Abstract: This work further investigates the manufacture and characterisation of a sustainable sandwich panel made from aluminium skins and a recycled thermoplastic bottle cap core, an innovative concept proposed in a previous paper. A full factorial design based on Design of Experiments (DoE) and Analysis of Variance (ANOVA) techniques has highlighted the complex influence of three manufacturing parameters (type of polymeric adhesive, adhesive layer thickness layer and core packing topology) on the absolute and specific physical and flexural properties of the panels. The ANOVA revealed that the use of higher amount of epoxy polymer led to enhanced panel strength and stiffness. The cell packing topology, however, did not provide a significant effect on most panel properties. Discarded bottle caps have proven to be a promising lightweight and inexpensive honeycomb component for structural applications.

Keywords: Sandwich composites; bottle caps waste; tubular honeycomb; Recycling; Design of Experiments.

1. INTRODUCTION

Sandwich panels consist of two sheets of rigid material connected by a soft and thick core, which can be chemically or mechanically bonded. A variety of core materials can be used in sandwich structures, from polymeric foams to honeycomb lattices [1]. The use of polymeric foam can help to reduce the structural weight and improve insulating properties, while honeycombs are more suitable for enhanced load bearing capabilities. The main advantage of sandwich panels consists in their high specific flexural stiffness, due to the core thickness and the reduced weight. Light-weighting and high strength are the main characteristics that make these materials suitable for aerospace applications [2].
Hexagonal honeycombs are the most common cellular cores used in structural applications [3], and different core topologies with 2D and 3D arrangements have been tested to optimise the mechanical performance of sandwich panels, assessing the effect of internal (e.g. geometry) and external factors (e.g. temperature, load rate) on panel properties [1, 3-5]. Circular cell honeycombs are worthy of a particular mention. These core structures, discussed in technical open literature since the 1960s [1] and investigated at length by Chung and Waas [6], can be used in a variety of applications, such as oil transport in on- and off-shore facilities, and structural applications in sandwich plates [7]. Circular cell honeycombs have been hailed as a very promising core topology to enhance the stiffness and strength of a structure. Oruganti and Ghosh [8] have found that panels with circular cell honeycombs feature a significantly higher stiffness when compared with similar cores made from hexagonal honeycomb cells, due to the higher cell wall buckling loading. Lin, Cheng and Huang [9] have observed similar results, with enhancements of the deformation, strength and yield stress in circular cell cores depending on the cell packing. Different cell packings have been previously used to optimise the strength and stiffness of the resulting sandwich panel (Figure 1). Hu et al. [10] have compared two circular cell packings, the cubic and the hexagonal ones (see Figure 1.a and 1.b, respectively); the configurations present different angles between the adjacent lines that connect the centres of neighbouring tubes. Circular honeycombs with hexagonal packing show a significant higher energy absorption capacity (23%) and better performance under out-of-plane loads than circular honeycombs in cubic packing because of the higher number of restrictions (i.e., the number of adjacent cells in direct contact - 6 for hexagonal packing vs. 4 for cubic packing). A more restricted structure prevents the cell wall buckling and promotes the plastic deformation, leading to a more efficient energy absorption configuration.

![Hexagonal and Cubic Honeycomb Topologies](image)

Figure 1. Two circular honeycomb topologies: hexagonal (a) and cubic packing (b) [10].

The adhesive layer must guarantee an efficient skin-core load transfer, and the enhancement of the structural stiffness can be also achieved by applying surface treatments of the skins, or by controlling the amount of adhesive. The use of surface primers on metallic skins can provide moisture and corrosion protection, and – at the same time - enhance the chemical bonding between the surface
of the skin and the polymeric adhesive [11]. Moreover, a mechanical pre-processing of the metallic skins based on abrasion with sandpaper tends to remove the passive oxide layer adsorbed on the surfaces, and that enhances the chemical interaction between the skin and the adhesive and increases the surface roughness [12]. Higher quantities of adhesive tend to correlate with an improved mechanical performance of the structures [13-16]. The quality of the adhesion is mostly attributed to the amount and the distribution of the adhesive layer. Okada and Kortschot [13] have found that larger and more regularly distributed adhesive fillets tend to enhance the energy absorption capacity. The presence of more uniformly distributed regular fillets is correlated to higher amounts of adhesive, which are able to form a meniscus near the cell walls [14]. Jen, Ko and Lin [15] have assessed the performance of three different adhesive levels (400, 700, and 1000 g/m²) on the fatigue behaviour of bonded aluminium honeycomb sandwich beams. The structures with the highest amount of adhesive showed the best fatigue life performance.

Sustainability is now one of the main concerns when developing innovative structural materials. The increasing costs of landfills in addition to new regulations for the disposal of end-of-life materials (such as the EU directive for vehicles [17]) impose the development of new solids with easy recyclability, and the use of eco-friendly components in manufacturing. An example of sustainable tubular honeycomb core is the structure proposed by Cabrera, Alcock, and Pejis [18]. This sandwich panel is composed by a tubular honeycomb made of polypropylene and bonded onto a polypropylene skin with a polypropylene (PP) based adhesive, creating therefore an all-PP structure. The use of a single-phase material facilitates the recyclability of the core and the structure, which could be melted in a single step. The overall mechanical performance of all-PP structures is considered adequate when compared to a similar structure made from glass fibre skins. Another type of sustainable sandwich structure is the one made from bottle caps (recycled tubular honeycomb core) and embedded in sandwich structures, as trialled in a previous work by the authors [19]. Polypropylene caps are used to close soft plastic bottles in a variety of beverages but cannot be recycled with the PET bottles, and that results in a more expensive recycling process. Moreover, recycled PP tends to possess less strength and stiffness due to the rupture of the PP polymeric chains [20, 21]. Our previous work [19] described the manufacturing of a sandwich panel made of aluminium skins and a bottle cap core bonded with epoxy polymer adhesive. The orientation of the bottle caps (single direction and/or alternated caps) and the use of adhesive between the caps were the fundamental design parameters of the concept. The panels with the alternated bottle caps configurations showed a 20% higher strength compared to the structures with the single aligned caps. The adhesive between caps and the adjacent cells contributed to a further enhancement of the panel strength between 24% and 33%.
The potential re-use of the PP bottle caps is very appealing, since their disposal is very damaging for the environment and wildlife. The estimated amount of bottle caps annually disposed of in landfills is around 320,000 tonnes [22]. Bottle caps have been ranked amongst the top ten items polluting the oceans [23]. In 2007 only, the annual production of PET bottles in Brazil was around 9 billion units [24]. However, there is a large difference in terms of PET and PP recycling rates in different countries. In the US, the recyclability of PET is around 25%, while for PP items is up to 9% only [25]. In Brazil, the recyclability rates of PET and PP were respectively 42% and 9% in 2016 [26]. These figures highlight the urgent need of innovative designs of sustainable products, potentially based on the use of recycled bottle caps.

The present work is focused on the development of a sandwich panel based on aluminium skins and a sustainable core made of bottle caps. The promising findings obtained in the previous research [19] stimulated further investigations on bottle cap panels by testing new architectural designs to improve mechanical properties. A Full Factorial Design was used to identify the effects on the mechanical and physical responses of the panels upon modifications of the bottle caps cells packing (hexagonal and cubic packing), the type of polymeric adhesive (epoxy and polyester adhesive), and the average thickness of the polymeric adhesive (0.8 and 1.5 mm). The mechanical properties considered here are the flexural strength, flexural modulus, skin stress, core shear modulus, core shear stress, and bulk density of the core/sandwich panel.

2. METHODOLOGY

a. Materials

Sandwich panels were manufactured using a pair of aluminium sheets connected to a core of polypropylene bottle caps by polymeric adhesives. The aluminium sheets (brushed aluminium type ISO 1200 [27] with 0.5 mm thickness, see Figure 2.a) were sourced from Barro Preto Metais (Brazil). Two thermoset polymers were evaluated: an epoxy (Renlam M-1 resin type with Amine-based hardener type - HY 951) and a polyester (Polylyte resin type with MEK hardener type), both supplied by Huntsman (Brazil). Tensile, compressive and flexural strength and modulus of both polymers are presented in Table 1. These properties were determined via tensile, compressive and flexural tests according to the D638 [28], D695 [29], and D790 [30] ASTM standards. A finishing primer supplied by Sherwin Williams was used to enhance the chemical interaction between the aluminium surface and the polymeric adhesive. The primer solution was composed by a Wash Primer (045 type) and a catalyst (051 type) (see Figure 2b). The disposed bottle caps were obtained from soft drink bottles (Figure 2c). The bottle caps were made with a special type of polypropylene for high impact strength [31]. The
caps were obtained from local recycling centres and were washed and dried at room temperature for approximately 24h.

Table 1. Tensile, compressive and flexural results for epoxy and polyester polymers in pristine condition

<table>
<thead>
<tr>
<th>Mechanical Responses</th>
<th>Epoxy Polymer</th>
<th>Polyester Polymer</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mean  SD  VC</td>
<td>Mean  SD  VC</td>
</tr>
<tr>
<td>Compressive elastic modulus (GPa)</td>
<td>2.57  0.07  2.6%</td>
<td>1.94  0.10  5.0%</td>
</tr>
<tr>
<td>Compressive strength (MPa)</td>
<td>80.46 2.96 3.7%</td>
<td>60.30 3.45 5.7%</td>
</tr>
<tr>
<td>Tensile elastic modulus (GPa)</td>
<td>2.25 0.11 5.0%</td>
<td>2.46 0.20 8.1%</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>48.27 1.55 3.2%</td>
<td>37.73 3.39 9.0%</td>
</tr>
<tr>
<td>Flexural modulus (GPa)</td>
<td>2.22 0.09 4.2%</td>
<td>2.30 0.15 6.5%</td>
</tr>
<tr>
<td>Flexural strength (MPa)</td>
<td>70.99 2.63 3.7%</td>
<td>55.82 7.18 12.9%</td>
</tr>
</tbody>
</table>

SD = standard deviation, VC = variation coefficient

Figure 2. Aluminium skins (a), Wash Primer components (b) and disposed bottle caps (c) used for sandwich panel manufacturing.

b. Full Factorial Design

A Full Factorial Design $2^3$ was used to verify the effects of the geometric and material factors over the mechanical responses using a Design of Experiment (DoE) technique. The factors considered were: (1) the packing of the bottle caps (cubic or hexagonal - see Figure 1), (2) the type of adhesive (epoxy or polyester polymer) and (3) the average thickness of the adhesive layer (0.8 and 1.5 mm). The adhesive thickness levels were chosen based on previous work [19], which used adhesive layer of 1mm. Three-point bending tests were carried out to determine the responses (skin stress, core shear stress and modulus, flexural strength and modulus). In addition, the bulk density of the panels was determined to obtain their specific properties. Other factors related to the manufacturing process were kept constant, namely: the polymer/catalyst ratio (10:1 for epoxy and 50:1 for polyester), the time for the polymer mixture (2 minutes), the curing time of the samples (7 days at room temperature, at approximately 22°C), and the type of aluminium skin (brushed, 0.5 mm thick, ISO 1200 aluminium
Manufacturing parameters were considered based on previous work [19], namely the alternated orientation of the bottle caps, the use of inter-cap connections, and the type of bottle caps (30.52 mm in diameter and 12.4 mm in height). The summary of the experimental conditions is shown in Table 2. The combination of the three factors are displayed in Table 3, providing 8 experimental conditions. Four samples were produced per experimental condition, with two replicates, resulting in a total of 64 samples. The Analysis of Variance (ANOVA) was used to evaluate the significance of each experimental factor and its respective levels on the mechanical responses, within a confidence interval of 95% [32, 33]. The software Minitab 17 [34] was used to analyse statistically the results via DoE and ANOVA.

Table 2. Factors and their respective levels analysed in this work.

<table>
<thead>
<tr>
<th>Factors</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Circular cell packing</td>
<td>Hexagonal</td>
</tr>
<tr>
<td></td>
<td>Cubic</td>
</tr>
<tr>
<td>Type of polymeric adhesive</td>
<td>Epoxy</td>
</tr>
<tr>
<td></td>
<td>Polyester</td>
</tr>
<tr>
<td>Thickness of polymeric adhesive layer</td>
<td>A (1.5 mm)</td>
</tr>
<tr>
<td></td>
<td>B (0.8 mm)</td>
</tr>
</tbody>
</table>

Table 3. Full factorial design ($2^3 = 8$ treatments)

<table>
<thead>
<tr>
<th>Treatments</th>
<th>Polymer Type</th>
<th>Polymeric adhesive thickness</th>
<th>Cell Packing System</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Epoxy</td>
<td>A (0.8 mm)</td>
<td>Cubic</td>
</tr>
<tr>
<td>2</td>
<td>Epoxy</td>
<td>A (0.8 mm)</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>3</td>
<td>Epoxy</td>
<td>B (1.5 mm)</td>
<td>Cubic</td>
</tr>
<tr>
<td>4</td>
<td>Epoxy</td>
<td>B (1.5 mm)</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>5</td>
<td>Polyester</td>
<td>A (0.8 mm)</td>
<td>Cubic</td>
</tr>
<tr>
<td>6</td>
<td>Polyester</td>
<td>A (0.8 mm)</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>7</td>
<td>Polyester</td>
<td>B (1.5 mm)</td>
<td>Cubic</td>
</tr>
<tr>
<td>8</td>
<td>Polyester</td>
<td>B (1.5 mm)</td>
<td>Hexagonal</td>
</tr>
</tbody>
</table>

c. Manufacturing and testing

The aluminium sheets were cut into the recommended dimensions (246.5 x 91.5 mm) according to the ASTM C393 standard [35]. The skins were subsequently degreased and sandpapered under flowing water to remove the passive oxide layer on the aluminium surface (see Figure 3a). The aluminium skins were then cleaned with acetone for full removal of the remaining dirt. Finally, the Wash Primer solution was finely pulverised over the clean aluminium surfaces (see the yellow Wash Primer layer in Figure 3b) and left for cure in 20 minutes at room temperature.
The manufacture of the sandwich samples was performed in two steps. The first consisted on bonding one skin to the bottle caps core. The external aluminium surface after treatment (Figure 4a) was then covered by a plastic film to avoid resin leakage from the treated aluminium surface (Figure 4b). The skins were inserted into specific wood moulds over an *Armalon* layer for mould protection and easier removal of the final specimen. Subsequently, the polymeric adhesive was prepared and spread over the aluminium skin. After that, the bottle caps were placed over the skin according to the packing system adopted for each condition (Figure 4c). The mould was closed with a wood lid and the specimen was compacted under 3500 Pa cold pressure for 24 h at room temperature. The second step of the manufacturing consisted in bonding the other skin in a similar process (Figure 4d) with a 24h compaction, followed by curing for 7 days at room temperature. An example of a resulting sample with dimensions 246.5 x 91.5 x 13.5 mm is displayed in Figure 4e.

The mechanical properties of the sandwich plates were measured from three-point bending (3P) tests performed using a universal testing machine Shimadzu AGX with a 100 kN load cell (see Figure
The test parameters (150 mm span and 6 mm/min cross head speed) were specified according to the ASTM C393 standard [35].

![Cross-head loading rig](image)

**Figure 5.** Three-point bending test rig in universal testing machine.

The responses analysed in DoE were the flexural stress and flexural modulus ($\sigma_f$ and $E_f$, according to ASTM D790 [30]), the core shear modulus ($G_c$, calculated following the ASTM D7250 protocol [36]), and the core shear ultimate and skin stresses ($F_s^{ult}$ and $\sigma$ [35]). The bulk density was determined by weighing each sample using a precision scale (ASTM D792-13 [37]). The core shear ultimate stress and the skin stress were obtained through Equations 1 and 2 (ASTM C393 [35]):

$$F_s^{ult} = \frac{P_{\text{max}}}{(d + c) \times b} \text{ [MPa]} \quad (1)$$

$$\sigma = \frac{P_{\text{max}} \times S}{2 \times t \times (d + c) \times b} \text{ [MPa]} \quad (2)$$

The term $P_{\text{max}}$ represents the maximum load detected in flexural test prior to failure (in N), $S$ is the span length, $t$ is the nominal skin thickness, $d$ is the measured sandwich thickness, $c$ is the estimated core thickness (obtained from $c = d - 2 \times t$), and $b$ is the measured sandwich width. The lengths were measured in mm. The calculation of core shear modulus is determined by the following equations (Equations 3 to 6):

$$D = \frac{E_{\text{skin}} \times (d^3 - c^3) \times b}{12} \text{ [N mm$^2$]} \quad (3)$$

$$U_i = \frac{P_i \times (S - L_1)}{4 \times \left( \Delta - \left( \frac{P_i}{96 \times D} \times (2 \times S^3) \right) \right)} \text{ [N]} \quad (4)$$

$$G_i = \frac{U_i \times (d - 2 \times t)}{(d - t)^2 \times b} \text{ [MPa]} \quad (5)$$
\[ G_f = \frac{\sum_{i=1}^{10} G_i}{10} \text{ [MPa]} \]  

The requisite for using equations 3 to 6 is the previous measurement of the elastic modulus of the upper/lower skins, which need to be considered identical (ASTM D7250 [36]). The flexural stiffness (D) is first determined, being \( E_{\text{skin}} \) the Young’s modulus of the skin (see Equation 3). The shear rigidity \( (U_i) \) and the correspondent core shear modulus \( (G_i) \) are calculated for ten evenly applied forces, up to the maximum load (see Equations 4 and 5). \( P_i \) is the load level considered (in N), \( L_1 \) is the load span length (only applicable for four-point bending test; for 3PB test, \( L_1 = 0 \)) and \( \Delta \) is the experimental mid-span deflection of the beam (in mm) at each force load considered. The overall core shear modulus \( (G_f) \) is calculated by the average value of the ten partial core shear moduli when the overall response is linear (see Equation 6). Finally, the flexural strength and stiffness are obtained through Equations 7 and 8 (ASTM D790 [30]):

\[ \sigma_f = \frac{3 \times P_{\text{max}} \times S}{2 \times b \times d^2} \text{ [MPa]} \]  

\[ E_f = \frac{S^3 \times m}{4 \times b \times d^3} \text{ [MPa]} \]

For equation (8), the slope coefficient \( m \) (in N/mm) indicates the slope of the straight-line portion of the load deflection curve.

3. RESULTS AND DISCUSSION

The results for the statistical analysis are presented in Table 4. The P-Values in the table indicate the factors and interactions of factors that significantly influence the mechanical responses. P-Values lower than 0.05 (in bold) indicate a statistically significant effect associated with that particular factor. In some situations, the interaction of factors is significant, which means that the effect of one factor is dependent on the levels of another factor. Whenever an interaction is significant, it should be analysed instead of considering the individual factors only [32, 33]. Table 4 also shows the \( R^2 \) values to indicate the reliability of the statistical model to predict responses for new observations. Higher values of \( R^2 \) imply a model of greater predictability. The values of \( R^2 \) varied from 83.9% to 98.8%, which show a more than satisfactory adjustment of the model to the experimental data. ANOVA assumes that the experimental data follow a normal distribution described by a homogeneous variance for all the responses considered. The normality of the data was verified via the Anderson-Darling test, in which P-Values higher than 0.05 indicate a set of data that follows a normal distribution. This condition was verified in all the responses, validating therefore the ANOVA
findings. Finally, the Bartlett test results ensure the homogeneity of the variances within the analysed data, which is verified when the P-Value is above 0.05. All the responses fulfilled this condition (see Table 4). The underlined P-values in Table 4 indicate the factors or interactions that will be analysed via effect plots.

Table 4. P-Value results from ANOVA.

<table>
<thead>
<tr>
<th>Resin Type (RT)</th>
<th>Maximum Load</th>
<th>Core shear stress</th>
<th>Skin stress</th>
<th>Core shear modulus</th>
<th>Flexural modulus</th>
<th>Flexural strength</th>
<th>Specific core shear stress</th>
<th>Specific skin stress</th>
<th>Specific core shear modulus</th>
<th>Specific skin modulus</th>
<th>Specific modulus</th>
<th>Specific flexural strength</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.041</td>
<td>0.011</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.034</td>
<td>0.028</td>
<td>0.000</td>
<td>0.000</td>
</tr>
<tr>
<td>Adhesive thickness (AT)</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
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<td>0.000</td>
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<td>0.000</td>
<td>0.000</td>
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<tr>
<td>Cell Packing (CP)</td>
<td>0.070</td>
<td>0.333</td>
<td>0.333</td>
<td>0.008</td>
<td>0.247</td>
<td>0.317</td>
<td>0.928</td>
<td>0.928</td>
<td>0.352</td>
<td>0.674</td>
<td>0.861</td>
<td>0.001</td>
</tr>
<tr>
<td>RT * AT</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.000</td>
<td>0.001</td>
</tr>
<tr>
<td>RT * CP</td>
<td>0.854</td>
<td>0.421</td>
<td>0.421</td>
<td>0.067</td>
<td>0.112</td>
<td>0.295</td>
<td>0.494</td>
<td>0.494</td>
<td>0.406</td>
<td>0.167</td>
<td>0.330</td>
<td>0.001</td>
</tr>
<tr>
<td>AT * CP</td>
<td>0.557</td>
<td>0.591</td>
<td>0.591</td>
<td>0.063</td>
<td>0.310</td>
<td>0.546</td>
<td>0.883</td>
<td>0.883</td>
<td>0.091</td>
<td>0.649</td>
<td>0.959</td>
<td>0.001</td>
</tr>
<tr>
<td>RT * AP * CP</td>
<td>0.125</td>
<td>0.188</td>
<td>0.188</td>
<td>0.675</td>
<td>0.387</td>
<td>0.229</td>
<td>0.202</td>
<td>0.202</td>
<td>0.226</td>
<td>0.341</td>
<td>0.213</td>
<td>0.001</td>
</tr>
<tr>
<td>R² (%)</td>
<td>98.82</td>
<td>98.64</td>
<td>98.64</td>
<td>97.63</td>
<td>95.01</td>
<td>98.46</td>
<td>97.11</td>
<td>97.11</td>
<td>90.22</td>
<td>83.91</td>
<td>97.05</td>
<td>97.05</td>
</tr>
<tr>
<td>Anderson Darling</td>
<td>0.981</td>
<td>0.886</td>
<td>0.886</td>
<td>0.922</td>
<td>0.692</td>
<td>0.669</td>
<td>0.906</td>
<td>0.906</td>
<td>0.127</td>
<td>0.258</td>
<td>0.991</td>
<td>0.001</td>
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<tr>
<td>Bartlett Test</td>
<td>0.671</td>
<td>0.632</td>
<td>0.632</td>
<td>0.612</td>
<td>0.384</td>
<td>0.675</td>
<td>0.440</td>
<td>0.440</td>
<td>0.367</td>
<td>0.264</td>
<td>0.367</td>
<td>0.001</td>
</tr>
</tbody>
</table>

3.1 Maximum load, core shear stress, skin stress and flexural stress

Core shear, skin and flexural stress directly depend on the maximum bending load. All these responses present similar results according to the ANOVA and will therefore be analysed together. Figure 6 shows that these responses were affected by the interaction between the resin type and adhesive thickness factors (Figures 6a to 6d). The highest amount of adhesive (1.5mm) led to an enhancement of the properties between 53% to 55.9% when the polyester was used. The adoption of the epoxy resin as adhesive further increased the mechanical properties, from 92 to 92.4%. Owing to the larger contact area between the adhesive layer and the remaining components (the core bottle caps and the aluminium skin), a larger thickness of the adhesive was able to create higher-quality adhesive fillets between the cap walls and the resin [13, 14], therefore enhancing the mechanical strength and preventing premature debonding. The creation of a high quality adhesive layer was only verified when increased amounts of resin were used, with a more uniform distribution among all caps and the core. Unlike the traditional tubular honeycombs, bottle caps present a particular shape, with one closed surface at one end. During the manufacturing process, the compaction tended to expel part of the resin between the closed surface of the bottle caps and the aluminium skin. When a 0.8 mm adhesive layer was used, the remaining resin to connect the closed cap surfaces and the skin was insufficient to provide a good quality bonding. The weak connection between the caps and the skin led to early
delamination of the sandwich panel. The type of resin had also a significant influence over the responses. The most remarkable mechanical response was achieved when the epoxy polymer was used. The polyester reduced the mechanical performance by 44.3 to 44.6% for the cases of the thicker adhesive layers, and from 17.6 to 18.3% for the thinner ones. Smaller reductions were obtained for the panels made with a thinner adhesive layer (0.8mm) due to the lower interaction between the adhesive, skins and core.

![Image](image_url)

**Figure 6.** Interaction effect plots between the factors Type of resin and Adhesive thickness for mean maximum load (a), core shear stress (b), skin stress (c) and flexural stress (d).

3.2 Elastic Properties: Core shear modulus and flexural modulus

The core shear modulus ranged from 13.3 to 30.4 MPa. All three main factors significantly affected this response (see Table 4). The individual effect plots are shown in Figure 7. A slight, yet significant, 7.9% enhancement of core shear modulus was achieved when the epoxy was used (Figure 7a). Thicker adhesive layers provided a significant increase (74.5%) of the core shear modulus (Figure 7b); this was attributed to the higher resin amount in the peripheral areas of the bottle caps. The presence of larger, high quality fillets also contributed to this result, creating a strong connection
between the caps and the resin, and preventing early cap debonding and shear failure of the core cells. The authors have previously reported [19] that the use of an inter-cap connection was effective in reducing the failure of the sandwich panels due to shear loads over the adjacent caps. Finally, the hexagonal packing system was less structurally efficient in the present work, with the core shear modulus slightly higher (11.5%) when the caps were arranged in a cubic packing. The reported increase in strength and stiffness using the hexagonal cells was based on the high number of adjacent cell walls in mutual contact. The bottle caps used in this research present a shape similar to a cone trunk, instead of the perfect cylinder used in the majority of works in open literature. The use of bottle caps along alternated directions in the hexagonal packing implied that some adjacent caps were in contact in a single point, instead of on a surface. This effect is illustrated in Figure 8. Therefore, the number of adjacent cells in full contact in both packings is the same (four cells). The cubic packing created however a more uniform adhesive layer among caps, which generated an increase of the core stiffness against shear loads.

![Figure 7. Main effect plots for the factors Type of resin, Adhesive thickness, and Packing System for mean core shear modulus.](image)

![Figure 8. Type of contact between adjacent cells for the hexagonal packing (a) and cubic packing (b).](image)

The type of polymer and the thickness of the adhesive significantly affected the flexural modulus, with a P-Value lower than 0.05 (see Table 4). Figure 9 shows the main effect plots for the average
flexural modulus. A reduction of 13.1% was observed when the epoxy was replaced with the polyester. The average flexural modulus of the polyester polymer sandwich panels was 2.01 GPa, whereas the mean value for the epoxy case was 2.25 GPa. Both values were similar to the average values of the compressive modulus of polyester and the tensile modulus of the epoxy, which were also the lowest elastic moduli reported in Table 1. This similarity between adhesives and panel moduli shows that the adhesive properties limit the results of the overall sandwich panel [38]. In each case, the variation of the flexural modulus was dependent upon the efficiency of the bonding. Thicker adhesive layer contributed to increase the flexural modulus by 55.4% in comparison with the thinner adhesive case (see Figure 9b). As previously discussed, larger amounts of adhesive improved the effective contact surface with the caps and provided bigger and higher quality fillets, which prevented the debonding of the core and increased the overall panel stiffness.

![Figure 9](image_url)

**Figure 9.** Main effect plots for the factors Type of resin and Adhesive thickness on mean flexural modulus.

### 3.3 Specific Properties: Core shear stress, skin stress and flexural strength

The specific properties of the core shear, skin and flexural strength were similarly affected by a second order interaction between the resin type and the adhesive thickness (Table 4). Specific properties were considered to understand the effect of the amount of adhesive on the sandwich panel efficiency. The use of a 1.5 mm adhesive thickness provided a ~ 50% higher specific properties than the 0.8 mm level, although it led to a ~ 24% increase of density. For a 1.5 mm thickness of adhesive layer, the specific properties increased by 25% when the polyester was used, and by 54% when the epoxy was adopted (Figure 10).
3.4 Specific properties: Core shear modulus and flexural modulus

The specific core shear modulus presented similar trends to the absolute modulus in terms of the influencing factors. Not only the adhesive thickness main factor, but also a second order interaction between the resin type and the packing topology significantly affected the specific core shear modulus (Table 4). Figure 11 shows the effect plots for the average core shear modulus. The use of higher amounts of adhesive provided an 34.5% enhancement of the core shear modulus (Figure 11a). The type of polymer provided different responses, depending on the packing system used. The core shear modulus was reduced by 13.2% when polyester was used with a hexagonal packed core instead of cubic packed core (Figure 11b). In contrast, the hexagonal packing led to the highest core shear modulus when combined with the epoxy polymer (7% higher than the cubic packing). The configuration with the epoxy polymer is the only one in which the hexagonal packing presented better results than the cubic configuration, in an opposite behaviour compared to the behaviour of the absolute value of the core shear modulus. On the other hand, the cubic packing did not exhibit significant
variation between polymers. In terms of the specific elastic modulus, a similar result to the absolute modulus was also found when compared with the sensitivity of the absolute modulus versus the same factors: the modulus increased by 11.5% when the epoxy was used, whereas the use of the thicker adhesive layer contributed to an increase of the specific elastic modulus by 25.3% (Figure 12).

Figure 11. Main effect plots for the factor Adhesive thickness (a) and interaction plot between the factors Type of resin and Packing System for mean core shear modulus (b).

Figure 12. Main effect plots for the factors Type of resin and Adhesive thickness for mean specific flexural modulus.

3.5. Failure analysis

The most common failure mode present in the sandwich panels with the two sets of polymers and adhesive thicknesses was the local debonding of the adhesive from the caps. Failure occurred mostly in the central areas (near the application point of the load), whereas the ends of the sandwich beams remained almost intact. Some common failure modes for the panels with the polyester and epoxy polymers are shown in Figure 13. It must be noted that a reduced number of samples presented however
some partial delamination of the treated aluminium skins, and this shows a better structural efficiency when compared to the untreated skin panels previously investigated [19].

Figure 13. Examples of common failure modes of sandwich panels made of epoxy (a) and polyester resin (b).

The samples with higher amount of polymeric adhesive presented higher toughness than those with low adhesive amount. This is illustrated in Figure 14, which presents the load-displacement plots of samples with epoxy and polymer adhesives for both thickness levels. Higher strength is achieved by epoxy polymer composites fabricated with higher amount of adhesive (see Figure 14.b). Moreover, most 1.5mm thick samples showed progressive failure, which was indicated by the smoother reduction of the load at the end of the curve (see Figure 14.b). On the other hand, a sudden drop in flexural load was identified for 0.8 mm adhesive samples (see Figure 14.a).

Figure 14. Load-displacement of sandwich panel with cubic packing for 0.8 mm (a) and 1.5 mm (b) adhesive thickness.
The panels made with untreated aluminium sheets in a previous research [19] exhibited a single failure mode based on the complete delamination of the facing material from the bottle caps core. The lack of skin support led to the catastrophic failure of the sandwich panel by core cracking (Figure 15.a). In the present work, the use of mechanical abrasion and chemical treatment led to an enhanced skin-polymer adhesion. A progressive failure with localised damage was observed (see Figure 15.b), instead of a fast and overall failure, which makes this material suitable for potential crashworthy applications. This conclusion is also evidenced by the comparison between the load-deflection plots for samples with and without aluminium treatment in Figure 16, the latter produced in the previous research [19]. The comparison was performed between samples of similar characteristics, namely 1.5mm adhesive thickness and cubic packing. A progressive failure was identified in the sample with treated aluminium surface, while a sudden drop was found in untreated aluminium samples corresponding to external skin delamination under tensile stress.

Figure 15. Differences of failure mode with (a) no surface treatment, leading to delamination [19] and (b) chemical and mechanical treatment, leading to local cap debonding.

Figure 16. Load-displacement plot of sandwich panels with and without aluminium surface treatment.
3.6. Applicability

Table 5 shows a comparison between C3 sandwich panels, which presented the best mechanical properties, and the PP panels investigated by Cabrera, Alcock and Pejis [18]. It is observed a notable increase in the most mechanical properties achieved by C3 bottle cap panels. Despite their greater bulk densities, bottle cap panels presented higher specific shear and skin stresses (in bold), respectively, 12% and 78% relative to PP composites (see Table 5). These results highlight the applicability of the proposed composites for lightweight secondary structural applications in engineering.

Table 5. Comparison between the proposed sandwich composite and other panels with circular cell honeycomb produced in [18]

<table>
<thead>
<tr>
<th>Response</th>
<th>C3 sandwich panel</th>
<th>PP composite [18]</th>
<th>Percentage increase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive thickness (mm)</td>
<td>1.5</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Core Bulk Density (g/cm³)</td>
<td>0.25 (0.01)</td>
<td>0.120</td>
<td>100%</td>
</tr>
<tr>
<td>Composite Bulk density (g/cm³)</td>
<td>0.59 (0.01)</td>
<td>0.195</td>
<td>202%</td>
</tr>
<tr>
<td>Maximum load (N)</td>
<td>2111 (73)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Shear stress (MPa)</td>
<td>0.86 (0.03)</td>
<td>0.27</td>
<td>218%</td>
</tr>
<tr>
<td><strong>Specific Shear Stress (N·m/g)</strong></td>
<td><strong>1.46 (0.05)</strong></td>
<td><strong>1.3</strong></td>
<td><strong>12%</strong></td>
</tr>
<tr>
<td>Skin Stress (MPa)</td>
<td>129.2 (5.0)</td>
<td>24</td>
<td>438%</td>
</tr>
<tr>
<td><strong>Specific Skin stress (N·m/g)</strong></td>
<td><strong>219.3 (6.9)</strong></td>
<td><strong>123.08</strong></td>
<td><strong>78%</strong></td>
</tr>
<tr>
<td>Shear core modulus (MPa)</td>
<td>30 (3)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Specific shear modulus (N·m/g)</td>
<td>0.06 (0.005)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Flexural Modulus (GPa)</td>
<td>2.83 (0.24)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Specific flexural modulus (N·m/g)</td>
<td>4.81 (0.38)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Flexural strength (MPa)</td>
<td>27.2 (1.4)</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>Specific flexural strength (N·m/g)</td>
<td>0.046 (0.001)</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

4. CONCLUSIONS

A class of sandwich structures made from aluminium skins and cores constituted by recycled PP bottle caps has been here evaluated from a Design of Experiments perspective. The main conclusions from the present work are:

i. The use of an epoxy polymer adhesive resulted in overall superior mechanical performances when compared with the polyester case, being more evident for the thicker adhesive layer (55%
higher in both the absolute and the specific properties). Core shear and flexural absolute moduli presented similar trends, whereas the specific core shear modulus had divergent results depending on the packing system used.

ii. The thicker adhesive layer provided superior mechanical performance for all the mechanical responses considered, with increments between 55.4 and 92% to absolute strength and stiffness, while specific properties increased from 25.3 to 52%. Higher strength and stiffness for the thicker adhesive layer was attributed to an improved contact area between the adhesive and caps, as well as more uniform adhesive fillets near the cap walls, which leads to more efficient load transfer between skins and core.

iii. The different packing systems did not significantly affect the overall mechanical responses. The packing system only affected the behaviour of the absolute and specific core shear moduli. The remaining properties did not present any significant changes due to cell packing. The caps present a very restricted contact area with adjacent cells in hexagonal packing, and therefore the reported strengthening of the core reported by other authors was not present.

The present work showed the feasibility of sandwich panel concept from disposed bottle caps. The eco-core sandwich panel is promising, with its use of a sustainable lightweight honeycomb core based on a recycled and low-cost material.

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