

Valorization of pistachio shells as green composite material: an approach that contributes to sustainable development

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Abstract: The increase in the production of conventional plastics has generated an environmental crisis that is difficult to reverse. In response to this problem, this study proposes the development of a green composite material (GCM) from pistachio shells and a matrix based on potato starch, calcium chloride (CaCl₂) and polyvinyl alcohol (PVA). A series of steps were followed to prepare the GCM, including cleaning, drying, milling, sieving, mixing, compression molding and drying. Once the GCM was obtained, a perceptual analysis was performed and its microstructure and hardness were characterized by optical microscopy and Vickers tests, respectively. The granulometric analysis revealed that the milled sample had a high proportion of coarse particles with sizes ranging from 0.71 to 1.70 mm, which accounted for 74.98%. These particles exhibited a slightly elongated complex morphology (circularity = 0.73 ± 0.04 and aspect ratio = 1.27 ± 0.16). The GCM exhibited a compact structure with a low degree of porosity and a visual finish similar to that of wood chipboard. The perceptual analysis carried out with experts and non-experts, showed a good acceptance of the material, highlighting its innovation, stiffness, and sustainability as the most outstanding attributes. In the microstructural analysis, a heterogeneous distribution of the particles was observed, as well as the presence of irregular pores. Finally, Vickers hardness tests showed that the GCM exhibited a hardness value of 33.63 ± 16.49 HV, which is comparable to that of other biocomposite materials. In conclusion, it was possible to produce a GCM from pistachio shell waste with adequate physical characteristics, which represents an attractive and viable sustainable alternative to replace conventional plastics and offers potential multiple applications in various industrial sectors.

Keywords: biocomposite material, pistachio shells, agro-industrial waste, sustainable material

Introduction

Human progress depends on the transformation of natural resources; however, this has led to the use of materials that do not return to natural cycles, causing serious damage to ecosystems. In particular, dependence on petroleum-based materials, such as plastics, has created an environmental crisis that is difficult to reverse. To satisfy growing demand, plastic production has increased significantly in recent decades and is estimated to reach 1.8 billion tons by 2050 (Taib *et al.*, 2023). Against this scenario, green composite materials (GCM) have emerged as an innovative and sustainable alternative. These are materials that incorporate a matrix and a filler, at least one of which comes from biological and sustainable sources (Rafiee *et al.*, 2021). Their development seeks to improve mechanical properties while reducing environmental impact, positioning them as a viable option compared to conventional materials, particularly petroleum-based plastics. On the other hand, the generation of agro-industrial waste is a global environmental problem, with approximately 140 billion tons per year (Balasundar *et al.*, 2019). This waste can be valorized instead of being discarded or incinerated, which would reduce the demand for natural resources. In this context, nut waste (shells) represents an interesting option for the development of green composite materials, with pistachios (*Pistacia vera* L.) being particularly important due to their abundance. Globally, 1,026,802 tons of pistachios are produced annually, of which 35-40% corresponds to shells, equivalent to approximately 389,180 metric tons (McNeill *et al.*, 2024).

The pistachio fruit is an almost oval-shaped drupe. It consists of a single seed (kernel), covered by a thin edible layer (testa or skin), enclosed by a hard, smooth, inedible shell (endocarp) (Kashaninejad & Tabil, 2011). Figure 1a shows an image of a pistachio with its main parts. An important aspect of pistachio shells is their chemical composition, which consists of 47.08% cellulose, 26.56% hemicellulose, 13.74% lignin, 7.52% moisture, 0.92% waxes, and 4.18% ash (Balasundar *et al.*, 2019). The high cellulose content in pistachio shells is higher than that reported for other nut shells, such as hazelnuts, almonds, and pecans, among others (Queirós *et al.*, 2020). This suggests that the mechanical and thermal properties of a composite material made from pistachio shells may be superior to those made from other shells (Arzumanova, 2021). Recent research has identified that pistachio shells are mainly composed of cells called puzzle-shaped polylobular sclereids, which have a three-dimensional (3D) structure, as shown in Figures 1b-c (Huss *et al.*,

2020; Xiao *et al.*, 2021). Figure 1b shows an optical microscopy image of pistachio shell tissue, highlighting the very compact arrangement of the sclereids. Similarly, Figure 1c shows a confocal laser scanning microscopy (CLSM) image where the polylobular sclereids with a puzzle-like morphology can be clearly seen, exhibiting a high topological cross-linking. Additionally, it can be observed that cellulose (green coloration) is abundantly distributed throughout all cells, and that lignin (red coloration) is mainly concentrated in the composite middle lamella. Thanks to these properties, pistachio shells are a viable and attractive alternative for use as a component in the manufacture of GCM. Therefore, this study aims to develop a green composite material from pistachio shells and an ecological binder, such as potato starch, in order to create a material suitable for the manufacture of objects that reduce or replace the use of conventional plastics.

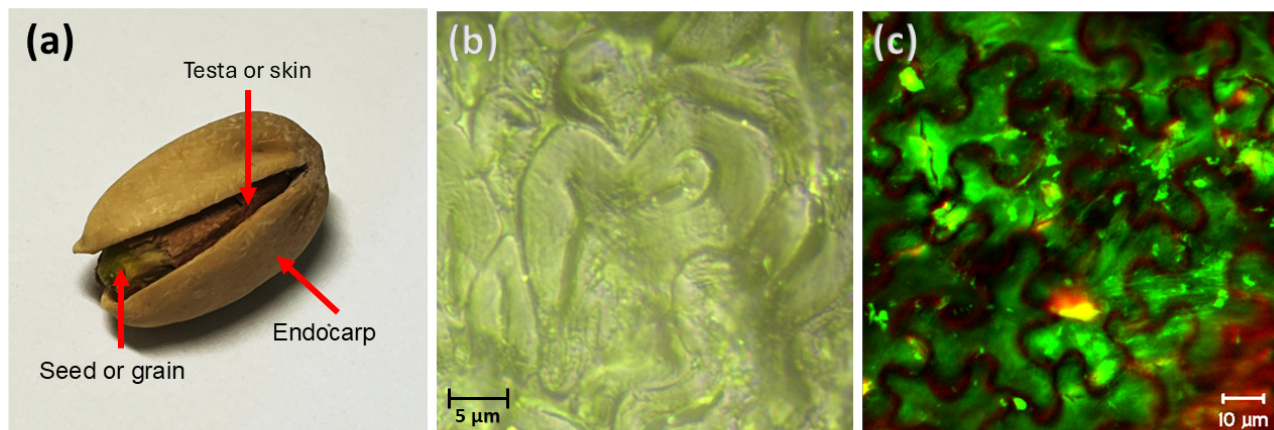


Figure 1. Morphology and microstructure of the pistachio shell: (a) image of a complete pistachio nut showing its main parts, (b) optical microscopy image showing the compact structure of the shell, and (c) MCBL image showing the topological cross-linking of 3D polylobular cells and their chemical composition (green corresponds to cellulose and red to lignin)

Materials and Methods

Raw materials and reagents

Whole pistachios (*Pistacia vera* L.) from the Wonderful brand (USA) were purchased at a supermarket in Mexico City. Potato starch (03967-500G), calcium chloride (C1016-500G), and polyvinyl alcohol (341584-1KG) were supplied by Sigma-Aldrich (USA).

Harvesting, conditioning, and milling of pistachio shells

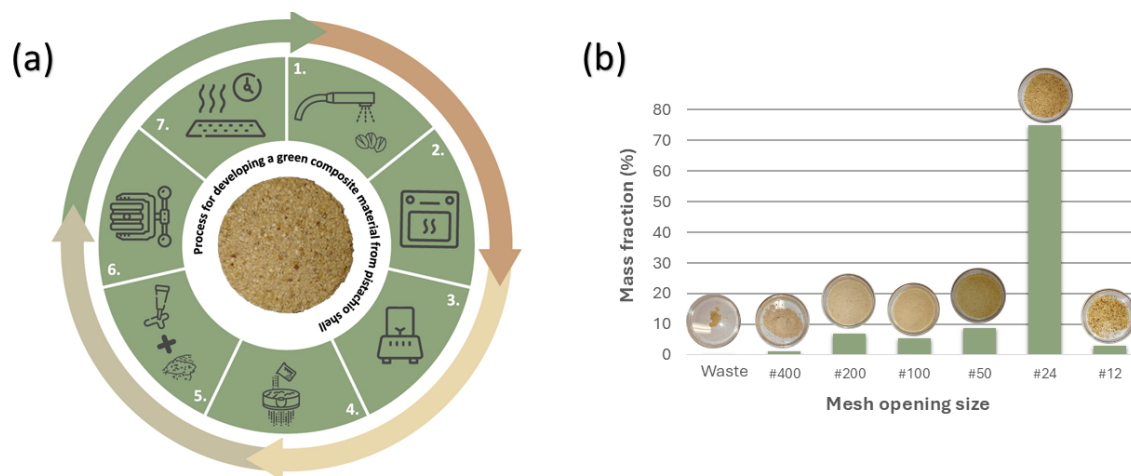


Figure 2. (a) Diagram of the process for producing a GCM from pistachio shells: (1) cleaning, (2) drying, (3) grinding, (4) sieving, (5) mixing, (6) compression molding, and (7) demolding and drying. (b) Histogram of the particle size distribution of the pistachio shell sample milled for 2 minutes

Figure 2a shows a general diagram of the steps carried out to elaborate the GCM. First, the whole fruits were shelled manually to separate the shells from the seeds. The pistachio shells were washed with tap water and dried in an oven at 40°C for 12 hours. They were then milled in a blade mill (GC-300, CGOLDENWALL, China) operating at a rotational speed of 28,000 rpm for a period of 2 minutes.

Particle size analysis

The particle size distribution of the milled pistachio shell sample was analyzed using a series of 8-inch diameter stainless steel sieves with different mesh sizes: #12, #24, #50, #100, #200, and #400 (WS Tyler, USA). The sample was shaken for 20 minutes in a vibrating sieve shaker. Subsequently, the fraction of retained mass (%) was calculated for each sieve, and a particle size distribution (PSD) histogram was generated. The average particle size (D_{mean}) and standard deviation (σ) were determined using Equations 1 and 2, respectively, where n_i and D_i correspond to the percentage of retained mass (%) and the average particle size retained in the sieve (μm), in that order.

$$D_{mean} = \frac{\sum n_i D_i}{\sum n_i} \quad (1)$$

$$\sigma = \left(\frac{\sum n_i (D_i - D_{mean})^2}{\sum n_i} \right)^{1/2} \quad (2)$$

Morphological analysis

The morphology of the particles in the most abundant mass fraction (sieve #24) was analyzed using image analysis techniques. Digital images of the particles were captured using an optical microscope (BX51, Olympus, Japan) with a magnification of 5x. The images were then processed with ImageJ v. 1.45s software (<https://ij.ijmjoy.io/>) to determine some morphological parameters such as circularity, Feret diameter, and aspect ratio (AR), following the methodology proposed by Nicolás-Bermúdez *et al.* (2018). The parameter values were expressed as mean \pm standard deviation.

Preparation of the matrix (binder)

After several experimental trials, the optimal proportions of the polymer matrix were determined: 20% (w/w) potato starch, 16.5% (w/w) calcium chloride (CaCl_2), and 2% (w/w) polyvinyl alcohol. Initially, the calcium chloride was dissolved in water using a magnetic stirrer for 2 minutes. Subsequently, the potato starch and PVA were added, and the solution was heated to 75°C for 50 minutes under constant stirring until the starch gelatinization was achieved.

Elaboration of the GCM (mixing, pressing, and drying)

To elaborate the composite material, the milled sample of the shells was added to the binder in a ratio of 60% (w) shells / 40% (w) polymer matrix. The components were mixed manually until all the pistachio particles were incorporated into the matrix, forming a homogeneous mass. The resulting mass was poured into molds of different geometries: a 5 cm diameter cylinder, a silicone unicorn mold, and a 12 x 12 cm² rectangular mold with abstract shapes printed in 3D. The counterpart of each mold was placed in position, and the samples were compacted with a manual press for 15 minutes. Finally, the counterpart was removed, and the samples were demolded and dried at room temperature for approximately 36 hours.

Physical characterization of GCM

Perceptual analysis

Once the GCM was obtained, a comprehensive description of the composite material was made in order to analyze its general appearance, homogeneity, and possible surface irregularities. To evaluate its potential application, a perceptual analysis was carried out with a sample of 30 people: 15 with experience in materials and 15 without experience in the area. The material was presented to the panelists in different shapes (cylinder, unicorn, and

rectangular panel). Participants rated various attributes on a scale from -3 to 3, where 3 represents the most desirable characteristic and -3 the least desirable.

Microstructural analysis of the GCM

The microstructure of the GCM was analyzed using optical microscopy and confocal laser scanning microscopy (CLSM). Cylindrical samples were used for both analyses. Several images at different magnifications were taken of different areas of the sample surface using an optical microscope (BX51, Olympus, Japan). The samples were also examined using a CLSM (LSM 710 NLO, Carl Zeiss, Germany), where the samples were excited with a 488 nm wavelength laser for lignin detection (detection range: 470-630 nm). Images at different magnifications were captured on the coarse particles and in the polymer matrix.

Vickers hardness test (HV)

Finally, the mechanical strength of the material was evaluated using the Vickers hardness test. The test was performed by applying a load of 1 kgf to the surface of the GCM (cylindrical samples) with a controlled descent of 0.1 kgf over 30 seconds. A total of 30 measurements were taken at different areas of the samples. After the tests were performed, the diagonals of the indentation marks were measured with an optical microscope, and the Vickers hardness HV was calculated using Equation 3.

$$HV = 1.854 \times \frac{F}{(d)^2} \quad (3)$$

Where HV is the Vickers hardness, F is the applied force (kgf), d is the average diagonal of the indentation mark (mm), and 1.854 is a conversion factor based on the geometry of the indenter. Hardness values were expressed as average \pm standard deviation.

Results and Discussion

Particle size analysis of the milled pistachio shell sample

Milling is an important pretreatment stage in the GCM manufacturing process. The main objective of milling is to reduce the initial size of the shells so that the particles can be properly mixed with the polymer matrix (binder). Figure 2b shows a histogram of the PSD of pistachio shells, which were milled with a blade mill for 2 minutes. As can be observed in the figure, most of the particles were retained in the 24-mesh sieve (74.98%), which corresponds to particle sizes of 0.71 to 1.7 mm. It was also observed that fine particles ($\leq 710 \mu\text{m}$) with a wide size dispersion were generated, which exhibited mass fraction values of less than 10%. The D_{mean} and σ values were 1038.75 μm and 462.94 μm , respectively. The high σ value is associated with the wide dispersion of fine particle sizes generated during milling. This fragmentation behavior, where mostly coarse particles are generated, has also been observed in pecan, almond, and pine nut shells (Queirós *et al.*, 2020). In particular, the mass fraction retained in sieve 24 reported in our study was significantly higher compared to the mass fraction retained (particle sizes of 1-2 mm) in pecan (36.5%), almond (42.4%), and pine nut (37.4%) shells. These results reveal that it is more difficult to reduce the initial size of pistachio shells due to their good mechanical properties. However, the fragmentation behavior of any material depends on multiple factors such as the type of mill used, operating conditions (e.g., grinding time), moisture and chemical composition of the material, microstructure of the material, etc. Likewise, the particle size of a milled sample of agro-industrial waste is a very important factor because it can strongly influence the process of manufacturing a GCM and its functional properties. In a study conducted by Nunes *et al.* (2024), the authors demonstrated that the properties of a rice-based biocomposite vary according to the particle size used, highlighting the importance of this variable in the development of sustainable materials.

Morphological analysis of particles retained in sieve 24

The morphology of the most abundant particles retained in sieve 24 was analyzed using image analysis techniques. The results showed that these particles exhibit complex morphologies with circularity and aspect ratio (AR) values of 0.73

± 0.04 and 1.27 ± 0.16 , respectively. Circularity is a parameter that measures the similarity of a particle to a circle, where a particle with a value of 1 has a perfect circle shape. Therefore, the pistachio particles analyzed do not approximate circular shapes. Conversely, the AR parameter is a measure of the degree of elongation of a particle. Based on the AR value obtained, the pistachio particles have slightly elongated shapes. Likewise, the Feret diameter value was $1146.66 \pm 127.42 \mu\text{m}$, which is very similar to the D_{mean} value ($1038.75 \mu\text{m}$). In addition to particle size, particle shape is another key factor that can influence the manufacturing process and mechanical properties of GCM. Specifically, the interaction between the polymer matrix and the particles, which is influenced by the size and shape of the particles, may exhibit regions or anchor points where there is greater affinity between the two elements. Both variables can also influence the compaction stage of the GCM and, consequently, its functional properties. The AR and the level of order (orientation) of the particles (fillers) in a composite material can drastically influence the properties and control the transfer of stresses throughout the material. Mariano *et al.* (2017) reported that composite materials that have fillers with high AR values and exhibit a high level of organization display excellent mechanical properties (e.g., high elastic modulus values).

Macroscopic evaluation of the GCM

Figure 3 shows images from different perspectives of a tablet of the GCM produced in this study. The macroscopic evaluation of the GCM made from pistachio shells was based on the expressive-sensory scale proposed by Sauerwein *et al.* (2017), which considers key tactile and visual attributes to describe the material experience. According to this scale, the GCM can be characterized as a matte-finish material with gradual coloring and a light beige tone (Figure 3). To the touch, it has a predominantly rough, dry, and hard texture with a slightly irregular surface, reflecting the nature of the reinforcement (pistachio shells) used. In terms of perceived temperature, the material was classified as neutral to slightly cold, which coincides with its low porosity and compact structure. No moisture was detected, so it was also considered non-sticky and non-damp. In addition, the surface showed no deformation or displacement when handled, so it was perceived as non-flexible. The material also showed excellent cohesion between its components and no shell particles detached when handled. Finally, the material is perceived as lightweight.

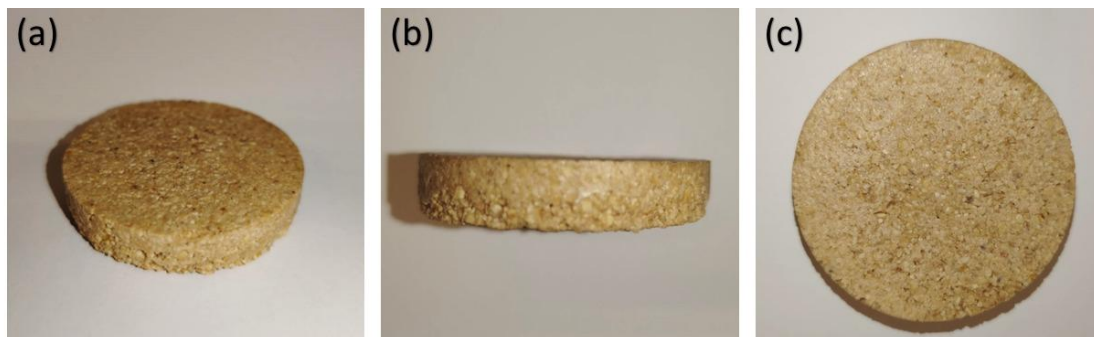


Figure 3. Images of a GCM tablet made from pistachio shell waste: (a) isometric perspective, (b) side view, and (c) top view

Perceptual analysis of GCM based on pistachio shells

The results of the perceptual analysis are displayed in Figure 4, which demonstrates that the material was perceived as natural, organic, and innovative. At the same time, it was considered opaque, rough, complex, and expensive. Among the main comments made by the participants, the following were highlighted: its lightness, resistance, and appearance similar to marble or MDF (chipboard). This type of response coincides with the materials classified within group 4 (Bimodal) of the study conducted by Thundathil *et al.* (2023), which stand out for evoking both visual and tactile sensations. However, the material was also perceived as coarse or rough, suggesting an affinity with the materials in group 3 (Dominantly Tactile, Rough/Natural), characterized by pronounced textures and a less refined appearance. Although this work does not formally address the machinability of GCM, it has been found that when the material is subjected to sanding processes, its surface roughness decreases. In this condition, the material could be associated with group 1 (Tactile dominant, smooth/textile), which includes materials perceived as smooth. In addition to these attributes, the perception of the material's complexity was reflected in a value of -1 on the bipolar scale between simple and complex, indicating a tendency toward complexity. According to Thundathil *et al.* (2022), complexity correlates positively with attributes such as interesting and unusual and negatively with boring, ordinary, and smooth. It has been

documented that greater complexity favors the perception of novelty, so this assessment may contribute positively to the acceptance of the material. On the other hand, GCM was also perceived as moderately expensive, with a score of 0.5. This assessment may be related to the general public's perception of sustainable materials. As Carus *et al.* (2014) point out, biopolymer-based products can cost up to three times more than conventional plastics due to factors such as economies of scale and technical limitations for mass production. Although this perception of higher cost is not always based on technical knowledge of the process, participants are likely to associate the natural aesthetics and novelty of the material with a more exclusive or limited-edition product, which may influence their judgment of economic value (Manu *et al.*, 2022).

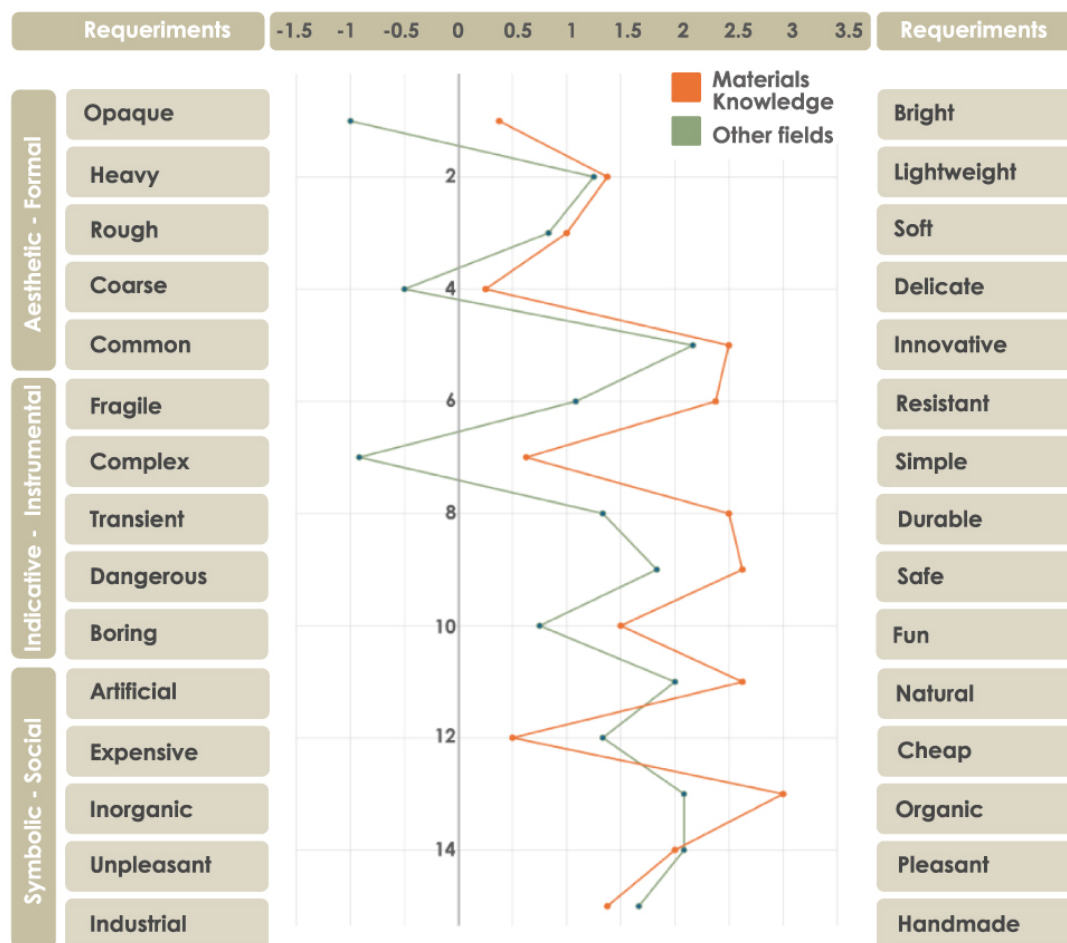


Figure 4. Results of perceptual analysis performed on the GCM, where different attributes were evaluated

Microstructural analysis

The surface microstructure of the GCM was analyzed using optical microscopy and CLSM (Figure 5). Both techniques revealed that the material consists of coarse particles (sieve 24), binder with fine particles, and interfacial regions between the particles and the binder (Figure 5 a-b). CLSM clearly showed the fine particles distributed over the binder (Figure 5b). The analyses also revealed the presence of some irregularly shaped pores smaller than 1 mm (Figure 5c). These pores were randomly distributed over the surface of the material. The presence of pores could be associated with the poor distribution of the polymer matrix over the particles and the compaction stage. This coincides with observations made in composites with a high walnut shell filler content (20% w/w), where rough surfaces, voids, and cracks have been reported due to heterogeneous distribution of the reinforcement (filler) (Lala *et al.*, 2018). These irregularities may also influence the biodegradability of the material, as it has been observed that fibers larger than 500 μm generate rougher surfaces, which favors water absorption and microbial attack, accelerating the degradation of the material (Nunes *et al.*, 2024).

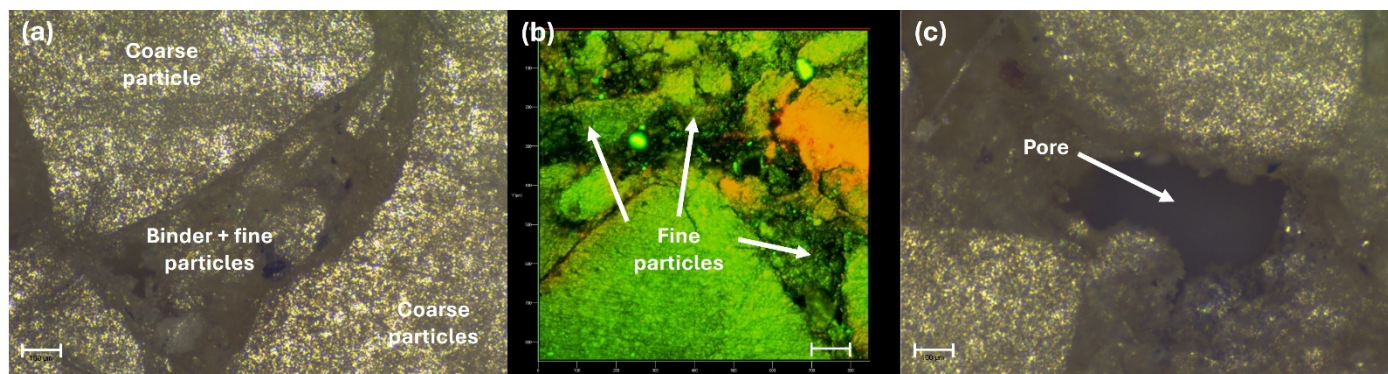


Figure 5. Images of the surface microstructure of GCM obtained using optical microscopy (a and c) and CLSM (b). For the MCBL images, the autofluorescence of the lignin present in the pistachio shell particles was used. For all images, the scale bar represents 100 μm

Vickers hardness test (HV)

The GCM obtained from pistachio shells had an average Vickers hardness value of 33.63 ± 16.49 HV. The standard deviation showed a high value, which can be attributed to the heterogeneity of the sample, which, as mentioned above, has different phases: coarse particles, binder plus fine particles, and interphase (particle-binder). In a recently published study, Yu *et al.* (2025) developed materials reinforced with bamboo and copper. The authors mechanically characterized their material and reported that oven-dried material had higher hardness (38.4 HV) compared to air-dried material (29.1 HV). This is attributed to the removal of moisture, which negatively affects the formation of the material. These results are very similar to those reported for our pistachio shell-based material.

Conclusions

Using the methodology proposed in this study, a green composite material was successfully developed from pistachio shell waste and a binder based on potato starch, calcium chloride, and PVA. The GCM exhibited good cohesion between its components and a compact, homogeneous structure. In general, the material was well received by participants in the perceptual analysis, who highlighted its innovation and perceived mechanical robustness. Microstructural analysis showed that the material consists of different phases: coarse particles, binder with fine particles, and interfacial regions (pistachio particles/binder). Mechanical testing revealed that the GCM has a Vickers hardness value comparable to that of other composite materials. In addition, possible applications of the material were explored through the design and manufacture of ornamental and functional prototypes. Figure 6 shows two representative examples: (a) a piece with a decorative unicorn design and (b) a panel with abstract shapes. The results obtained in this study suggest that this GCM represents a sustainable and viable alternative for the development of products that reduce the use of conventional plastics, while promoting the valorization of agro-industrial waste such as pistachio shells.

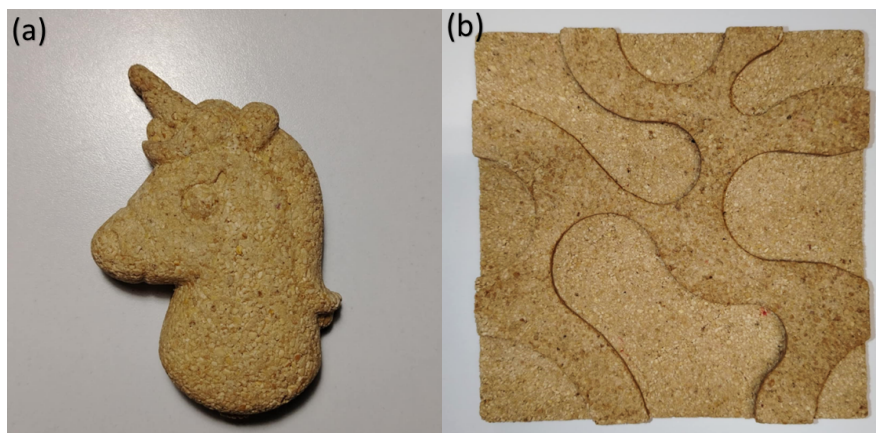


Figure 6. Examples of applications of GCM based on pistachio shells: (a) a piece with an ornamental design in the shape of a unicorn and (b) a panel with abstract shapes inspired by the cellular structure of pistachio shells

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