

Article

Thermal, Physical and Mechanical Performance of Orange Peel Boards: A New Recycled Material for Building Application

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Abstract: More than 124 million tons of oranges are consumed in the world annually. Transformation of orange fruit generates a huge quantity of waste, largely composed of peels. Some attempts to reuse by-products derived from citrus waste have been proposed for energy production, nutrient source or pharmaceutical, food and cosmetic industries. However, their use in the building sector had not been researched. In this study, orange peels, in five different ratios, from 100% of wet peels to 75% and from 0% of dry peels to 25%, were submitted to a thermo-compression procedure. They were evaluated according to their physical (bulk density, water absorption, thickness swelling, surface soundness and thermal conductivity) and mechanical properties (bending strength and modulus of elasticity). The results showed that orange peels can be used as thermal insulation material. The addition of dried peels makes the structure of the board heterogeneous and thus increases its porosity and causes the loss of strength. Hence, the board with the sole use of wet peel, whose thermal conductivity is 0.065 W/mK while flexural strength is 0.09 MPa, is recommended.

Keywords: orange peels; thermo-pressing; citrus board; bio-composite; thermal insulation; sustainable building



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1. Introduction

The construction sector represents one of the largest raw material consumers in the world with more than 42 billion tons of materials consumed in one year (WorldGBC2019). Furthermore, construction and demolition waste comprised about 25–30% of the total wastes in the EU (https://ec.europa.eu/environment/waste/construction_demolition.htm, accessed on 13 April 2021), with an estimated account of 180 million tons per year, which is more than 480 kg per person, per year [1]. It is especially noticeable in developed countries, and it became the largest in most developing ones [2]. A circular approach, in which the use of subproducts or wastes could be introduced into the construction chain, could help the building sector to reduce the environmental impact, as well as embodied energy [3].

In the search of circular economy, the use of agricultural by-products as a potential raw material for building applications has increased. Their low bulk density together with the porous structure make them suitable for this application. Based on this, different research projects have been developed in which the use of rice straw [4], rice husk [5], sugar [6], corn cob [7,8], pineapple leaves [9,10], peanut shell [11,12], coffee grounds [13], coffee chaff [14], coconut husk [15,16], sunflower [17], walnut shell [18], durian peel [19] and Opuntia [20], among others, were proposed, not only as thermal insulation material but also as acoustical absorbance [21]. Many of these products have not been commercialized on a large scale yet, but they are an alternative for the bioconstruction sector, with thermal values often comparable to commercial products. Indeed, specifically for their high sound absorption

coefficients, different researches with hemp shiv, sunflower bark and pith, flax shiv and rape straw showed their potential [22].

Orange is the most widely cultivated fruit worldwide, and it accounts for about 50–60% of total citrus production. In 2016, more than 124 million tons of citrus were produced. About 50–60% of these were consumed as fresh fruit, and the remaining 40–50% were destined for industrial processing [23]. An enormous amount of waste results from this production; considering that the 50% is composed of peels, the waste produced is about 3.4 million tons. Citrus processing waste has been subjected to different valorization studies, with the aim of searching alternative use of this by-product that can increase its demand on the market and its economic value. Its use must be highlighted in composting, animal food and organic fertilizers, but also in producing biogas as well as the extraction of pectin, bioactive and essential oils [24–26]. However, its use in the building sector has not been found in the literature despite its potential use as thermal insulation material following previous experiences on biopolymer aerogel [27–29] as well as its agglomerate properties based on the high pectin contents [30].

On the other hand, due to the high energy consumption of binders, in the recent years, different studies have been developed in which thermo-compression technique of manufacturing is used [31,32]. This technique is based on the compression of a panel with two hot plates and has been used in boards with lignocellulosic fibers [33], kenaf [34], sunflower [17] and bagasse [6], among others. In those cases, the sugar residues from bagasse, the resins expelled from the sunflower and the fibers act as a natural binder for the panels' production. This method has the advantage of avoiding the use of energy in the manufacture and the reduction of costs associated with it [3].

Hence, the aim of this research is to evaluate the potential use of the thermocompression technique in the manufacture of thermal insulation boards based on citrus fruit waste.

2. Materials and Methods

2.1. Characterization of Orange Peels

A part of orange cultivation is for the production of juices, for which the procedure is similar to the technique used in small-scale machines for coffee shops and supermarkets.

In order to guarantee an homogeneity in the wastes, peels were collected from the same place (a small scale center) twice per day, to guarantee that the peels were still fresh. Material was characterized each time, and a variation of the bulk density of the peels was found ranging from $900 \pm 20 \text{ kg/m}^3$. The percentage of by-products obtained from the small-scale production (supermarkets) is the same as the juice factories at the industrial level in terms of content percentages. When peels were introduced in a chamber at $40 \text{ }^\circ\text{C}$, the bulk density was $200\text{--}300 \text{ kg/m}^3$. This is in such a way that the water content varies from 90% for wet material to 8–10% for “dried” biomass.

Furthermore, wet orange wastes were composed by 60–65% of the weight of “peels”, 30–35% of pulp and the residue part of seed [35]. Furthermore, peels were composed of two layers (Figure 1 C,D) a compact external layer and an internal porous one. The former is commonly called “flavedo” and represents about 10% of the orange weight (Figure 1A); this is while the inner porous peel called “albedo” represents 17% of the citrus weight [36] (Figure 1B).

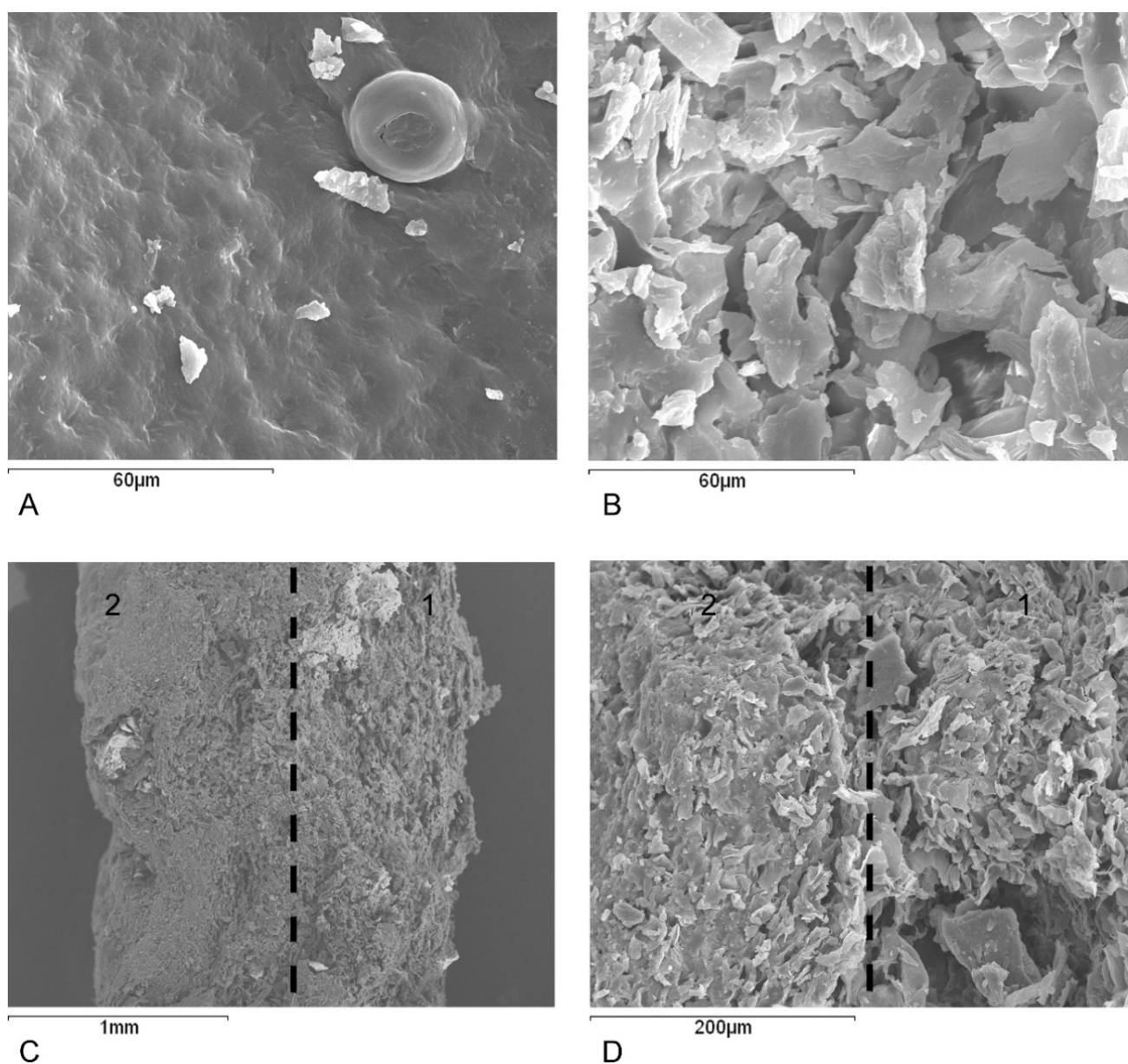


Figure 1. Scanning electron microscopy images of dried orange peel. (A) the external layer called flavedo; (B) the inner layer called albedo; (C,D) showed a transversal section of the peel, in which the right part is albedo and the left is flavedo.

The chemical composition is influenced by the external climatic condition, the cultivation method and the maturation and type of fruit. However, following the literature (Table 1), it is mainly composed of cellulose, pectin, sugar, acids, lipids, mineral elements, essential oil and vitamins [24,25,35,37,38] with a pH ranging from 3.5 to 5.8.

Table 1. Range of physico-chemical composition of CPW [24,25,35,37,38].

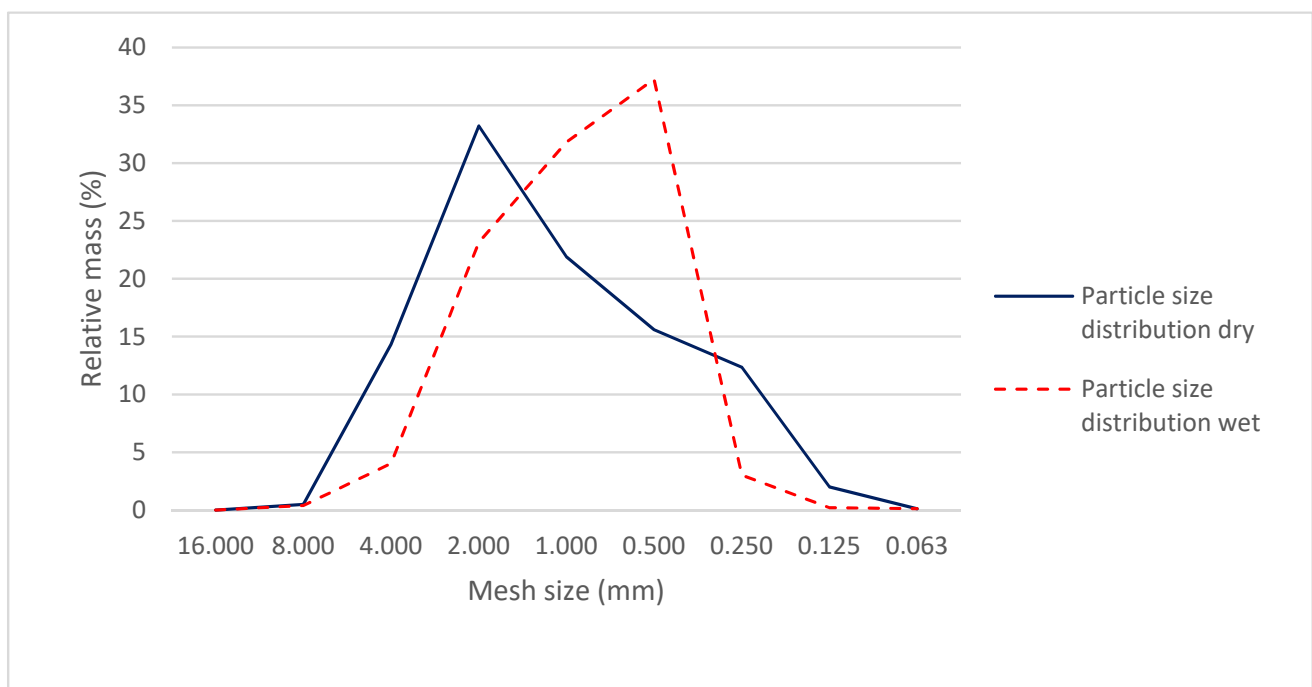
Parameter	Wet Citrus Waste [% Dry Matter]
Water content	72.5–87.0
Volatile solids	93.8–96.7
Protein	5.3–8.3
Fat	0.9–3.7
Fiber	10.6–42.1
Starch	1.0–2.9
Sugar	15.0–46.6
Calcium	0.7–0.8
Pectin	15.3–25.0

2.2. Procedure

2.2.1. Citrus Fruit Preparation

Wet and dry citrus wastes were used in the preparation of the boards.

Wet peels were shredded with a manual mill up to achieve a particle size between 0.5 mm and 10 mm with 80% of water (Figure 2). On the contrary, dry peels were obtained by placing the orange by-product into the ventilated chamber at 40 ± 5 °C until the masses were constant. The constant mass was defined as three subsequent measurements with a difference of less than 1%. An electronic balance with a precision of 0.01 g was utilized. Subsequently, the dried material was crushing with a manual mill and was sieved to divide the granules types.

**Figure 2.** Particle size distribution of dried and wet orange peels.

In all the cases, particle sizes between 4 mm and 0.125 mm were used, whose granulometric curve is shown in Figure 2, which represents the highest percentages obtained from

the milling. Particles larger than 4 mm (Figure 3e) and lower than 0.125 mm (Figure 3a) were discarded.



Figure 3. Particle size distribution obtained from sieving (a) $s < 0.63$ mm; (b) $0.63 < s < 1.25$ mm; (c) $1,25 < s < 4.00$ mm; (d) $s > 4.00$ mm; (e) dried citrus peel.

Five types of samples with different ratios of dried and wet orange peels were formulated ranging from 100% wet to 75% wet (Table 2). Therefore, panels were obtained from a mixture of wet and dry material in different percentages. For labelling, the W letter refers to wetness and the number indicates the percentage of wet material introduced in the mixture.

Table 2. Compositions of orange peel panels.

	W100	W90	W85	W80	W75
Wet:Dry [%]	100:0	90:10	85:15	80:20	75:25
Wet:Dry [g]	2000:0	1800:200	1700:300	1600:400	1500:500

2.2.2. Manufacturing of Particulate Panels

The preliminary version without mold and springs provoked the deformation of the boards during the drying process. It was due to the high content of water released and the consequent formation of cracks and waves.

Citrus fruit panels were prepared using a manual press, designed for this research, in which the samples were compacted while drying. They consist of a waterproof wooden mold on the sides and two steel plates drilled above and below the sample (Figure 4). Two perforated plates were used to allow the evaporation of the water during the drying process in the oven. Moreover, on the top and bottom surfaces between the perforated

plates and the samples, two pieces of glass fiber sheets were placed to prevent the boards from sticking onto the hot plates during pressing. The pressure is kept constant by four springs placed on the sides.

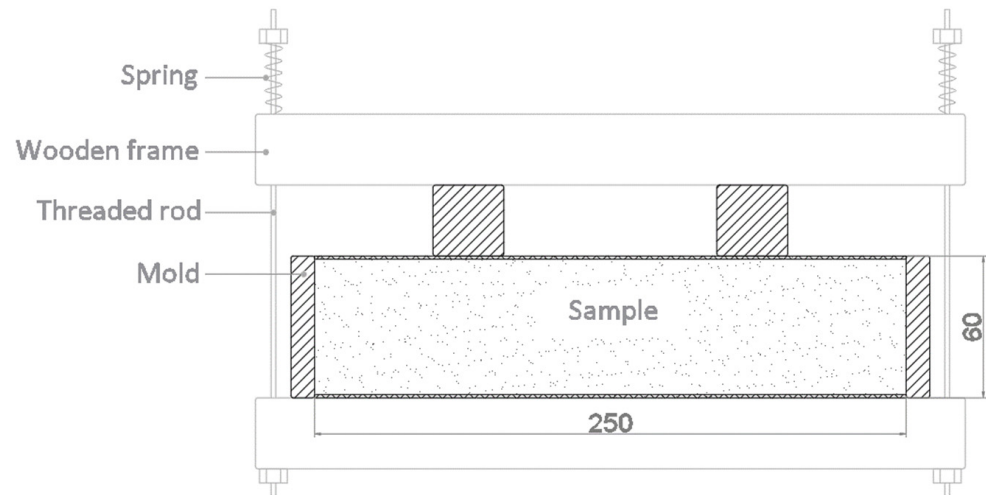


Figure 4. Drawing of the mold used.

The pressing and drying phase of the panel were in a ventilated dryer, at a temperature of 40 °C for 48 h.

A homogeneous mixture of dry and wet material was introduced in the mold. In all the cases, the same amount of weight was introduced in order to control the amount of material. All the samples were pressed using springs that were placed in the upper part of the press. The equivalent force applied to the panel was calculated based on the reduction of the length of the springs and is about 0.003 MPa. During the drying process, the springs were periodically tightened in order to apply to the sample a constant pressure. A view of the boards is shown in Figure 5.

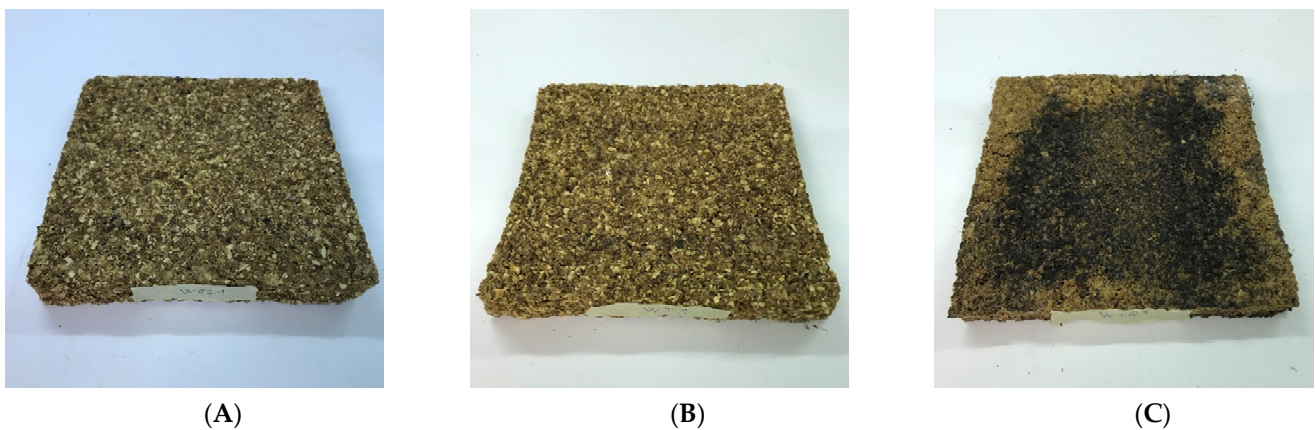


Figure 5. Orange peel panel W75 (A) W90 (B) and W 100 (C).

Eight samples of 250 × 250 mm were achieved; four of them were used for mechanical tests and four for hydric tests.

2.3. Characterization of Boards

2.3.1. General Properties

Shrinkage

During thermo-compression, the panel loses a large amount of water, with a consequent reduction in volume. Shrinkage was measured to assess the amount of volume reduction of the samples after the pressing and drying process. The formula used to calculate the restriction was the following:

$$\Delta V [\%] = \frac{(V_w - V_d) \times 100}{V_w}$$

where V_w is the volume of the panel before the pressing and dry process and V_d is the volume after the pressing and dry process. Testing was carried out on dry samples under laboratory conditions (21 ± 1 °C and $50 \pm 10\%$ relative humidity), and the results were the average of the eight samples of each blend. The measurement on the panel as the average of three measurements at the sides and center of the sample was obtained. A digital calibre VOGEL model 202112 with a resolution of 0.01 mm was used.

Bulk Density

Bulk density was determined as the relationship between the mass and the volume of the boards by direct measurement according to European Standard 1602 [39]. Samples were firstly balanced with the laboratory conditions (21 ± 1 °C and $50 \pm 10\%$ relative humidity). An electronic balance with a precision of 0.01 g and a digital calibre VOGEL model 202112 with a resolution of 0.01 mm were used. The results were calculated as the average of eight measurements of each blend.

2.3.2. Thermal Properties

A guarded hot plate apparatus, model HFM 436 Lambda from Netzsch company was used. The test was conducted according to the European Standard EN ISO 13787 [40]. The samples were placed between two heated plates at different temperatures. The temperature of the plates and the average temperature of the sample were defined by the user. The heat flux q through the sample was measured by a calibrated heat flux transducer. The measurement was performed once the thermal equilibrium was reached. Plate temperatures were controlled by two-way Peltier heating/cooling systems, integrated with a forced air heat exchanger that generates a closed-loop flow.

Samples of 250×250 mm with an average thickness of 20 mm were used. According to the standardized test technique [41] the samples were measured at 10 °C and 23 °C and a temperature gradient of 20 °C to evaluate the influence of temperature in the thermal conductivity. The measurements were carried out on the dry sample ($\mu = 0$) and after seven days of curing time under laboratory conditions (21 ± 1 °C and $50 \pm 10\%$ relative humidity).

2.3.3. Mechanical Properties

Mechanical characterization using a three-point bending strength test was performed. A Universal Test Machine was used, which provided values describing the force-displacement curve. The load cell was 5 kN with a displacement rate of 10 mm/min and a span of 100 mm. The maximum displacement value that could be registered was limited to 14.6 mm. According to the European Standard EN [42], values for Modulus of Rupture (MOR) by flexural stress can be determined using the following equation:

$$\sigma [N \text{ mm}^{-2}] = \frac{3 F_m L}{2 b d^2}$$

where F_m is the force applied (N), L is the distance between the supports (mm), b is the width of the sample (mm) and d is the thickness of the sample (mm). Due to the capacity of deformation of the samples, RILEM TFR1 [43] was used as a reference for the acquisition of load. According to this, the maximum load was considered when the displacement value was 10% of span support. The results were considered as the average of four measurements on specimen size of 250×250 mm.

In addition, the module of elasticity (MOE) at flexural strength was also calculated with the following equation:

$$MOE \left[N \text{ mm}^{-2} \right] = \frac{F L^3}{y 48 I}$$

where F is the force applied (N), L is the distance between the supports (mm), y is the strain of the sample (mm) and I is the moment of inertia (mm^4).

2.3.4. Hydric Properties

Water absorption by sorption test, according to the European Standard EN ISO 29767:2020 [44], was conducted. Samples were firstly dried and conditioned at 23°C and 50% of r.h. Then, they were placed in contact with distilled water on one surface and the change of weight at different intervals was measured: 5, 10, 20, 30, 90 and 360 min up to 24 h. The results were the average of four samples of each blend. The coefficient of sorptivity is calculated by:

$$Wp \left[g/\text{mm}^2 \right] = \frac{m_i - m_0}{A}$$

where m_i , in g, is the weight at different times; m_0 , in g, is the original weight of the dried sample; and A , in mm^2 , is the area of the sample in contact with water.

2.3.5. Microscopical Observations

A microstructural analysis on fractured samples was developed by a backscatter Scanning Electron Microscope (SEM) model JEOL JSM 6400. In this case, the samples were previously sputtered with a thin gold film.

This test was performed on W100 and W75 blends to evaluate the change of texture between the two blends with maximum and minimum dry peel content during blending.

3. Results and Discussion

3.1. Shrinkage

It was corrected in a second step, in which press with perforated plates and springs was added. The springs allow keeping the pressure constant even after the volume reduction and a planar panel were obtained. Significant differences were found in the water content of the blends, and, as expected, shrinkage decreased with the addition of dried orange peels. Consequently, as shown in Figure 6, the thickness from W90 to W75 panels increased linearly, although the maximum was observed in W100 (board with 100% of wet orange peels), with 70% of thickness loss.

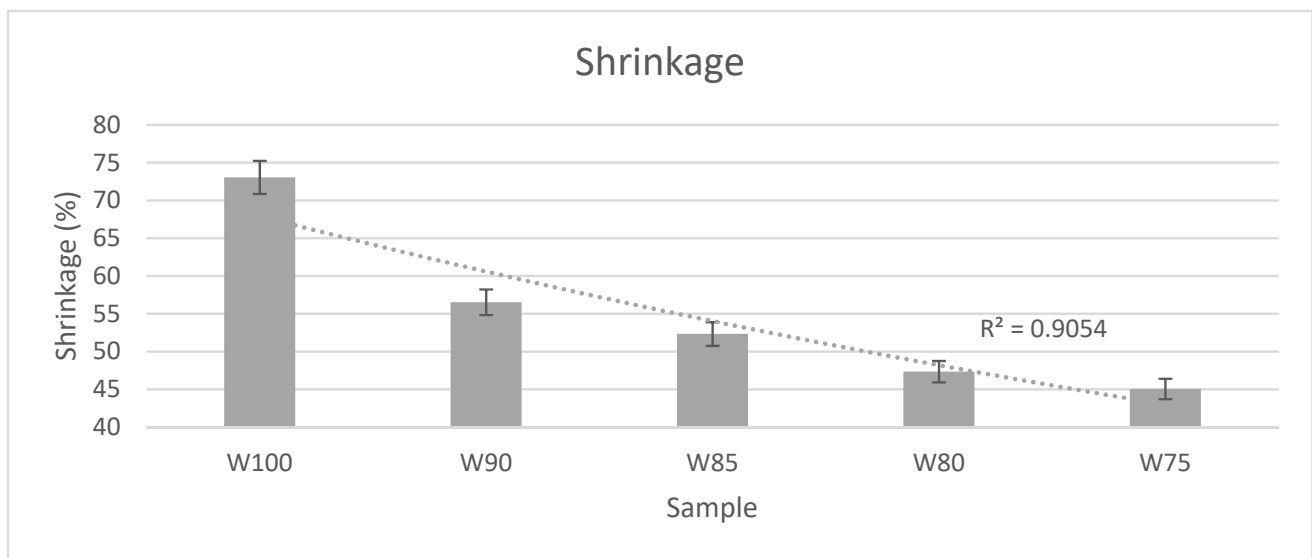


Figure 6. Shrinkage of composite panels after dry process.

3.2. Bulk Density

Bulk density varies between $468.63 \pm 12 \text{ kg/m}^3$ (W75) and $558.46 \pm 13 \text{ kg/m}^3$ (W100). Figure 7 shows that bulk density decreased as the number of dried peels increased. On the contrary, the lower bulk density was achieved with the maximum compacted W100 panel (only wet orange peels).

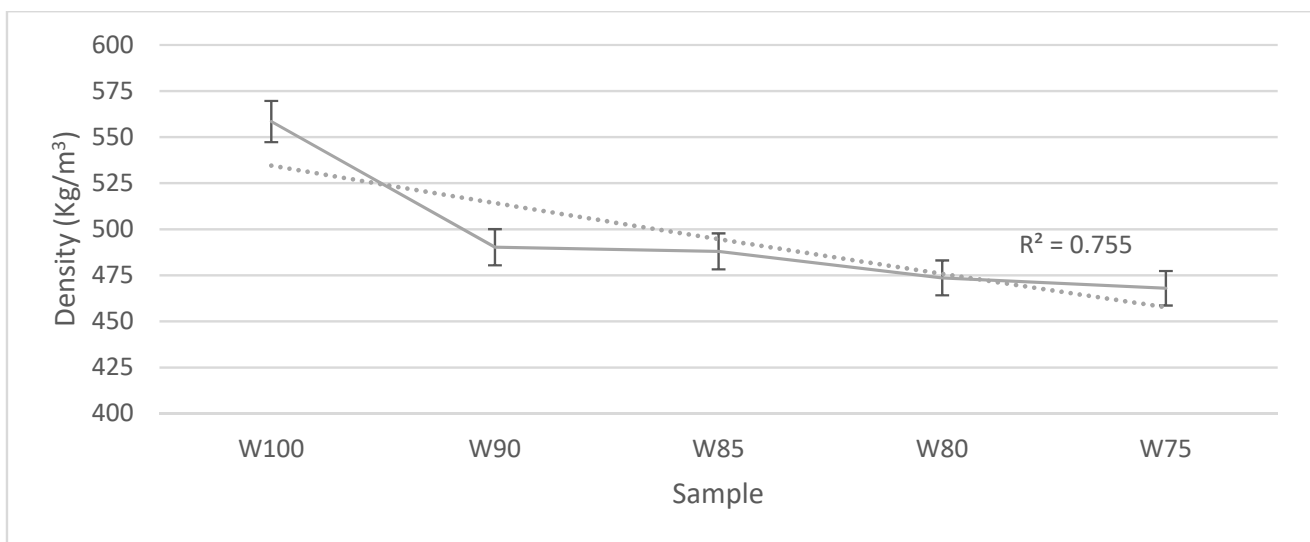


Figure 7. Density and thickness of composite panels after dry process.

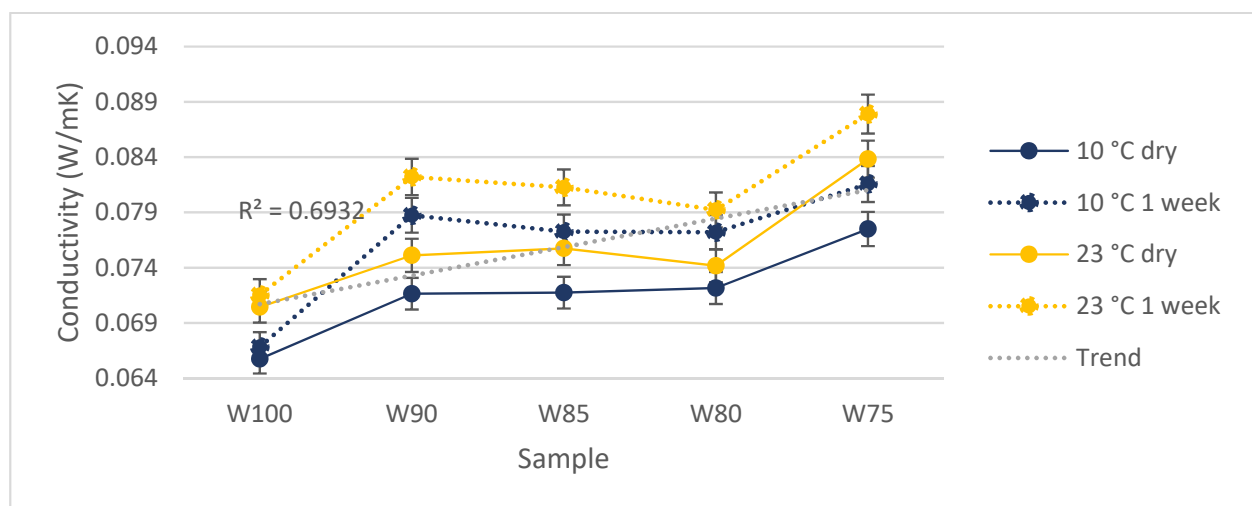
Since all of them were under 640 kg/m^3 , they can be graded according to ANSI as a low-density particleboard [45]. In addition, its average density of 509.19 kg/m^3 is similar to the values achieved in the literature with agricultural wastes such as corncob [7], walnut [46] and durian [19], as summarized in Table 3.

Table 3. Density of building panels made with agricultural by-products.

Material	Density [Kg/m ³]	Reference
Cork	108.7	[47]
Sunflower	500.0	[48]
Corn cob	413.0	[7]
Walnut	599.1	[46]
Sugar cane	686.0	[49]
Coconut	338.0	[19]
Pineapple	210.0	[9]
Durian	442.0	[19]
Rice husk granules	153.8	[46]
Rice straw	250.0	[4]
Opuntia	450.0	[20]

3.3. Thermal Properties

Contrary to the bulk density, W75 (sample with the higher amount of wet pulp during the manufacture) showed a higher thermal conductivity of the boards [50]. While the mean thermal conductivity of W100 was 0.066 W/mK, it increases 1.2 times to achieve 0.077 W/mK in W75 (Figure 8).

**Figure 8.** Thermal conductivity of orange peel panels calculated at 10 °C and 23 °C after dry process and after 1 week.

This performance can be justified by the type of microstructure achieved by the use of wet or dry peels on the composition, as would be observed in the microscopy. On the one hand, the wet material could provoke closed air pores, which showed higher thermal resistance than the open ones in the case of dry peels, as could be observed by microscopy. Furthermore, the replacement of water by air becomes higher in samples with a higher percentage of wet peels (W100). This hypothesis is in line with the increase in thermal conductivity when the samples absorbed moisture from the atmosphere [51], which is higher in samples with open porosity, contrary to W100, which almost keeps its thermal conductivity.

The differences must be highlighted between the results at 10 °C, which is the reference temperature for building materials, and those at 23 °C. As was expected [52], the higher the test temperature, the higher the thermal conductivity on a range of 4% to 12%. It is especially interesting in the case of hygroscopic materials such as this.

Compared with the literature, the samples from orange peel showed a higher thermal conductivity than other non-natural commercial insulations such as mineral wool, polystyrenes, and so on. However, the conductivity values obtained from orange peel

panels are in line with the conductivity of other agro-waste insulation materials such as durian [19] and ficus indica [20]. Furthermore, the conductivity is lower than the particle-board made with corn-cob [7], sunflower [48] and walnut shell [46], and greater than the particleboard made with coconut shell [19], pineapple leaves [9], sugarcane [49] and rice straw [4].

3.4. Mechanical Properties

Figure 9 showed that the incorporation of dried peels in the composition of the boards (W75) provoked a dramatic reduction of flexural strength. While W100 showed 0.09 MPa of flexural strength, it fell down to 0.02 MPa in W90 and almost kept constant in the others.



Figure 9. Flexural (a) strength and Modulus of elasticity (b) for the blends.

It can be explained by the homogeneity of the microstructure in the case of W100 compared to the blends in which dried peels were added. Furthermore, the reduction of the volume provoked in the samples with high wet peel content generates a microstructure that is denser and more continuous compared with the addition of dried peels. A discontinuity in the microstructure generated a loss of bond between the particles and a loss of strength. This performance is similar to the addition of aggregates in other studies, for example, cement with the addition of perlite [53] or plastic [54].

In any case, compared with the literature, strength is lower than other agro-waste boards with corncob [8], coconut coir [19] and sunflower [21], although similar to the bagasse ones [6]. However, the differences can be explained not only by the material itself but for the manufacturing process due to the higher compression strength in the preparation phase or even the presence of an external binder, contrary to the research.

Modulus of elasticity (MOE) followed the same pattern as the MOR (Figure 9b), and the stiffness is higher in the sample without dried peels (W100). Indeed, W100 showed a modulus of elasticity almost 10 times higher than the W75 with 34.79 N/mm² and 3.57 N/mm², respectively. This performance can be explained due to the formation of closed porous and homogeneity in the mixture compared to the others, observed by microscopy. In addition, Maillard reactions, caramelization, or pyrolysis of organic material [55] were expected to take place due to the brown color of the surface. The Maillard reactions from the decomposition of the sugar and amino acid could lead to interaction bonds and improve mechanical properties [56]. Indeed, as can be observed in Figure 10, the curve stress-strain showed a higher stiffness of samples W100, which is drastically reduced in the W90. It must be highlighted that the performance of W75 shows an increase in strength and could be justified by the balance between dried and wet peels in the case of W75, for which an interesting increase in strength is found.

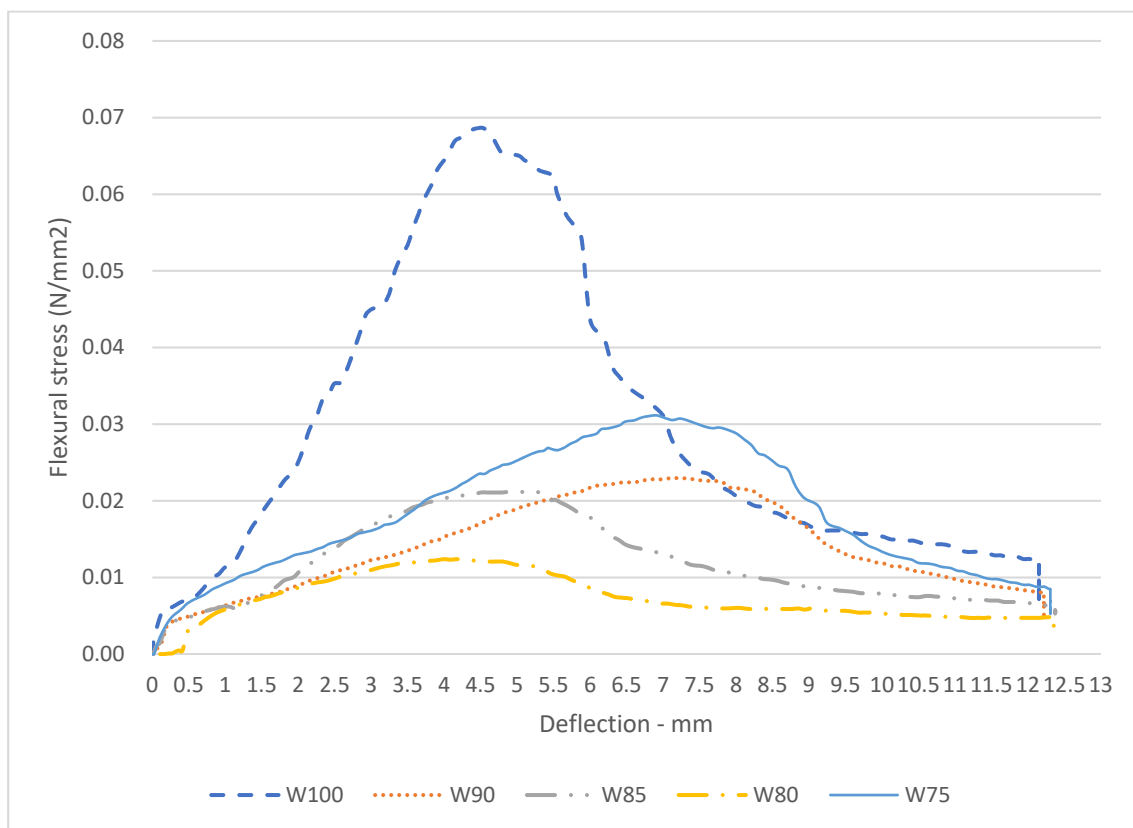


Figure 10. Flexural stress–deflection curve of orange peel samples.

3.5. Water Absorption

Once again, the lowest absorption by capillarity in W100 samples was observed while the highest in W80 samples was shown (Figure 11). It justified the hypothesis about differences in porosity and pore size among blends, as well as the higher open porosity in samples W80, compared with W100. At the same time, a correlation between porosity and strength was found and could be ratified by microstructural observations in which a highest porosity was observed in the case of W75, compared with W100. The greater absorption of the W80 sample compared with the W75 can be justified by a greater distribution of the pores and therefore by a release of the water in the dripping phase before measurement.

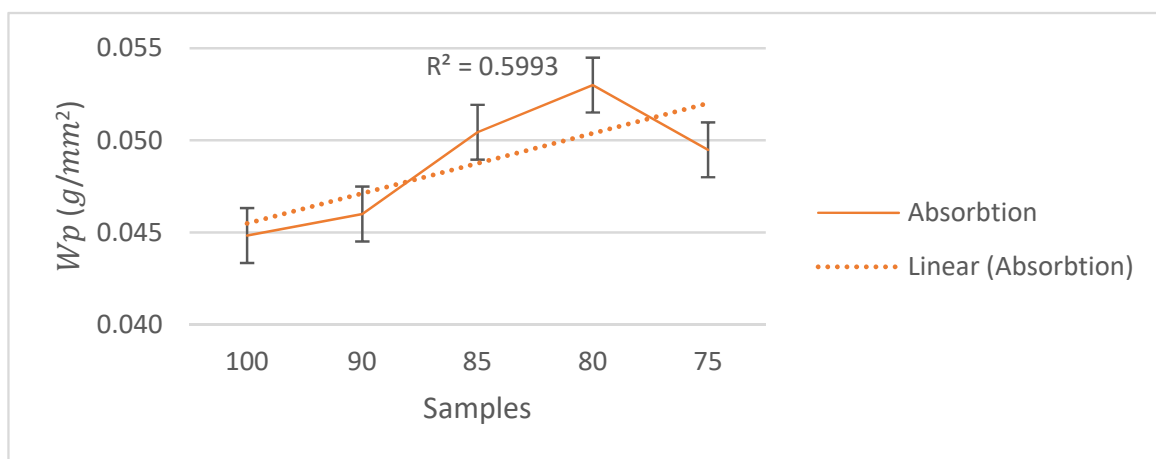


Figure 11. Water absorption of orange peel panels after 24 h.

Furthermore, the loss of cohesion in samples with dried peels is observed when they are submitted to water absorption. Indeed, the higher amount of dried peels, the higher the crumbled and lost particles during the tests. This performance is related to the shrinkage of the boards in contact with water, and an exponential linear increase of up to 40% of the swelling was observed in samples with higher dried peels (Figure 12). The homogeneity in the structure in the case of W100 led to all the particles to absorb water in the same way; on the contrary, the W75, with particles with a different procedure, absorb differently and provoked an increase in the swelling together with the heterogeneity in the structure and the higher open porosity detected.

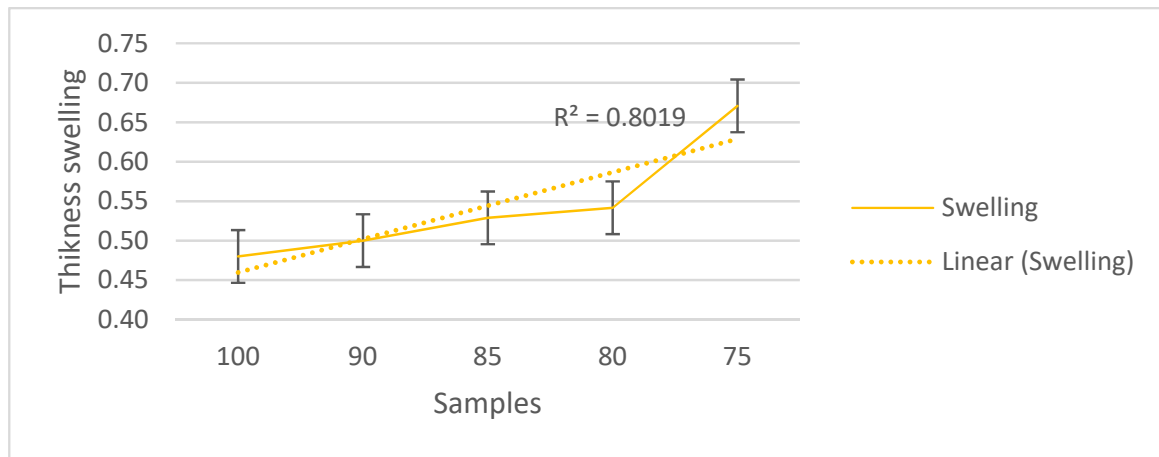


Figure 12. Thickness swelling of orange peel panels after 24 h.

3.6. Microstructural Observations

Differences in the microporous structure between W100 and W75 corroborated the mechanical and hydric results (Figure 13). The higher amount of dried peels provoked a porous structure in line with the hydric and mechanical performance, while a compact structure was observed in the W100. It can be explained by the better attachment between orange particles due to the pectin, the sugar and the homogeneity in the structure, compared with the addition of other particles. The samples showed chromatic variation, ranging from orange to brown (W100). The browning of the material is most likely caused by Maillard reactions involving sugars and proteins present in the orange peels [55]. Alternatively, it may be caused by the caramelization of plant sugars or by pyrolysis of organic material [57,58].

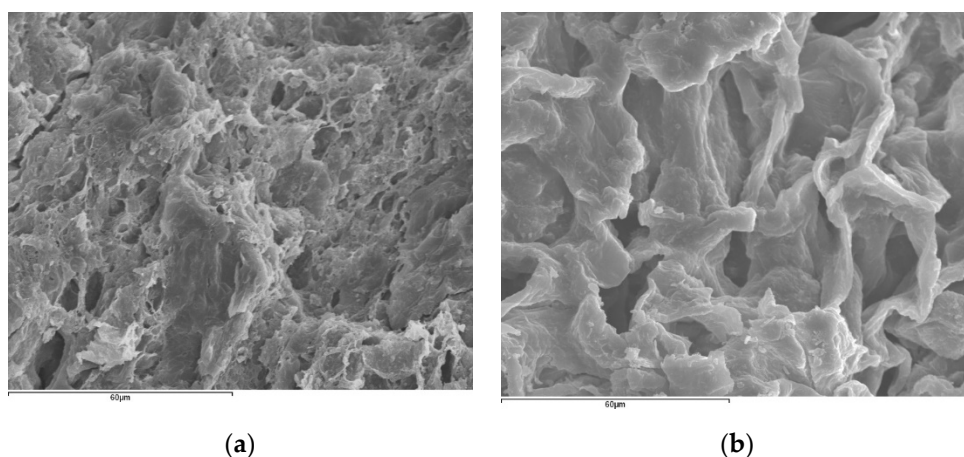


Figure 13. Scanning electron microscopy image of W100 panel with lower porous size and more compactness in the texture (a) and W75 panel with higher porosity (b).

4. Conclusions

A new building material composed of orange peels was analyzed from the point of view of its availability as a thermal insulation board. In this research, the influence of the procedure method was evaluated by the use of two types of particles, wet and dry peels. By the variation of those, five types of mixture were analyzed by the combination of different percentages of wet and dried orange peels ranging from 100:0 (wet:dried), called W100, to 75:25, called W75.

The results showed that it is possible to produce orange peel particleboards without chemical addition. The use of wet or dry particles notably influenced the performance of the boards. The highest thermal conductivity was achieved by W75 for which the flexural strength was 63% lower than W100. However, the optimum composition for this application is found in the board W100 (100% of wet orange peels) for which the thermal conductivity is 0.065 W/mK. Furthermore, their performance in terms of mechanical and physical tests is the most suitable for the application. In this case, the caramelization of the sample induced higher compactness and homogeneity in the structure [59,60].

In spite of the interest of the boards, the main drawback could be the durability, so long-term tests are required to evaluate the degradation of the samples.

5. Patents

Patent with application No. 10202000024475 is pending.

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Conflicts of Interest: The authors declare no conflict of interest.

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