Natural Cork Agglomerate as an Ecological Alternative in Constructional Sandwich Composites

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The investigations presented in this article include a comparative study of static and fatigue four-point flexural tests performed for sandwich composites. The investigated composites consisted of a glass-epoxy laminate as a cladding material and core materials, such as synthetic foams and natural cork agglomerates, in different densities. The sandwich composites were prepared with the vacuum bagging method using the same resin, reinforcement, and additives. Although using cork agglomerate in sandwich composites instead of synthetic foam resulted in a decrease of the static flexural strength in such composites, it increased their resistance to fatigue cycles considerably and benefitted their eco-friendly image. However, only the reproducibility of all the factors in the production process and testing of composites allows a direct comparison of their test results to be made.

Keywords: Natural cork; Synthetic foam; Sandwich composites; Static four-point flexural test; Fatigue four-point flexural test

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INTRODUCTION

Natural cork is a renewable raw material obtained from cork oak (*Quercus suber*) of which the outer bark can be stripped every 9 to 14 years for more than 2 to 3 centuries without endangering the tree itself. Nearly 2.3 million ha of cork oak forests in the world are valuable ecosystems both ecologically and socio-economically. They are naturally distributed in Portugal and around western Mediterranean basin, especially in Spain as well as in China, Japan, and Korea. Portugal has the largest cork oak forests followed by Spain, accounting for about 34 and 27% of the global cork oak area, respectively (APCOR 2013). Portugal and Spain are also leading producers of raw cork, yielding about 200 and 150 kg ha^{-1} $year^{-1}$ and with shares of 50 and 31% of the worldwide production, respectively (APCOR 2013; Dias *et al.* 2014). Nowadays, cork is the key element in the preservation of suchlike agro-forestry systems because it represents the highest economic incomes and environmental advantages (Rives *et al.* 2013).

Currently, bio-based materials receive widespread interest due to their positive influence on the environment. This interest results from the increasingly commonplace presence of ecology in the transport industry. Accordingly, materials that enable highly efficient production while reducing the risk of disturbing the balance of the environment are desired. Many known, already bio-based materials could successfully replace the existing solid components that are based on synthetic materials. This would lead to a substantial reduction of CO_2 emissions and decrease the level of synthetic waste. For this reason, it is very important to conduct research aimed at proving that bio-based materials are the ecological and economical alternative for synthetic solutions.

One of these materials is cork agglomerate, which consists of cork granules with a precisely specified diameter and organic binder. Due to an advanced, reproducible processing technology, cork agglomerate panels with a specific density, performance, and technological properties can be created (Fernandes *et al.* 2014). A wide range of compositions of this material is available on the market in a variety of forms in terms of the grain size, type, and density of the bonding material (Moreira *et al.* 2010). Moreover, cork agglomerate is biodegradable whereas core made of synthetic foam are non-recyclable and non-biodegradable (Sargianis *et al.* 2012).

The main environmental advantage of the use of forest materials such as wood or cork is storing a large amount of carbon dioxide (CO_2) until the end-of-life of the products (Gill 2011; Rivers *et al.* 2013; Demertzi *et al.* 2017). Being beneficial for the environment and to the fight against global warming, the cork oak increases its ability to absorb the carbon dioxide during the natural regeneration process following barking (Gill 2011; Amorim 2012). Apart from carbon storage the cork oaks show a significant impact in biological processes such as water retention and soil conservation (Rivers *et al.* 2013; Sierra-Pérez *et al.* 2016a).

Cork oak forests make a sequestration of about 5.7 tonnes CO_2/ha/year. The 2.3 million ha of cork oak forests worldwide are seen as promoting the retention of about 14.4 million tonnes CO_2/year. It should be noted that during the production of 1000 cork stoppers 1.5 kg of CO_2 were emitted, although 14 kg, and 37 kg of CO_2 were emitted for the same amount of plastic stoppers and Al-screw-caps, respectively. Whereas the annual worldwide consumption extends to more than 15 milliard stoppers (Gil 1998 and 2011; Pereira 2007; Amorim 2012). How the cork sector could help to mitigate climate change is possible for estimation by statement of a fact that a tonne of raw cork in forest converted into products generates emissions of about 3.4 tonnes of CO_2 eq., while the fixation is about 18 tonnes of CO_2 (Rives *et al.* 2013).

When assessing the biogenic CO_2 balance (CO_2 emissions and removals resulting from biogenic sources) in life cycle assessment (LCA) studies of materials, the forest-based products are mainly treated as potentially carbon-neutral materials since it is considered that the amount of CO_2 sequestrated by the forest is then emitted into the atmosphere at the end-of-life stage of the product (Guo 2012; Demertzi *et al.* 2017). Therefore, biogenic CO_2 sequestration and emissions are usually excluded from the life cycle assessment (LCA) studies for the production of cork (González-García *et al.* 2013; Dias *et al.* 2014; Demertzi *et al.* 2017). However, recent studies suggest that biogenic CO_2 should be taken into account in order to have a more complete view of the system under study (Müller-Wenk and Brandão 2010; Levasseur *et al.* 2013; Demertzi *et al.* 2015 and 2017). Nowadays LCA methodology (ISO 14040, 2006) has gained increased international acceptance. Within the environmental field, there has recently been increased interest in the use of LCA to evaluate natural cork as insulation material in different construction situations in building sector especially (Pargana *et al.* 2014; Sierra-Pérez *et al.* 2016a).

Cork is a natural material that consists of closed microcells filled with a gas mixture similar to atmospheric air. In one cubic centimeter there are approximately 40 million cells (Gil and Moiteiro 2003). The cellular structure of cork gives its low density, which is why the application of natural cork agglomerate as the core material in a sandwich composite significantly reduces the construction weight (Belgacem and Gandini 2008). A reduction of the transportation costs is an additional benefit with a lower weight. Materials with a cellular structure, like cork or synthetic foams, have been employed as medium to absorb and dissipate energy for a long time. The ability of these materials to absorb energy can be attributed to their ability to absorb energy. Cellular structure becomes increasingly compact if energy absorption is to be intensified (Pereira 2007; Pires *et al.* 2007).

Light sandwich composites with cork agglomerate are used widely in the aircraft industry, where the strength properties of cork agglomerate panels were investigated in static bending and dynamic compression tests. The results were found to be dependent on grain size, density, and bonding technology of the agglomerate. Using cork agglomerates as the core material can impart many interesting features into the final sandwich composite, such as low density, while keeping other specific properties at high levels, including the modulus of elasticity, shear resistance, and stiffness (Zenkert 1997). Excellent acoustic and thermal insulation properties, high resistance to impact damage, and good vibration damping properties are also very important features of these materials (Zenkert 1997; Pereira 2007).

All constructions based on sandwich composites with a cork core have considerably higher load values than other traditional materials. In addition, and of more importance, the presence of cork in the composites decreases the possibility of crack propagation. Composites based on cork agglomerates are easy to obtain and recycle because of the origin of the material. Moreover, in comparison to highly stiff foams, these materials are capable of absorbing a higher amount of energy, and display a better resistance to impact (Alcantara *et al.* 2013).

The fundamental differences between the behavior of cork when subjected to loading and that of synthetic foams are the elasticity and the possibility of increased the thickness. A cork sample that has been compressed and deformed up to a strain of 90% at first, after removing the load recovers up to approximately 75% nominal height, and more than 90% of the height 12 h later. In contrast, synthetic foam permanently deforms (Alcantara *et al.* 2013).

Additionally, natural cork agglomerate has excellent thermal and acoustic insulation properties, and anti-vibration properties. Furthermore, thanks to the high content of suberin, cork is chemically neutral. Cork also constitutes a barrier for microorganisms and fungi, it is impermeable to liquid and gas, and it is a hydrophobic material (Anjos *et al.* 2008; Sargianis *et al.* 2012).

Currently, the mechanical properties of cork are largely unknown, making widespread application of this material difficult. Sandwich composites are usually tested in three-point bending mode only (Karahan *et al.* 2013). In this article, the comparative research of static four-point flexural tests and fatigue four-point flexural tests performed on sandwich composites with epoxide-glass laminate are investigated as the surface material, and polyurethane foam and cork agglomerate (with differing densities), as the core materials. The composites were manufactured using the vacuum bagging method in the same conditions of manufacturing and research.

In this study, selected light sandwich composites, consisting of natural and synthetic core materials (natural cork and synthetic foam with different densities), were investigated by means of static and fatigue four-point flexural tests. The obtained results were compared and analyzed from the point of view of the transport industry, where possibly economic and sturdy light materials are required.

EXPERIMENTAL

Materials

Composite manufacturing process

The sandwich composites studied had epoxide-glass laminate as their surface layer, which consisted of four layers of Aeroglass 280 g/m^2 fabric (Havel Composites CZ, Přáslavice, Czech Republic) with a twill weave from glass silk, and was impregnated using an epoxide system: resin CR132 and hardener CH132-5 (Sika Deutschland GmbH, Stuttgart, Germany). This epoxide system was characterized by good wettability of the reinforcing fibers, non-toxicity, and high strength properties, even at room temperature. This system was applied in aviation, wind energy, automotive, and mold production (Sika 2016).

Divinycell H60 and Divinycell H130 (Diab Inc., Laholm, Sweden) are foams that are cross-linked by isocyanates and were obtained from the combination of polyvinyl chloride, aromatic polyuria, and polyamide (Diab 2009). The number in the second part of the foam names stands for its density. For example, Divinycell H60 has a density level of 60 kg/m^3, and Divinycell H130 has a density level of 130 kg/m^3 (Diab 2016). The market price of Divinycell H130 foam was about 67 €/sqm while the price of Divinycell H60 was about 30 €/sqm. The properties of the Divinycell foams are presented in Table 1.

Table 1. Properties of Divinycell H60 and Divinycell H130

Property	Divinycell H130	Divinycell H60	Test Proced.
Density (kg/m^3)	130	60	ISO 845
Thermal Conductivity (W/mK)	0.036	0.029	EN 12667
Coefficient of Linear Heat Expansion (1/°C)	40×10^{-6}	40×10^{-6}	ISO 4897
Heat Distortion Temperature (°C)	125	125	DIN 53424
Operational Temperature Range (°C)	-200 ÷ 70	-200 ÷ 70	-
Max Process Temperature (°C)	+110	+90	-
Dielectric Constant (-)	1.15	1.06	ASTM D2520
Poissons Ratio (-)	0.4	0.4	ASTM D638
Compressive Strength (MPa)	3.0 (2.4)	0.9 (0.7)	ASTM D1621
Compression Modulus (MPa)	170 (145)	70 (60)	ASTM D1621
Tensile Strength (MPa)	4.8 (3.5)	1.8 (1.5)	ASTM D1623
Tensile Modulus (MPa)	175 (135)	75 (57)	ASTM D1623
Shear Strength (MPa)	2.2 (1.9)	0.76 (0.63)	ASTM C273
Shear Modulus (MPa)	50 (40)	20 (16)	ASTM C273
Shear Strain (%)	40 (30)	20 (10)	ASTM C273

Table 1 shows the average and minimum values of each of the properties (minimum values are in brackets). In comparison to cork core materials, Divinycell foam was characterized by high strengths – both compressive and shear strengths – even when exposed to increased temperatures. In addition, its spheroidal structure makes this material highly resistant to fatigue cycles and impact loads. Additional and exceptional qualities of this type of foam include: good adhesion, energy absorption during impact without sustaining damage, a high chemical resistance, low water absorption, and good thermal and acoustic insulation. Its maximum processing temperature was 90 °C (Diab 2016).

Corecork NL10 and Corecork NL20 (Amorim Cork Composites, Porto, Portugal) are natural cork agglomerates. As in the case of the foams, the second part of their name refers to their density. However, Corecork NL10 has a density level of 120 kg/m^3, and Corecork NL20 a density level of 200 kg/m^3 (Amorim 2009). The properties of these core materials are presented in Table 2. The materials were characterized by high insulation properties and good vibration damping. All of these effects were caused by the cork's structure, which has the ability to disperse stresses in its microstructure. Furthermore, important properties of cork are: chemical and biological neutrality, fire resistance, and the ability to withstand many fatigue cycles (Marszalkiewicz 2012; Sousa-Martins 2013). The market price of Corecork NL20 was about 50 €/sqm while the price of Corecork NL10 was about 40 €/sqm.

Table 2. Properties of Corecork NL 10 and Corecork NL 20

Property	NL 10	NL 20	Test Proced.
Density (kg/m^3)	120	200	ASTM C271
Compressive Strength (MPa)	0.3	0.5	ASTM C365
Compression Modulus (MPa)	5.1	6.0	ASTM C365
Tensile Strength (MPa)	0.6	0.7	ASTM C297
Shear Strength (MPa)	0.9	0.9	ASTM C273
Shear Modulus (MPa)	5.9	5.9	ASTM C273
Thermal Conductivity (W/mK)	0.042	0.044	ASTM E1530
Loss Factor (at 1kHz) (-)	0.0022	0.043	ASTM E756

To manufacture composite sandwich panels, light cores with a nominal thickness of 30-mm were selected and eight panels were manufactured, each measuring 1000 mm × 500 mm (*L* × *W*) in size. The panels were created using the vacuum bagging method, which is a modified method of hand lay-up lamination (Fig. 1).

Fig. 1. Sandwich composite sheet manufactured using the vacuum bagging method

After curing the composites (24 h), the panels were heated under specific conditions. Heating the epoxy matrix was a key factor for achieving enhanced strength parameters in the composites. It was necessary to underline the importance of the heating rate, the temperature, heating time, and also, the cooling rate, because they are all factors that can contribute to the formation of stress in the cured compound, and can result in a deformation of the laminate. After heating, the sheets of composites were cut into test samples using a plotter WaterJet Streamcut (Kimla, Czestochowa, Poland). Their dimensions adhered to ISO 14125 (1998), which was designated for testing mechanical properties.

Methods

During the investigations of the sandwich composites with a static four-point flexural test, the sample was symmetrically loaded perpendicular to the lamination surface with two forces equal in value. The static four-point flexural test was conducted according to ISO 14125 (1998) on the universal testing machine Instron 8501 Plus (Instron, High Wycombe, Great Britain) using the Series IX Automated Materials Testing System 8.34 and a flexural speed of 5 mm/min. The tests were conducted at 22 °C ± 1 °C and in relative humidity level of 45%. The dimensions of the samples and spacing of the supports were dependent upon the final thickness of the sample. The sample dimensions are shown in Table 3.

Table 3. Dimensions of Tested Composite Samples

Sample Thickness *h* (mm)	Sample Length; *l* = 30 *h* (mm)	Sample Width *b* (mm)
32.9	987	50

Spacing between the lower supports was selected in compliance with the ISO 14125 (1998) standard using the following relation: $L = 22.5\ h$ (mm).

The bending stress was calculated by Eq. 1, according to the following formula (which complies with the ISO 14125 (1998) standard),

$$\sigma\ (N/mm^2) = F \cdot L / b \cdot h^2 \tag{1}$$

where F is the maximum loading (N), L is the spacing between supports (mm), b is the sample width (mm), and h is the sample thickness (mm).

The deformation of the sample was calculated according to Eq. 2, which complies with the ISO 14125 (1998) standard,

$$\varepsilon\ (\%) = \left(4.7 \cdot s \cdot h / L^2\right) \cdot 100 \tag{2}$$

where s is the bend deflection (mm).

The fatigue four-point flexural test was conducted on the universal testing machine Instron 8501 Plus (Instron, High Wycombe, Great Britain) using the Wave Maker Series 9.1 software (Wave Maker Inc., London, UK). The preload amounted to 85% of the maximum bending load of the sample, and the amplitude was 2 mm. The frequency of the sample bending was 2 Hz, and the test was conducted in accordance with a sinusoidal varying cycle. Test parameters were selected with care to enable a comparison of the number of cycles necessary to destroy material structures with cork and foam cores. The dimensions of the samples complied with those listed in Table 3, and the spacing between the supports of the universal testing machine complied with the ISO 14125 (1998) standard. Based on the results, the number of cycles after which the sample was destroyed while subject to loading was specified.

RESULTS AND DISCUSSION

Figure 2 presents the bending stress-strain curves of the composites with the foam core (Divinycell H130 and H160 type) and the composites with cork agglomerate as the core (Corecork NL10 and NL20 type). The area under the stress-strain curves in Fig. 2 presents toughness as the ability of a material to absorb energy. However material resilience makes up only a small portion of the total energy that the material can absorb before failure. Energy absorbed per unit volume without permanent deformation defines modulus of resilience that equals the area the stress-strain curve up to the elastic limit often approximated by material's yield point dependent on modulus of elasticity.

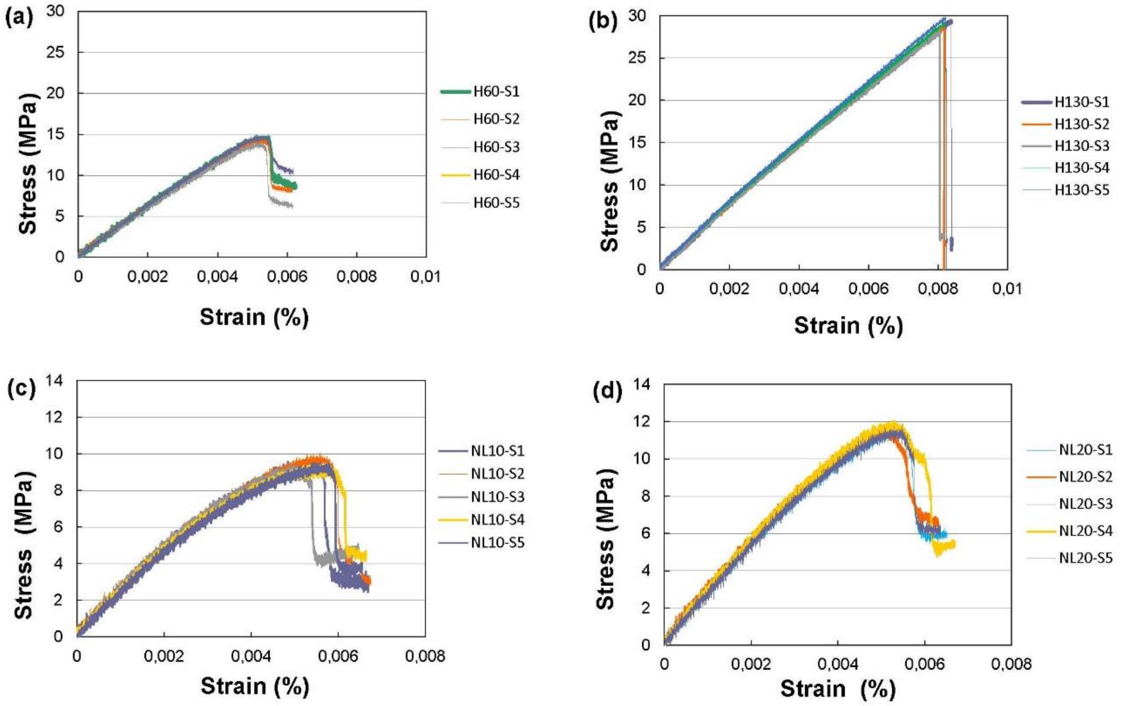

Fig. 2. Bending stress as a function of strain for composites with cores consisting of: (a) Divinycell H60 foam, (b) Divinycell H130 foam, (c) cork agglomerate NL10, and (d) cork agglomerate NL20

The material that scored the highest parameters in terms of its flexural strength in the static four-point flexural test was the composite with the Divinycell H130 core. Its flexural strength equaled approximately 28.9 MPa and its flexural modulus of elasticity was 3505 MPa. In the case of Divinycell H60 foam, these values equaled approximately 14.6 MPa and 2817 MPa. The composite with the Corecork NL20 core displayed average values of 11.9 MPa and 2536 MPa. The lowest values were obtained by the composite with Corecork NL10 which were approximately 9.6 MPa and 2093 MPa (Fig. 3). The composite with the Divinycell H130 core displayed better strength parameters than the composite with the Divinycell H60 core due to its higher material density. Similar dependences were observed between the composite with the Corecork core. However, the synthetic foams displayed a much higher static flexural strength.

Sandwich composites with polymeric foam or with cork owing to their good energy dissipation properties and flexural strength can improve significantly the passive safety of articles manufactured for the building trade and the automotive industry, especially.

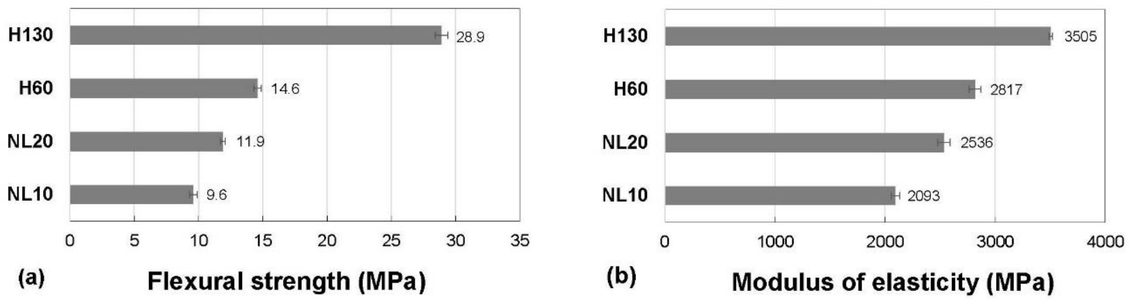

Fig. 3. Results from the static four-point flexural test of sandwich composites: (a) Flexural strength, and (b) Flexural modulus of elasticity

During the tests, it was observed that the sandwich composites with a cork core returned to their original shape after the load was removed, and the structure of the core material was not damaged. When maximum flexural load was exceeded only the cladding layers (glass-epoxy laminate) were subjected to cracking in a gentle way (Fig. 4). An inverse relation was observed in the case of the synthetic foams. After the load was removed, the structure of the core was considerably thicker in the areas of the supports, and destruction happened violently (Fig. 5).

Fig. 4. Sample with Corecork NL20 core after four-point flexural test

Fig. 5. Sample with synthetic Divinycell H60 foam core after four-point flexural test

Figure 6 shows the number of fatigue cycles after which composites were destroyed. The analysis of the results of the fatigue four-point flexural test revealed that the sandwich composite with the Corecork NL20 core was the most resistant to the variable (cyclic) load. This composite was not destroyed after approximately 8000 cycles. Composites with low-density foam (Divinycell H60) were destroyed when exposed to sinusoidal loading, and the lowest number of cycles achieved was approximately 860.

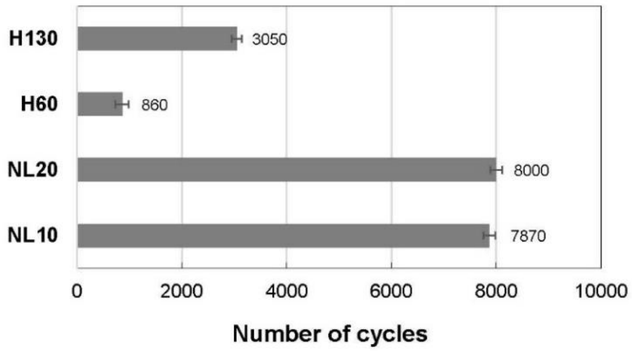

Fig. 6. Number of loading cycles after which composites were destroyed

The destruction mechanisms of particular composite materials when exposed to cyclic loads were completely different. Samples of foam core with a high concentration of Divinycell H130 were destroyed by violent cracking. The composites with Divinycell H60 foam were destroyed by asymmetrical cracking and delamination of the cladding material from the core material as well as gradual cracking of the core (Fig. 7). Composites with cork (Corecork NL10 and Corecork NL20) cores were destroyed by a plastic deformation and cracking of the external glass-epoxy laminate (Fig. 8). In the case of both Corecork NL10 and Corecork NL20, the composites returned to their original shape after the load was removed, and they were free of visible damage in their structures. Only deformation of the cladding material was observed.

Fig. 7. Asymmetrical cracking of the cladding surface (glass-epoxy laminate) in the sample with Divinycell H60 during the fatigue test

Fig. 8. Cracking of the cladding material (glass-epoxy laminate) in the sample with NL20 during the fatigue test

Sandwich structures with synthetic foam core are mainly applied due to their ability to absorbing high energy in a crash. Such sandwich composites are used, for example, in exterior panels for the ADtranz Regio Shuttle trains (Marsh 2002). The composites are also widely used for the sporting goods such as windsurfing and kite boards, skis, *etc*. They are used for the refrigerated transportation containers, pleasure boats and commercial vessels, aircraft, and building panels as well (Karlsson and Åström 1997). Synthetic foam composites are still prevalently used because other available materials with improved properties and high fatigue strength have not become perfected yet.

Nowadays ecological alternatives for constructional sandwich composites are becoming available. Novel sandwich composites with the cork core can improve composite behavior very much in the aspect of energy absorbing and fatigue strength, especially. The sandwich composites with the cork core already find many different applications. They are used for thermal insulation in the mini-bar refrigerator, cooling chambers and rockets, acoustic insulation in submarines, theater's and recording studios, seals and joints in woodwind instruments, combustion engines and concrete constructions, and as energy-absorbing media in floor coverings, shoes and packaging, and as stoppers (Gil 2003; Mestre 2015). There is high commercial interest for the introduction of cork material in the home furniture and decoration sector such as tableware, ceiling and table lamps, hammocks, sofas, sunbasket, *etc*. Regarding the building market, the main applications of cork make insulation boards (Mestre 2015, Sierra-Pérez *et al.* 2016b). Moreover, the cork composites are used also for the flooring system of the tram, train, and metro. A leading example of the uses the cork composites is Siemens Metro Inspiro. The panels inside of the carriage are made of layered composite consisting in GRP system and core cork. The floor systems also are based on cork composites, and such composites consist of core cork and sheets of the aluminum, so-called AluCork (Amorim 2012). The AluCork composites are also used for floor system in the trams produced by polish makers from PESA Bydgoszcz SA.

Sandwich composites with cork are not easy subject to fire because cork is flame-proof and non-toxic in fire. These unique features, besides high static flexural strength and good fatigue resistance as well as insulation properties, make the composites with the cork very interesting eco-material in the building trade and means of transport at first. Recently, due to cork's good physical properties, new ideas generated for both applications and markets are very diverse (Sierra-Pérez *et al.* 2016a). In this way sustainable development and product innovation are initiated with the discovery of a market or technological opportunity, followed by the process of finding new ideas, designing the new product concepts, detailing the product, preparing it for production, and finally launching the new product on the market. Sustainable product innovation is concerned with the creation of new added-value eco-efficiency products and/or services that can be successfully implemented in the market (Mestre 2015).

CONCLUSIONS

Constructional sandwich composites that consisted of natural and synthetic core materials with different densities were investigated by means of static and fatigue four-point flexural tests.

The composites with synthetic foam Divinycell H60 and Divinycell H130 possessed a much higher static flexural strength that was 14.6 MPa and 28.9 MPa, respectively, compared to the composites based on the cork agglomerates NL10 and NL20, which were 9.6 MPa and 11.9 MPa, respectively. On the other hand, the composites based on the cork agglomerates NL10 and NL20 displayed better fatigue resistance, which was 7870 and 8000 number of loading cycles, respectively, compared to the composites with synthetic foam Divinycell H60 and Divinycell H130, which were 860 and 3050 number of loading cycles, respectively.

By comparing the characteristics both of the core materials, it can be seen that the composites with natural cork agglomerate could successfully replace traditional synthetic foam in cases where a high fatigue resistance is required, in particular as floor systems and partition walls in the mobile refrigerated and public transport industry, as well as materials for construction watercrafts such as kayaks, sailboats, and yachts. Innovative solutions for replacing the traditional foam for the cork agglomerate are already patented. One of them is table top of the operating table (No. P.413912) which is 235 cm in length. Despite its being mounted from one side, it can withstand a load of 1000 kg. During the loading the operating table top deforms elastically (12 cm), but after removing the load it returns to its original dimensions. This product with a foam core during such test is completely destroyed. The composites with the synthetic foam, which are characterized by high static bending strength, can be competitive in the building sector (*e.g.* as walls, floors, and roofs) especially.

In regards to the market prices of core materials, the most expensive was Divinycell H130 foam, it was more expensive than Corecork NL20 by about 25%, and Corecork NL10 by about 40%, and Divinycell H60 by about 55%. So, the cheapest but also the weakest in terms of fatigue resistance was the Divinycell H60 foam in comparison to the other core materials.

Possibility of the application on natural cork agglomerate as an ecological alternative to sandwich composite construction instead of synthetic foam was established.

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