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TEMA: Technologies Engineering Materials Architecture**Vol. 8, No. 2 (2022)**

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Editorial**5****Research perspectives in the domain of the built environment***Riccardo Gulli*

DOI: 10.30682/tema0802l

CONSTRUCTION HISTORY AND PRESERVATION**Proposal for a new housing model for the inland areas regeneration. The BioVillage 4.0****7***Emanuela D'Andria, Pierfrancesco Fiore, Enrico Sicignano*

DOI: 10.30682/tema0802a

Brick masonry staircases of the early 20th century: historical research, condition assessment and diagnostic investigation of a “transition” construction type**16***Mariella De Fino, Fabio Fatiguso*

DOI: 10.30682/tema0802b

Technological analysis of a prefabricated timber-based system for the integrated renovation of RC framed buildings**30***Carola Tardo, Giuseppe Margani*

DOI: 10.30682/tema0802f

The marble envelope of the *Casa delle Armi* by Luigi Moretti: documentary and experimental knowledge finalized to digital modeling**44***Marco Ferrero, Gabriella Arena, Adriana Ciardiello, Federica Rosso*

DOI: 10.30682/tema0802h

The disused precious stone elements are not CDWaste. A digital management chain to save them**56***Raffaella Lione, Ornella Fiandaca, Fabio Minutoli, Alessandra Cernaro, Luis Manuel Palmero*

DOI: 10.30682/tema0802i

CONSTRUCTION AND BUILDING PERFORMANCE**Orange peels as a potential ecological thermal insulation material for building application****76***Matteo Vitale, Santi Maria Cascone*

DOI: 10.30682/tema0802d

**Impact of modelling on the assessment of energy performance in existing buildings:
the case of Concordia Sagittaria**

Lorna Dragonetti, Davide Prati, Annarita Ferrante

DOI: 10.30682/tema0802e

85

SLICE - Solar Lightweight Intelligent Component for Envelopes: application for the ICARO pavilion

Angelo Monteleone, Gianluca Rodonò, Antonio Gagliano, Vincenzo Sapienza

DOI: 10.30682/tema0802g

95

BUILDING AND DESIGN TECHNOLOGIES

Application of adhesive technology to a new type of glazed panel for curtain walls with an integrated frame

Francesco Marchione, Rosa Agliata, Placido Munafò

DOI: 10.30682/tema0802c

108

ORANGE PEELS AS A POTENTIAL ECOLOGICAL THERMAL INSULATION MATERIAL FOR BUILDING APPLICATION



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Matteo Vitale, Santi Maria Cascone

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Abstract

Most of the national orange production, estimated at 900,000 tons per year, is processed in several cities in the south area of Italy. Half part of this amount corresponds to the peels, which are separated in the orange selection and processing plants. In this work, the possibility of using orange peels as raw material for the manufacture of building materials for the civil construction industry is studied. Recently, some attempts to reuse by-products derived from citrus waste have been proposed. For example, it was used as a source of nutrients in food, pharmaceutical, and cosmetic industries and as a source for energy production. There are precedents in the use of biomass residues in different building blocks, mainly with the aim of generating insulating materials. With this objective, insulating materials were obtained from agriculture by-products also manufactured without a binder. After a drying process, orange peels were characterized with electronic scanning analysis and thermal analysis in order to analyze the application in the building sector. The same by-products for the production of samples in the form of panels were used. In order to establish the best panel composition, physical and thermal properties, as well as mechanical and durability performances of the samples, were characterized.

Keywords

Agro-waste, Citrus fruit, Thermal insulator, Circular economy, Environmental impact.

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1. INTRODUCTION

In Italy, citrus agricultural surfaces are 143.540 ha. Most of the citrus crops are in Sicily, which represents 52% of national citrus production, with 80.478 ha cultivated [1]. From the citrus industrialization, different by-products are generated, which are separated in the selection and processing plants. Half part of the citrus weight is a by-product, mainly composed of peel, seeds, and residue of pulp. Incineration or use as animal food is the usual disposal method of orange peels. Other reuse methods applied to recover orange by-products are based on com-

post production, organic fertilization, biogas or pectin, bioactive and essential oils extraction [2, 3]. The huge quantity of this waste can be an environmental problem for the land. When the peels are burned in the open air, significant impacts are caused. On the one hand, large amounts of CO₂ and microparticles in suspension are produced, on the other hand, the soil is rendered unusable, and the degradation of the burning area is caused. A circular approach could help the building sector to reduce the environmental impact and rising costs [4]. There are

precedents in the use of this type of residue of biomass to create construction panels or bricks. Agricultural residues from rice [5], sugar [6], corn cob [7], or pineapple [8] are used to make thermal insulation material. Other sources are represented by industrial by-products from the agricultural transformation of peanut [9], coffee [10], coconut [11], sunflower [12], or walnut [13]. Novel and green approaches to directly using the extract of citrus fruit's peel as a print transfer medium, solvent for recycling polystyrene waste, and natural polymers were also developed [14]. Shan et al. studied a new type of plastic made from citrus peel whose properties are similar to those of polystyrene [15]. Orange peel ash was analyzed in addition to cement, mixing orange peel ash with cement in different percentages between 2.5 and 10% [16]. In Sicily, orange peel is an abundant by-product with a low market value but with potential use as building materials. In this work, the possibility of using oranges by-products as raw material for the manufacture of bio-insulation for the civil construction industry is studied. So, in order to obtain a new ecological material, a microscopical analysis was conducted on orange peel. After the characterization of the grains, the creation of particleboards with a pressing and drying process was investigated. Three different types of panels have been made in order to obtain the best thermal and mechanical performances. After thermal characterization, each panel was divided in two pieces and mechanical performances were compared before and after a test in a climatic chamber.

2. MATERIAL AND METHOD

2.1. CHARACTERIZATION OF ORANGE PEEL

The citrus peels used for this research were obtained from the production waste of the orange juice machines. This type of machine is widespread in coffee shops and supermarkets, which produce a large quantity of waste every day. The citrus by-product obtained is composed of 60-65% of the weight of peels, 30-35% of pulp, and the residue part of the seed, in variable percentage as a function of the orange type used [17]. The peel is composed of two different parts; the flave-

do (exterior yellow peel) and the albedo (interior white spongy peel). Albedo is about 17% of the citrus weight and is rich in pectin. [18] The chemical composition of the orange peel is mainly influenced by the external climatic condition, the cultivation method, and the type and maturation of the fruit. It is mainly composed of cellulose, pectin, sugar, acids, lipids, mineral elements, essential oil, and vitamins. Table 1 presents the chemical characterization of wet citrus waste. Furthermore, citrus peels are characterized by a high acidity with a pH from 3.5 to 5.8. Bulk density, which depends on the possible storage techniques, varies approximately from 900 kg/m³ for the wet peel to 200-300 kg/m³ for dried biomass. The large density variation between wet and dry depends on the large water content of orange citrus fruit. Water content varies from 90% for wet material to 8-10% for dried biomass.

Parameter	Wet citrus waste [% dry matter]
Water content	72.50 – 87.00
Volatile solids	93.80 – 96.70
Protein	6.53 – 8.30
Fat	0.90 – 3.30
Fiber	10.60 – 42.10
Starch	1.00 – 2.90
Sugar	15.00 – 46.60

Tab. 1. Range of physical-chemical composition of CPW.

2.2. MICROSCOPICAL OBSERVATION

A scanning electron microscopy of the materials was made in order to evaluate the microstructure and the elementary chemical composition of orange peel and panels. This experimental work has been performed at the Microscopy Laboratory of the Universidad Complutense de Madrid. The dry orange peel without compaction process and the orange peel panels obtained from the pressing phase were analyzed. In order to increase the conductivity, samples were coated with a gold film. An analysis of the microstructure of the samples was obtained using a scanning electron microscope (SEM) model JEOL JSM 6400, with an acceleration voltage of 20kv.

2.3. MANUFACTURING OF SAMPLES

In order to prepare the material to be used in the tests, shredding and drying activities were carried out, obtaining wet and dry products. The wet product is only derived by shredding the by-product by a manual mill, while the dry product has undergone a drying process and subsequent shredding with a manual mill. A ventilated oven with a temperature of 50°C was used for the drying of the material carried out for 48h. A weight variation of less than 1% on three consecutive measurements has been defined as the end of the drying process. After drying, the peels were shredded with a manual mill and sieved, obtaining the grain sizes as shown in Table 2. Diameters smaller than 0.65 mm and larger than 6 mm have been discarded. Three types of samples were obtained from granules: G1, with $0.65 < d < 1.25$ mm, G2, with $1.25 < d < 4$ mm and G3, with $4 \text{ mm} < d < 6$ mm. For the tests, a 250 x 250 mm mold with a thickness of 30 mm was used, and the granules without compaction were placed inside.

Samples	N. Samples	Particle size
G1	3	$0.65 < d < 1.25$ mm
G2	3	$1.25 < d < 4.00$ mm
G3	3	$4.00 \text{ mm} < d < 6.00$ mm

Tab. 2. Granulometries of dried and sieved orange peels.

For the realization of specimens in the form of panels, a pressing process and subsequent drying in a ventilated oven were carried out. A wooden mold 250 mm x 250 mm x 30 mm was used, and a pressure of 0.02 MPa, equivalent to a weight of 126 kg, was impressed on all the specimens. According to the scheme shown in figure 1, the load from two marble slabs of 9 kg and cylindrical concrete blocks of 12 kg each was obtained. Each sample

was kept under pressure for 15h at the ambient temperature of $21 \pm 1^\circ\text{C}$ and $50 \pm 10\%$ of relative humidity.

Different types of samples can be distinguished depending on the starting material used. As shown in Table 3, three specimens from the compaction and pressing of the material of the wet product were obtained (W100), and three from the mixing of wet product and dried product in granules with a diameter of $1.25 \text{ mm} > d > 0.65$ mm were derived (CW75_G2) and others three from the mixing of wet product and dried product in granules with a diameter of $4 \text{ mm} > d > 1.25$ mm (CW70_G1). After pressing, the specimens were unmolded and dried at 50°C for 48 hours in a ventilated dryer.

Samples				Weight of specimen parts	
Name	N. of samples	% wet	% dry	Wet [g]	Dry [g]
CW100	3	100	0	500	0
CW75_G2	3	75	25	500	180
CW70S_G1	3	70	30	500	250

Tab. 3. Samples from cold pressing and subsequent drying.

2.4. THERMAL PROPERTIES

A guarded hot plate apparatus, model HFM 436 Lambda from Neszcht was used for the thermal characterization. The test was carried out according to the European Standard EN ISO 13787 and consisted of the placement of the samples 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 by a calibrated heat flux transducer was measured. Once the thermal equilibrium had been reached, the measurements were performed.

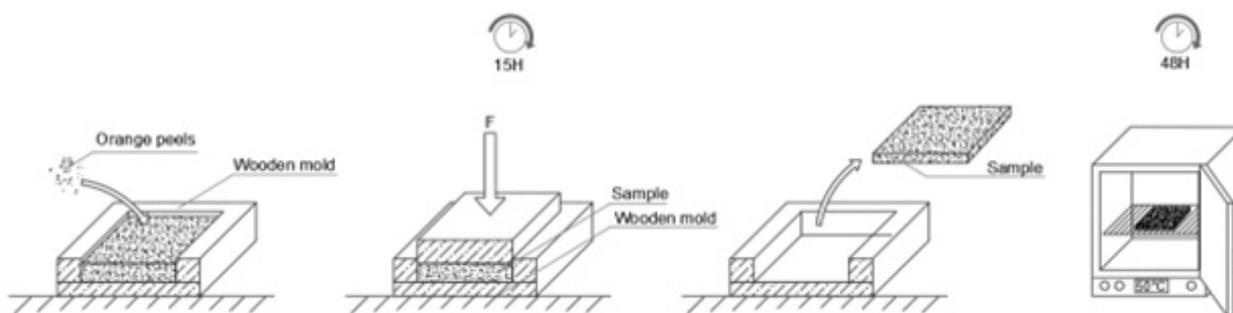


Fig. 1. Cold press scheme.

Plate temperatures were controlled by two-way heating/cooling systems integrated into a forced-air heat exchanger that generates a closed-loop flow.

According to the standardized test technique, samples measuring 250 x 250 mm with an average thickness of 30 mm were tested. Both granular samples (G1, G2, and G3) and samples in panel form were measured (CW100, CW75_G2, and CW70S_G1). For the granular samples, a containment mold was used. The samples were measured at 10°C, 20°C, and 30°C with a temperature gradient of 20°C.

2.5. MECHANICAL PROPERTIES

Mechanical characterization was performed using a three-point bending strength test. A Universal Test Machine was used, which provided values describing the force-displacement curve. The load cell was 10kN 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 [17], Values for Modulus of Rupture (MOR) by flexural stress were determined using the following Eq.:

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

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 [18] 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. Results were considered as the average of four measurements on specimen size of 250 mm x 125 mm x 30 mm.

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

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

Where F is the force applied (N), L is the distance between the supports (mm), y is the strain of the sample

(mm), I is the moment of inertia (mm^4). Samples in the form of panels were measured (CW100, CW75_G2, and CW70S_G1) before and after the durability test.

2.6. DURABILITY TEST

Durability tests were carried out in order to evaluate the mechanical properties of the samples after hot-rain cycles. According to EN12467, the test consists of the execution of 50 dry-wet cycles in a climatic chamber. Each dry-wet cycle consists of drying for 3h at 60°C and 20% of relative humidity, followed by 3h at 25°C and 90% of relative humidity. Before and after the test, the weight and the dimensions of each sample were measured. Before performing the test, each sample type 250 mm x 250 mm x 30 mm was divided into two equal parts. One part for each type was placed in the climatic chamber to perform the test; the other part was taken as a reference without the climatic chamber. After 50 cycles, a three-point bending test on both samples was conducted in order to compare their mechanical performance.



Fig. 2. Climatic chamber used for durability test.

3. RESULTS

3.1. MICROSCOPICAL OBSERVATION

The microscopical observation of the dried orange peel shows two different layers. The external part (Fig. 3a) is characterized by a compact structure and small hole; this part corresponds to the exterior orange part, called flavedo. The interior part (Fig. 3b) is much porous and corresponds to the inner white part of the orange peel, called albedo. This part is composed of a small sac or cavity, which gives the material the thermal insulation properties. Fig. 3c and 3d show the joint between the two parts and show that the peel is half composed of the external part and half of the inner part. The microstructure of the peel, although heterogeneous, can be

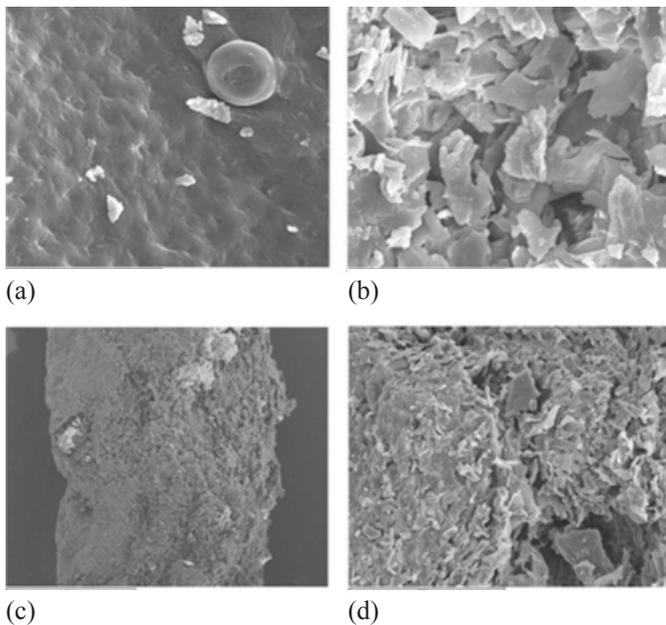


Fig. 3. Scanning electron microscopy images of dried orange peel.

therefore divided into two main parts, a rigid external that performs the “structural” function and a spongy internal that performs the function of thermal insulation.

The internal structure (Fig. 4a) can be compared to commercial products such as extruded polystyrene (XPS) (Fig. 4b) or expanded perlite (Fig. 4c). By comparing the three materials, they all have a closed-type cellular structure, and microstructure similarities can be found. As expected, with respect to the cell structure of the orange peel, a much more regular and uniform shape characterizes the XPS. Polystyrene has sacs with a diameter of about 200 μm ; the perlite has holes of about 40 μm . In terms of microstructure, expanded perlite is similar to dried orange granules. Other differences in density and thermal conductivity can be found. The XPS has a density of 35 Kg/m^3 and a conductivity of 0.035 W/mk , values significantly lower than the citrus granules. Expanded perlite, with a density of 100 Kg/m^3 and a conductivity of 0.045 W/mk , has similar properties to orange granules but differs in the microstructure. A homogeneous structure characterizes the expanded perlite, while a heterogeneous structure characterizes the orange peels, composed of an inner spongy layer and an outer rigid layer. The comparison of microstructures was made using only the inner part of the orange granules as a reference. This part provides samples with good insulating properties, but represents only 60% of the base material. The remaining 40% of the base material, consisting of the outer part of the peel and the seeds, contributes to the improvement of the mechanical performance of the samples.

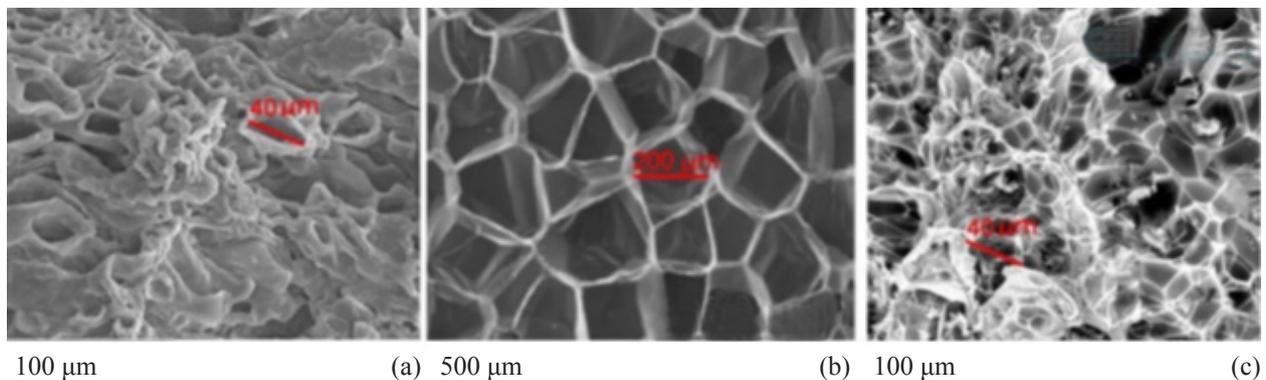


Fig. 4. Images from SEM: microstructure orange peel (a); XPS microstructure (b); expanded perlite microstructure (c).

3.2. DENSITY

Bulk density was determined as the relationship between the mass and the volume of the boards according to European Standard 1602 [35]. Testing was carried out on dry samples under laboratory conditions ($21 \pm 1^\circ\text{C}$ and $50 \pm 10\%$ relative humidity). An electronic balance with a precision of 0.001 g and a digital caliber VOGEL model 202112 with a resolution of 0.01 mm were used. Table 4 shows that, for the granular samples, the density increases as the diameter of the granules decreases. A mean density of 500 kg/m^3 was measured in granular samples without compaction.

Samples	Density [kg/m^3]
G1	509.52
G2	497.37
G3	492.71
CW100	507.32
CW75_G2	503.00
CW70S_G1	520.74

Tab. 4. Density of dried orange peel granules.

An average density of 510 kg/m^3 of the samples in the form of panels is obtained. Compared to traditional building panels (Tab. 5), the samples are medium-high density panels. Other panels from agricultural by-products have similar densities as those derived from Sunflower (500.00 Kg/m^3) [19], Corncob (413.00 Kg/m^3) [20], and Durian (442.00 Kg/m^3) [21]. Other panels derived from agricultural by-products have higher densities, such as panels from walnut (599.10 Kg/m^3) [22] and Sugar cane (686.00 Kg/m^3) [6], or lower densities, such as panels from Coconut (338.00 Kg/m^3) [21] and pineapple (210.00 Kg/m^3) [23].

3.3. THERMAL PROPERTIES

Thermal conductivity is calculated at 10°C , 20°C , and 30°C on dry specimens. Table 5 shows the conductivity of the grains, which is directly related to the density of the specimens. Thermal conductivity increases as the diameter of the grains decreases. The higher percentage of voids reduces density and ensures better thermal in-

sulation. Therefore, the smaller diameter of the granules conducts more heat than the larger ones. By comparing the results obtained, a higher conductivity is reached by the granules with respect to others from agricultural by-products such as rice husk (0.050 W/mk), sugar cane fiber (0.048 W/mk), coconut fiber (0.053 W/mk), and a comparable conductivity, although higher, than the granules from the cladodes of *Opuntia ficus-indica* (0.057 W/mk) is obtained. Samples from citrus with higher grains have a lower conductivity than some commercial products such as expanded clay (0.090 W/mk), pumice (0.100 W/mk), expanded vermiculite (0.080 W/mk), and cellulose granules (0.069 W/mk).

Orange by-products	Density [kg/m^3]	Thermal conductivity		
		10°C [W/mK]	20°C [W/mK]	30°C [W/mK]
G1	509.52	0.068	0.072	0.078
G2	497.37	0.066	0.071	0.075
G3	492.71	0.066	0.070	0.073
CW100	507.32	0.067	0.069	0.073
CW75_G2	503.00	0.079	0.082	0.088
CW70S_G1	520.74	0.081	0.085	0.090

Tab. 5. Density of building panels from agricultural waste.

An average conductivity of 0.075 W/mk , in a range between 0.067 W/mk and 0.081 W/mk , is obtained from the samples in the form of panels. The minimum conductivity of 0.067 W/mk is from samples CW100 and shows this formulation's interesting property.

Equal or superior properties are shown by this sample in comparison with others of similar density. The sample CW75_G2 has the lowest density with thermal conductivity of 0.079 W/mk . The CW100 thermal conductivity is 0.067 W/mk and increases by 1.2 times to reach 0.081 W/mk in W70S_G1. As shown from microscopic analyses, these properties can be justified by the type of microstructure obtained from the use of the composition of a wet and dry part. The wet material, in fact, generates closed air pores with a higher thermal resistance than the open ones in the case of the addition of dry matter. During drying, the replacement of the water contained in the citrus with air becomes greater in samples with a higher percentage of wet matter (CW100).

Some differences between the results at 10°C, which is the reference temperature for construction materials, and those at 20°C, need to be highlighted. The increase in test temperature corresponds to an increase in thermal conductivity in a range from 4% to 12%. The hygroscopic characteristics of this material affect the thermal properties of the samples. Compared to literature, orange peel samples showed higher thermal conductivity than other non-natural commercial insulators such as mineral wool or polystyrene. However, the conductivity values obtained from orange peel panels are in line with the conductivity of other insulating materials from agricultural by-products such as durian [21]. In addition, the conductivity is lower than the panelboard made from sunflower [19] and walnut shells [13] and higher than the panelboard made from coconut shells [21], pineapple leaves [23], and sugar cane [6].

3.4. MECHANICAL PROPERTIES

The mechanical properties of the samples in the form of panels are shown in Figure 5. The addition of dried peels provoked a reduction of flexural strength. CW100 samples have a flexural strength of 0.36 MPa, which fell down to 0.15 MPa in CW70_G1. This can be explained by the homogeneity of the microstructure of CW100 panels compared to the mixtures of the other samples. The addition of dried granules in the samples CW75_G2 and CW70S_G1 generated a discontinuity in the microstructure, resulting in a loss of particle bond and mechanical strength. Other research has shown that the addition

of aggregates reduces the mechanical properties of mixtures, such as cement with the addition of perlite [24] or plastic grains [25].

Compared to the literature, strength is lower than other agro-wastes boards with corncob [7], coconut coir [21], and sunflower [26], although similar to the bagasse ones [27]. However, the differences can be explained by the different manufacturing process or the presence of an external binder. The use of a higher compression pressure would increase the material's mechanical strength and density, resulting in an increase in thermal conductivity. Moreover, the addition of external binder would improve the bonds between the particles, resulting in a non-biodegradable panel.

Modulus of elasticity (MOE) followed the same pattern as the MOR (Fig. 5b), and the stiffness is higher in the sample without dried peels. Indeed, CW100 samples show a modulus of elasticity higher than the other two. This performance can be explained due to the formation of closed porous and homogeneity in the mixture compared to the others.

3.5. DURABILITY TEST

Samples after 50 cycles in the climatic chamber have a weight reduction directly proportional to the increase in the percentage of dried particles. The weight difference before the cycles, calculated as the average between 3 samples, is -3,62% in the samples made with the only wet part. As shown in table 6, a larger decrease has characterized CW75_G2 and CW70S_G1 samples.

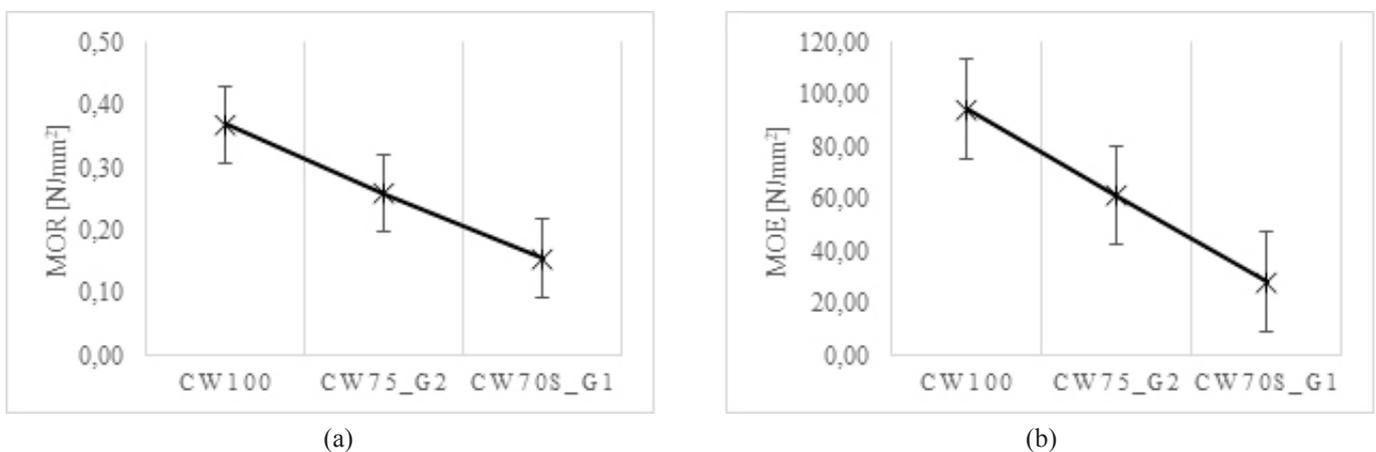


Fig. 5. Flexural (a) strength and modulus of elasticity (b) of the samples.

The higher weight decrease indicates a better ability of the material during the hot cycle to release the water absorbed during the wet cycle. A greater weight reduction in the samples with a greater quantity of pre-dried grains is measured. So, the panels' ability to absorb and release environmental humidity is related to the dry grains' quantity. In particular, the addition of the dry part results in a better hygrometric behavior of the panels. This improvement can be explained by the heterogeneity of the panel and by the presence of more empty parts inside the material.

Sample	Weight before cycles [g]	Weight after cycles [g]	Weight difference [%]
CW100	96.64	93.14	-3.62
CW75_G2	154.68	148.60	-3.93
CW70S_G1	177.36	169.51	-4.44

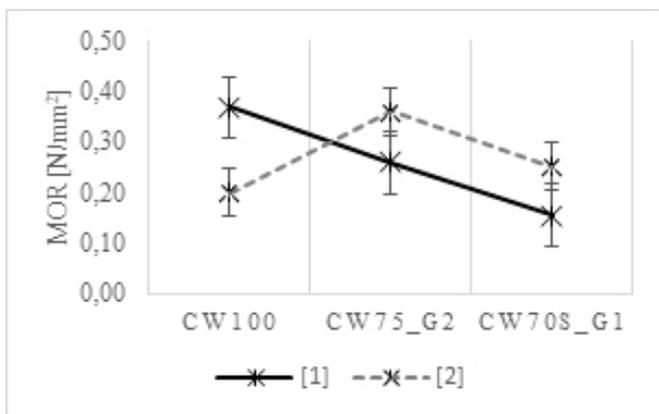
Tab. 6. Weight before and after climatic chamber.

As a consequence of the weight reduction of the samples after the durability test, figure 6 shows an increase in the modulus of rupture. In particular, flexural stress increases in the CW75_G2 and CW75_G2 samples while decreases in the CW100 samples. Results of the mechanical tests on samples after 50 dry-wet cycles show an increase in mechanical performance on materials with low water content. As for thermal tests, this is demonstrated by the microscopic structure of the panels and the increased presence of voids in the samples with dry grains. Despite the more porous structure, the tests show that the

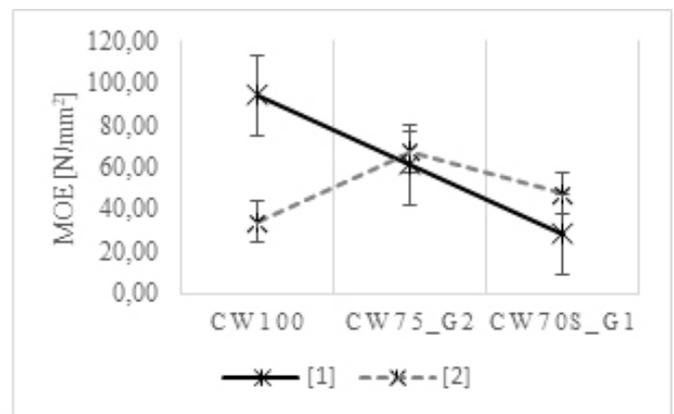
bonds between the citrus granule particles are retained in the mixture with dry grains, and that is improved with lower water content.

4. CONCLUSION

In this work, the possibility to use orange by-products as based material for building insulation is shown. Some similarities from microscopical observations are founded in microstructure between orange peels and commercial thermal insulators. Comparing the density of citrus peels with other building insulation materials from agriculture by-products, it appears that the results are similar. Panels by a cold-pressing process without the use of binders can be produced using citrus by-products and without sophisticated machines. A pressing and subsequent drying process creates a rigid panel with a density of 507 kg/m³. The conductivity of 0.066 W/mk classifies the panel as a thermal insulator. The climatic chamber test shows that mixing wet and dry parts of citrus granules improves the material's ability to absorb environmental moisture and release it. After 50 cycles in the climatic chamber, mechanical properties have seen samples with dry parts and an improvement in the rupture modulus. Panels with higher water absorption showed worse thermal and mechanical properties. Moreover, the presence of water could also create an environment conducive to biological attacks. However, the ability of the panel to absorb and release moisture could be a good way to maintain constant thermo-hygrometric conditions in an indoor environment.



(a)



(b)

Fig. 6. Flexural (a) strength and modulus of elasticity (b) of the samples before [1] and after [2] durability test.

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