2.1	3.3	3	1.8	2	1.6
Top surface	12.86	6.81	6.81	3.19	4.37	3.19
Bottom surface	1.96	8.57	8.57	4.37	4.37	3.19
Resin	1.88	8	1.88	8	8	8
Total [€]	16.7	23.38	17.26	15.56	16.74	14.38

#### 4. Conclusions

After the onset of synthetic sandwich materials over the last decades, environmental concerns are bringing back attention to natural materials, both fibers and core materials. The natural sandwiches herein studied, based on flax fibers and agglomerated cork, were tested in quasi-static and dynamic conditions, showing very promising results in terms of specific strength and toughness.

On the downside, the specific weight and cost are still higher when compared to synthetic rivals. If the first issue can be solved optimizing quantities and thicknesses of the several sandwich components, the second will need a large scale production to deliver cost-reduction. Cork agglomerate, in the end, is produced from the scrap of wine-stoppers production, a low cost material that must be exploited.

Bearing in mind that not only cork structural properties can be very interesting, future research lines will certainly try to ally its well-known acoustic, thermal and vibration properties.

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