A NOVEL RECYCLING ROUTE FOR POLYETHYLENE BOTTLE CAPS AS CIRCULAR HONEYCOMB CORE

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Abstract

The disposal of plastic wastes in the environment has been an urgent issue due to pollution of the soil and the sea. The incorporation of polymeric residues into innovative and sustainable structural composites can be considered a promising recycling route. This work investigates the use of disposed polyethylene (PE) caps from drinking water bottles as a sustainable honeycomb core with circular cell geometry. A full factorial design was conducted to identify the effect of bottle caps orientation (single and alternated directions), polymeric adhesive (epoxy and polymer), and aluminium roughness (smooth and rough surface) on the mechanical properties of sandwich panels. The use of epoxy polymer contributes significantly to enhance panel strength and stiffness. Smooth aluminium skins lead to increased panel strength, suggesting a limited effect of roughness increase to enhance aluminium and skin bonding. Finally, single directed caps in the core achieve better mechanical performance than alternated caps. The results evidence the feasibility of alternative uses for bottle caps into structural and sustainable applications.

1. INTRODUCTION

In recent years, issues related to sustainability have motivated a significant amount of material´s research involving a new pro-ecological perspective. This green concept can be obtained either by designing the recycling of the material after its useful life [1] or by using industrial waste as its structural components [2,3]. In this way, recycled wastes have awakened a new interest to the development of innovative materials which can alleviate the impact from global warming [4-6].

Several wastes are of interest for their reuse in structural components. One of them is the disposable bottle caps. The production of PET bottles in Brazil was approximately of 562 thousand tons in 2012 [7], while more than 56 million tons of PET were annually consumed in 2010 around the globe. From this total, 18.9 million tons consisted of PET bottles [8]. Although PET is considered an easy-recycled material, its bottle caps made of polyethylene (PE) do not have a mutual processing with the PET bottle, bringing high processing costs. In the United States, while

PET bottles have a 25% recycling rate, their caps represent only 9% of the total plastic components available for recycling [9]. The critical scenario is also found in Brazil, where the recycling rate of high density polyethylene is around 23%, while PET represents 42% of the total of plastics recycled [10].

Sandwich panels are characterized by a three-layer structure, in which the external facings are stiff and resistant, consisting of a dense material. Its core, on the other hand, is composed of a less rigid and ultra-low-density material, which may be arranged in several configurations or even be made up of foam [11]. The bond between the core and the external skins can be ensured by means of an adhesive film or resin injection in its interface [12]. The honeycomb core is the most common core type in sandwich panels. This structure is based on a biaxial support of the facings. The most used honeycomb cell geometry is the hexagonal cells. However, the use of an innovative cell geometry based on circular tubes as cells core has been reported as a promising configuration by recent researches. Oruganti and Ghosh [13] have compared the circular and hexagonal cell honeycombs under fatigue and shear loads. Circular honeycombs have exhibited enhanced strength due to wall restrictions to buckling. The results were later confirmed by Lin, Cheng and Huang [14]. In addition, a sustainable panel based on bottle caps and aluminium skins was proposed by Oliveira et al. [15] in order to reduce the impact of disposing of PP wastes in the environment. These panels achieved acceptable mechanical strength and stiffness for secondary structural applications, revealing a new recycling route for plastic bottle caps.

Considering that PE is the most consumed plastic in Brazil between 2008 and 2012 [16], those results evidence a viable method to address the large disposal of polyethylene caps. In addition, the divergent properties of polyethylene when compared to polypropylene [17] create further challenges in their utilization in structural applications. Therefore, this work investigates a recycled circular honeycomb core in sandwich panels made by polyethylene (PE) caps from drinking water bottles and aluminium facings. A full factorial design (DoE) was carried out to verify the effects of the following factors, type of adhesive, geometric position of the caps, and aluminium faces roughness on the core shear stress, core shear stiffness, facing stress, flexural stiffness and flexural strength of the sandwich panels.

2. MATERIALS AND METHODS

2.1. Sandwich panel

The sandwich structures were manufactured by manual lamination at room temperature (~25oC) consisting of aluminium sheets as facing and polyethylene water bottle as core material. Aluminium sheets (type 1200 H14 [18] with dimensions 243 x 91 x 0.5mm) were supplied by Alumiaço (Brazil) in two types of surface finishes: smooth and brushed. Epoxy (Huntsman type M, Amine-based hardener type HY951) and polyester (Huntsman type Polylyte, Organic Peroxide-based hardener type MEK) polymers were used as adhesive bonding between the adjacent facings and the core and were sourced by MaxiEpoxi (Brazil). The disposed caps were collected from water PET bottles. The recycled caps were washed and dried at room temperature for 24h.

2.2. Experimental planning and testing

The Design of Experiment (DoE) is a methodology involving of a collection of statistical techniques that offers a robust method for planning and analysing the experiments [19-20]. A full factorial design 2^3 was performed to investigate the effect of the factors, orientation of the bottle caps (caps placed along the same direction or in an alternated pattern – see Figure 1), type of adhesive (epoxy and polyester) and surface roughness of aluminium facings (smooth and rough surface) on the mechanical properties of the panels. The experimental factors are summarised in Table 1. The following factors were kept constant: type of aluminium (Type ISO 1200), type of bottle cap (from water bottles), adhesive volume fraction (equivalent to 1 mm of thickness), resin-hardener mixture time (5 min), drain direction of rough aluminium surfaces (longitudinal direction), and cure time (7 days at approximately 23ºC). Preliminary tests pointed out the use of longitudinal drains promoted higher mechanical performance under flexural loadings. Four test samples were fabricated for each one of the 8 (eight) experimental conditions from 2^3 factorial (see Table 2). Two replicates were considered in the experiment, running the total of 64 samples. Minitab^{TM} v.17 [21] was used to manipulate the data. Table 2 exhibits the experimental matrix design.

Table 1. Factor analysed in the 2^3 full factorial design

Fators	Experimental levels
Bottle caps orientation	Same direction
	Alternated direction
Type of adhesive	Epoxy
	Polyester
Surface roughness	Smooth surface
	Rough surface

Table 2 – Experimental matrix design

Condition	Bottle caps orientation	Surface roughness	Type of adhesive
C1	Single direction	Smooth surface	Polyester
C2	Single direction	Smooth surface	Epoxy
C3	Single direction	Rough surface	Polyester
C4	Single direction	Rough surface	Epoxy
C5	Alternated direction	Smooth surface	Polyester
C6	Alternated direction	Smooth surface	Epoxy
C7	Alternated direction	Rough surface	Polyester
C8	Alternated direction	Rough surface	Epoxy


(a)


(b)

Figure 1: Alternated (above) and single directed (below) bottle caps configurations (a) and micro-hardness bottle caps samples (b).

The bottle caps were characterised using micro-hardness test in square samples of 10 mm size (see Figure 1.b). The test was conducted in a Mitutoyo MVK -G1 machine, which determined the Vickers hardness (HV) of the material. Elastic modulus, tensile strength and shear strength were then obtained by Equations (1) to (3), as summarised by Oliveira et al. [15].

$$\sigma_{max} = \frac{H_V}{3} \tag{1}$$

$$\tau_{max} = \frac{H_V}{6} \tag{2}$$

$$E = H_V * 20 \tag{3}$$

The sandwich panels were characterized using three-point bending test following the ASTM C393 protocol [22]. The mechanical tests were performed in a Shimadzu universal testing machine (AG-X model) with a load cell of 100 kN. The 3P flexural loading had a cross-head rate of 6 mm/min and span length of 150 mm. Tests were performed at room temperature (~23°C) at a humidity level of 58%.

The mechanical properties (responses) evaluated via DoE were: the core shear modulus (G_f, calculated according to ASTM 7250 [23]), the core shear ultimate and facing stresses (F^{ult} and σ, respectively, determined via ASTM C393 [22]), and the flexural stress and the flexural stiffness (σ_f and E_f, following the ASTM D790 protocol [24]). The core shear modulus was calculated following the set of equations (4) - (7):

$$D = \frac{E_{facing} * (d^3 - c^3) * b}{12} [N.mm^2] \tag{4}$$

$$U_i = \frac{P_i * (S - L_1)}{4 * \left(\Delta - \left(\frac{P_i}{96 * D} * (2 * S^3 - 3 * S * L_1{}^2 + L_1{}^3) \right) \right)} [N] \tag{5}$$

$$G_i = \frac{U_i * (d - 2 * t)}{(d - t)^2 * b} [MPa] \tag{6}$$

$$G_f = \frac{\sum_{i=1}^{10} G_i}{10} [MPa] \tag{7}$$

Equations (4) - (7) can be used only if the elastic stiffness of both facings are identical and

can be previously determined (ASTM D7250 [23]). D is the flexural stiffness, with Efacing being the elastic moduli of the facing. The shear rigidity (Ui) and the core shear modulus (Gi) are calculated based on flexural stiffness (D) for a series of ten applied forces evenly spaced up to the maximum force. Pi is the force level considered (in N), d is the sandwich thickness, t is the nominal facing thickness, b is the sandwich width, L1 is the load span length (for 3-point loading configuration, L1 = 0), S is the support span length, and Δ is the beam mid-span deflection (in mm) at each force level considered. If the response of the sandwich structure is linear, then the overall core shear modulus (Gf) can be calculated using the values from all forces level (Eq. (7). The core shear ultimate stress and the facing stress can be calculated based on Eqs. (8) and (9) (ASTM C393 [22]):

$$F_s^{ult} = \frac{P_{max}}{(d + c) * b} [MPa] \tag{8}$$

$$\sigma = \frac{P_{max} * S}{2 * t * (d + c) * b} [MPa] \tag{9}$$

Pmax is the maximum force and c is the core thickness (calculated from c = d – 2*t). Finally, the flexural strength σf and modulus can be described as Eqs. (10) and (11) (ASTM Ef D790 [24]).

$$\sigma_f = \frac{3 * P_{max} * S}{2 * b * d^2}\ [MPa] \quad (10)$$

$$E_f = \frac{S^3 * m}{4 * b * d^3}\ [MPa] \quad (11)$$

The additional parameter m in Eq. (11) is the slope coefficient of the initial straight-line portion of the load deflection curve.

2.3 Fabrication

The sandwich panels were fabricated under manual uniaxial compaction (3.5 kPa) at room temperature (~23°C) for 24h. All aluminium sheets (with smooth and rough surfaces) were washed and cleaned by sandpapering and acetone to remove possible oxides formed on the metallic surface see Figure 2.a). This treatment has been used to eliminate poor adhesion between the polymer and the substrate surface [25]. Subsequently, a mixture containing Wash Primer Phosphate 045 and Catalyst 051 in a 2:1 ratio was uniformly spread on the aluminium surfaces. The Wash Primer is based on phenolic compounds to improve the adhesion between metal surfaces and polymer films. After 21 minutes cure interval of the Wash Primer, the sheets were covered by a PVC plastic film to avoid resin leakage (Figure 2.b). The covered facings were then introduced into the moulds lined with release fabric (Armalon). The polymer resin used was then prepared according to the ratio specified by the manufacturer and spread uniformly within the prismatic mould, forming an adhesive layer of approximately 1 mm of thickness. Finally, the bottle caps were hand inserted within the mould as defined orientation (Figure 2.c). After the 24-hour compaction time, the second aluminium skin was attached considering an analogous procedure. A curing time of 7 days was adopted to obtain the resulting sample (Figure 2.d).

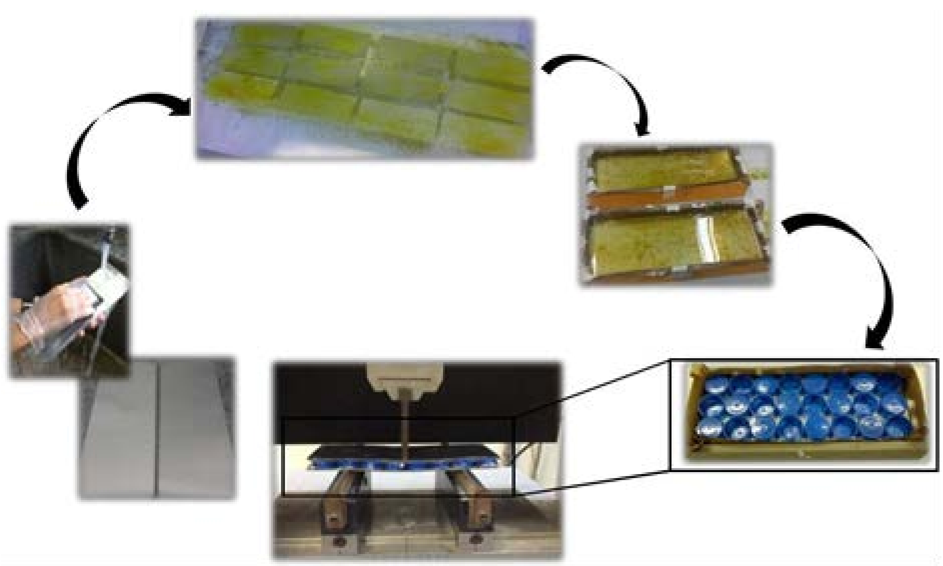


Figure 2: Sample manufacturing process based on surface treatment (a), preparation of the mould (b), bottle caps allocation (c), and finished sample during test (d).

3. RESULTS

The Analysis of Variance (ANOVA) is shown in Table 3, exhibiting the P-Values for each factor and interactions. When a P-Value is equal or less to 0.05 (see bold values in Table 2), it indicates that the factor or interaction significantly affects the response within a confidence interval of 95%. Superior order interactions are preferably analysed instead of individual factors when considered significant. In this case, P-Values are highlighted in italic and underlined. Table 3 also reports the results of R^2(adj), which indicates the adjustment of the experimental data over the statistical model. Higher values of R^2 (near 100%) indicate a greater predictability of the statistical model. ANOVA results reveal R2 varying from 98.82 to 99.89%, which demonstrate excellent adjustments of the data to the model.

A mean micro-hardness of polyethylene was 4.06±0.02 HV. The converted value for micro-hardness in MPa and the results obtained from Equations (1) to (3) for polyethylene elastic modulus, tensile and shear strength are presented in Table 4. The results are in agreement with the literature [17].

Table 3: ANOVA, P-Values for the analysed mechanical responses.

	Factors	Core Shear Stress (MPa)	Facing Stress (MPa)	Core Shear Stiffness (MPa)	Flexural Stiffness (MPa)	Flexural Strength (MPa)
Main	**Bottle Caps Orientation (BO)**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**
	Surface Roughness (SR)	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**
	Type of Adhesive (TA)	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**
Interactio	**BO*SR**	**0.000**	**0.000**	**0.000**	**0.000**	**0.000**
	BO*TA	**0.000**	**0.000**	**0.000**	0.303	**0.000**
	SR*TA	**0.000**	**0.000**	0.354	0.144	**0.000**
	BO*SR*TA	***<u>0.000</u>***	***<u>0.000</u>***	***<u>0.001</u>***	***<u>0.008</u>***	***<u>0.000</u>***
	R^2 (adj) (%)	**99.89**	**99.89**	**97.27**	**98.82**	**99.60**

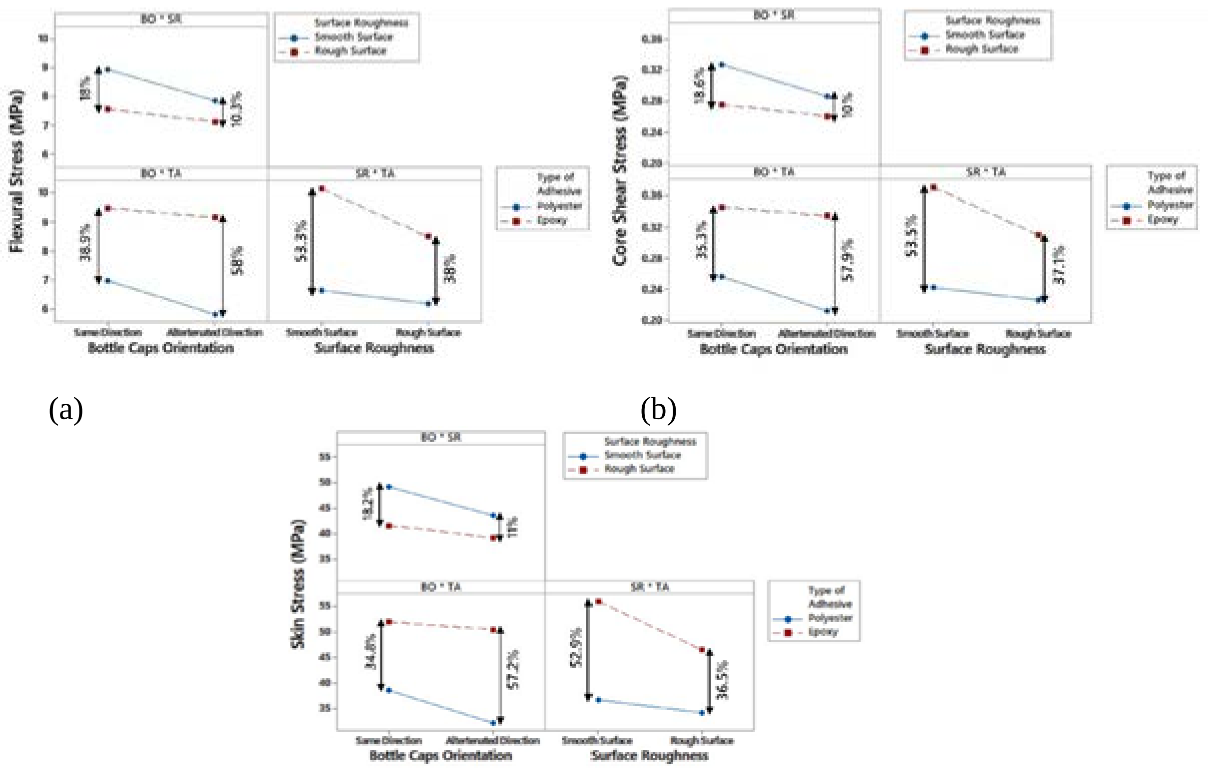
Table 4: Characterisation of core material by micro-hardness test.

Parameters	Values
Hardness (MPa)	**39.8**
Elastic Modulus (MPa)	**795.6**
Tensile strength (MPa)	**13.3**
Shear Strength (MPa)	**6.3**

3.1. Flexural Stress, Core Shear Stress and Facing Stress

The flexural stress, core shear stress and facing stress provided similar mechanical behaviour, since they are directly dependent on the maximum flexural load and geometrical parameters, as shown in Equations (5) to (7). For this reason, these response-variables were analysed together. The flexural stress varied from 5.69 to 10.29 MPa. Core shear stress results were between 0.209

and 0.378 MPa. Finally, the results for skin stress ranged between 31.31 and 56.75 MPa. Third order interaction effects were identified for these responses (see Table 2), which are presented in Figures 3(a) to 3(c). The use of aluminium skins with smooth surface led to enhanced mechanical performance compared to the rough surface. In contrast, Oliveira et al. [15] revealed a reduction in bonding when smooth aluminium surface was used without any treatment. In this case, however, a surface treatment based on sandpapering, full cleaning with acetone, and WashPrimer pulverisation was more effective when applied to smooth aluminium skins, reaching moderate roughness values. As shown in plot (i) of Figures 3(a), 3(b), and 3(c), the smooth treated surface achieved higher response values when the bottle caps were placed in a single direction, revealing increases between 18 and 18.6%. The type of adhesive factor had major influence on the panel strength, in which epoxy polymer provided higher stresses under flexural loads, as seen in Figures 3(a), 3(b) and 3(c), plots (ii) and (iii). The enhancement in strength by the use of epoxy polymer varied from 34.8 to 38.9% for single directed caps, which had the most favourable configuration, and from 57.2 to 58% for caps in alternated directions. Caps in alternated directions led to reduced strength compared to the configuration of single directed caps when associated with the polyester adhesive (Figures 3(a) to 3(c), plot ii). An opposite behaviour was found in a previous study [15], which used polypropylene bottle caps. In this case, alternated caps presented a better distribution of the surface contact area between the caps and the skins, enhancing the adhesion and the panel strength. In the present study, however, the flat geometry of bottle caps and their reduced elastic properties may have affected the alternated configuration reinforcement parameters. In alternate configuration, it was identified that the flat-closed surface of PE cap expels the excess resin into the adjacent open cells when pressed against aluminium skin, which creates a weak connection between the skin and the core. This process was favoured by the lower viscosity of the polyester polymer, which flowed easily between the caps during manufacture. In the single direction configuration, although the pressure of the flat surface was also present, the resin could not be expelled into the adjacent cells. It resulted in a greater peripheral contact between the adhesive and the caps, besides greater thickness of the adhesive layer. On the other hand, the caps orientation had almost no significant influence when associated with epoxy adhesive. Finally, the aluminium smooth surface promoted a positive effect on the type of polymer, showing significant improvements between 52.9 and 53.5% when considering the epoxy polymer (Figures 3(a) to 3(c), plot iii). This behaviour suggests less interaction between the polyester resin and the other components. In addition, higher strength of samples with a smooth surface indicates a limited surface roughness effect to promote better bonding between the skin and the adhesive.

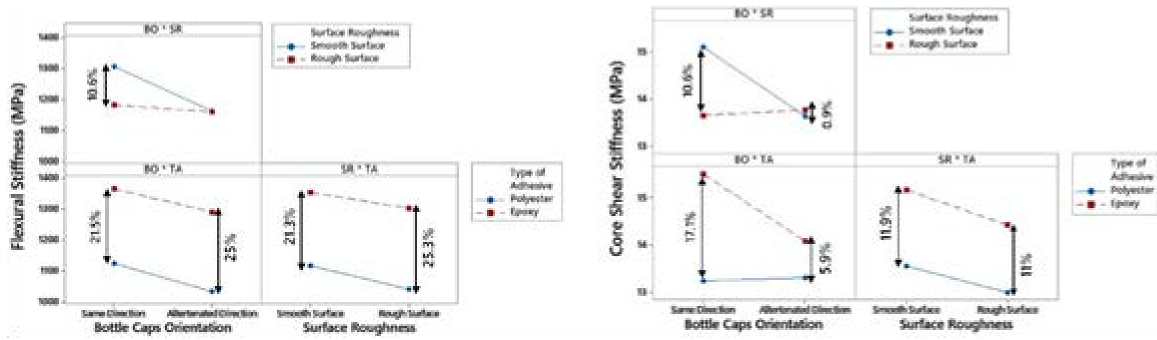


(a) (b) (c)

Figure 3: Interaction plot for flexural stress (a), core shear stress (b) and skin stress (c).

3.2 Flexural and Core Shear Stiffness

In general, the sandwich panel stiffness relates to the facing-core interface, the core and facing elastic behaviour and the inertial effect between the core and the facing constituents [26-27]. Besides, the stiffness of the structure is directly dependent on the configuration and topological parameters (Eq. (6) and Eq. (11)). The flexural stiffness of the sandwich structure varied from 991.90 MPa to 1437.47 MPa, while the core shear stiffness ranged from 12.73 MPa to 16.70 MPa. A third order interaction effect was significant for both responses. The interaction effect plots are shown in Figures 4(a) and 2(b). The flexural modulus and core shear stiffness reveal similar behaviour to the responses previously mentioned. In this way, the sandwich panel stiffness is predominately dependent on the type of adhesive. Epoxy polymer presented further enhanced flexural and core shear stiffness values when compared with polyester adhesive. The type of adhesive provided reduced interaction with the remaining factors for the flexural modulus. However, the adhesive led to significant enhanced stiffness (about 17.1%) when associated with single directed caps configuration, and a slight increase for smooth aluminium skins. Finally, the aluminium roughness presented divergent behaviour depending on the bottle caps configuration. The use of alternated caps led to similar and reduced results for both types of aluminium surfaces. On the other hand, caps in a single direction achieved superior stiffness for smooth and treated aluminium surfaces, with an increase of 10.6% when compared with rough skins.



(a) (b)

Figure 4: Interaction effect plot for flexural stiffness (a) and core shear stiffness (b).

3.3 Results comparison

The results obtained in this research were compared with those of a previous research with PP bottle caps honeycomb developed by Oliveira et al. [15] and the sustainable honeycomb developed by Cabrera, Alcock, and Pejis [1], which consisted of a sandwich panel made of polypropylene tubular honeycomb. The most favourable experimental condition based on epoxy adhesive, aluminium smooth skin and single directed caps (Condition 2) was considered for comparison, which is shown in Table 3. The sandwich panel presented in this work obtained reduced mechanical properties compared to the similar bottle caps panel manufactured by Oliveira et al. [15]. This can be explained by the reduction of the mechanical strength and stiffness presented by the polyethylene caps, in addition to the low interaction between the PE caps and the polymer. However, C2 panels led to enhanced absolute and specific properties when compared to the sustainable panel designed by Cabrera, Alcock, and Pejis [1], despite their higher core density. This comparison evidences the feasibility of the proposed panel as a lightweight and low-cost component for secondary structural applications in engineering.

Table 3: Comparison between mechanical properties for PE and PP based panels [1, 15]

Responses	PP Bottle Caps Composite (C2)	PP Bottle Caps Composite [15]	PP composite [1]
Composite Bulk density (g/cm³)	0.418 (0.003)	0.532 (0.104)	0.195
Shear stress (MPa)	0.38 (0.02)	0.77 (0.07)	0.27
Specific Shear Stress (N-m/g)	**0.91 (0.01)**	**1.45 (0.12)**	**1.3**
Skin Stress (MPa)	56.75 (2.98)	115.38 (9.69)	24
Specific Skin stress (N-m/g)	**135.76 (1.53)**	**216.87 (18.22)**	**123.08**

4. CONCLUSIONS

A new concept of circular sandwich panel based on aluminium facings and disposed bottle caps as core were investigated in this work. The structures were evaluated under three-point bending test. The main conclusions are described as follows:

i. The statistical analysis (ANOVA) revealed that a third order interaction between 'Bottles Caps Orientation', 'Surface Roughness' and 'Type of adhesive' significantly affected the core shear stress, facing stress, core shear stiffness, the flexural stiffness and flexural strength;
ii. The type of adhesive was the most relevant factor in the mechanical properties. The epoxy polymer led to enhanced mechanical performance when compared to the polyester adhesive;
iii. Bottle caps oriented along to the same direction achieved superior mechanical properties;
iv. The smooth surface of the aluminium facings promoted a further increase in the shear stresses, maximum force and stiffness;
v. Finally, this sustainable composite design featured a viable route to reuse discarded bottle caps in a lightweight panel for secondary structural applications.

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