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Ceramic bricks using pistachio shells as controlled porosity former

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Abstract. The present work studies the use of pistachio shells as a porosity-forming raw material in the manufacture of ceramic bricks. It focuses on the characteristics of the ceramic pieces obtained with different residual biomass contents (from 5 to 20% by volume). The specimens were shaped by uniaxial pressure of 25 MPa, and then were treated at 950°C for 3h. Based on the results obtained it is possible to conclude that the use of this residual biomass, as a pore former in the ceramic industry is feasible. The proportion of added biomass that generates the best characteristics and properties in the final product is 10%. Up to this percentage of aggregate, ceramic pieces with very good macroscopic and microscopic characteristics are obtained, with porosity and flexural strength values that meet market requirements for this type of product. Optical microscopy technique has been used to carry out a detailed analysis of the shape and size of the formed pores, which is a specific objective of this work. It has been observed that for contents of 5 and 10% biomass, the shape of pores is similar to the shape of the particles of added pistachio shells, while for higher contents (15 and 20%) it is different with elongated characteristics. In relation with the size of the pores, at lower contents is around 60% of the original particles size, and for the higher percentages of biomass, they are larger, indicating that agglomerates of particles are formed and when combusted, produce that porosity characteristics.

Key words: Residual biomass, pistachio shells, lightweight ceramics, pore characteristics.

1. Introduction

The present work corresponds to the study of pistachio shells as a raw material in the manufacture of ceramic bricks. It focuses on the characteristics of the clay used and the ceramic pieces obtained with different residual biomass contents. The properties of the obtained bricks in relation to the market requirements for this type of materials are also analysed.

This waste is a lignocellulosic material, which means that it is mainly formed by cellulose, hemicellulose and lignin, or biopolymers with similar structure complexity. Its behaviour in biomass-energy processes has been studied since by calcination it exhibits decomposition and combustion reactions, which release significant amounts of energy (Hosseinzaei et al., 2022). The pyrolysis processes of this biomass have been extensively studied, both as a bulk material and as a source of various biopolymers that compose it (Acikalin et al., 2012; Peters, 2011).

In the last decade there have been numerous investigations aimed at the valorization of agricultural wastes for diverse uses (Bhuyan, 2020). In the case of pistachio shells, they have been studied for various applications, such as activated carbon production (Niksiar et al., 2017; Niksiar et al., 2018; Foroushani, 2016), heavy metal adsorption (Banerjee et al., 2019; Cheng, 2022), source of other compounds like essential oils (Marett et al., 2017; Kasiri et al., 2018; Mohammadi et al., 2019), catalyst supports manufacture (Taghizadeh et al., 2018), etc.

The studies on complete pyrolysis of this biomass have found relatively low calorific capacities when compared with those of coal traditionally used for that purpose, but interesting

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intermediate products have been determined when the pyrolysis is performed in certain controlled pressure and temperature conditions. One such intermediate product is known as PSC, pistachio shell carbon, and has been successfully used as a heavy metal biosorbent (Siddiqui et al., 2017). Dolatabadi et al. (2021) for example, have used pistachio shell based activated carbon as an economical and promising treatment for the removal of insecticides from groundwater.

In relation to the use of pistachio shells as a structural material, due to its fibrous constitution, it has been studied as a reinforcement of polymeric matrices (Koodalingam, et al., 2020; Thiagarajan et al., 2021). Also, Tekin et al. (2021) have analysed pistachio shell ash as supplementary cementitious material in the construction industry. The authors have found that the presence of graphitic structures in the ashes contributed to the early strength properties of cement. Besides, 10% ash incorporation improved the cement compressive strength values by 17% at long times, and for short periods, strength values were found to be satisfactory at even 20% aggregate.

In the case of ceramic materials, their physical and mechanical properties depend largely on the raw materials used in their manufacture. One of these properties is porosity, which is usually managed during industrial production by incorporating organic additives derived from petroleum. When combusted, they generate pores, which lighten the bricks and provide insulating properties, although the use of these pore-forming agents implies an increase in the cost of the products.

In recent years, some agro-industrial residues have been studied as porosity-forming materials in bricks because they present very high weight losses due to combustion, leaving very small proportions of inorganic material (ash) which is incorporated into the brick matrix (Bories et al., 2014; Al-Fakih et al., 2019; Martín-Morales et al., 2018; Ukwatta and Mohajerani, 2017; Raut et al, 2011; Quaranta et al., 2020; Pelozo et al., 2022; Quaranta et al., 2023).

The results obtained in preliminary studies, in relation to the characteristics of the pistachio shells for the formation of porous matrices have allowed determining that there is a high feasibility of its use. Suitable particle sizes can be obtained in a simple way, and its combustion occurs in a wide temperature range, which would not affect the structure of the ceramic piece during the formation process. In addition, its combustion is complete, with production of CO_2 and water, with negligible concentrations of CO. The amount of ash produced, which would be incorporated into the ceramic matrix is very small, so the properties of the ceramic material would not be modified by its presence. The only consideration to take into account is that the residual powder material has eco-toxic characteristics in relation to the development of sensitive plant species such as ryegrass, so special care must be taken during the storage process in the company.

The main objective of this research is to achieve the manufacture of lightweight ceramic bricks with the incorporation of pistachio shells, and in particular their use as a controlled porosity former. The characteristics of the obtained bricks and their properties in relation to the market requirements will be analysed. A specific objective of this work is to study the shape and size of the obtained pores in relation to the size and shape of the aggregated biomass particles.

2. Experimental program

The raw materials used for the manufacture of bricks are treated by means of drying (100°C for 4h) and grinding processes, in order to obtain the suitable particle size distribution according to the design carried out, taking into account the procedures used in ceramic brick production companies.

The grinding of the materials was carried out using a ball mill for 30 minutes for the clay, and a ring mill for 2 minutes for pistachio shells. The ground materials (clay and pistachio shells) were sieved to obtain particles smaller than 1 mm, which will be used for the formation of the compacts.

The analysis of the particle sizes distribution was made according to the ASTM standards, with a ZonyTest equipment. The process was carried out using 500 g of dry material, maintaining continuous vibration for 10 minutes. The material retained on each sieve was then determined. Table 1 shows the correspondence between the standardized mesh used and the passing particle size.

Table 1. ASTM mesh number and particle size correspondence.

ASTM mesh	10	18	35	60	120	170	200	325	Plate
Sieve opening [μm]	2000	1000	500	250	125	88	74	44	< 44

The materials used for the production of ceramic specimens were characterized by various techniques: optical microscopy (OM), scanning electron microscopy (SEM), dispersive X-ray analysis (EDS), differential thermal and thermogravimetric analysis (DTA-TGA), X-ray diffraction (XRD) and weight loss on ignition (LOI).

Optical observations were made with a Zeiss-Axiotech equipment with a Donpisha 3CCD camera. SEM analyses were carried out with a Philips 515 scanning electron microscope, with dispersive energy analyser (EDAX-Phoenix). The chemical analysis was performed with dispersive energy analyser EDAX-Phoenix. DTA-TGA essays were made with a Shimadzu DTA-50, TEG-50, with TA-50 WSI analyser, using heating rate of 1°C/min, in temperature range ambient-1100°C, in air atmosphere. The clay weight loss on ignition was carried out with a laboratory oven at 800°C for two hours, with a heating rate of 5°C/min, on samples of 20 grams.

The green compacts are formed from these ground materials with different volume percentages of pistachio shells (biomass), and the obtained samples will be identified as follows:

- P0: bricks without biomass
- P5: bricks with 5% biomass incorporated
- P10: bricks with 10% biomass incorporated
- P15: bricks with 15% biomass incorporated
- P20: bricks with 20% biomass incorporated

Several samples (bricks) were produced from each composition, in order to carry out different tests and properties analysis in a reproducible way. The sample P0, without incorporated biomass, is taken as a reference sample.

For the bricks preparation, 80 g of clay, the corresponding volume percentage of the biomass, and finally 8% by weight of water on the total mixture, were used. The dry materials are mixed until homogeneous mixture before incorporating the water, and the moisture is added slowly, trying to avoid the formation of lumps, since the clay has hydrophilic surface characteristics greater than those of pistachio shells.

Once the mixtures are obtained, the specimens are shaped by uniaxial pressure of 25 MPa, using a hydraulic press, Cific brand, in moulds of 70mm x 40mm, resulting thicknesses of c.a. 15mm. The compacts obtained with different percentages of residue are arranged to dry at room temperature, in an aerated place for 24-48 hours, constituting the so-called green bodies, and then at 100°C (24h).

For the sintering of the bricks, the pieces are heat treated at 950°C for 3 hours, at a heating rate of 1°C/min. This stage is carried out in an electric oven (ORL brand) following a heating curve similar to the one used regularly by companies that produce ceramic bricks.

The products obtained after the firing process were characterized with various techniques: permanent volumetric variation (PVV), weight loss on ignition (LOI), apparent porosity (P), optical microscopy (OM) and flexural strength (MOR). The objective of applying these techniques is to determine the properties of these materials, in order to evaluate if they meet the market requirements in their performance in service.

PVV determination is carried out by measuring the length, width and thickness dimensions of each sample, before and after firing, calculating the volumes in each case and the volume variation that occurred due to the firing process.

The test to determine LOI is carried out by making the difference in weight of each brick, before and after firing, and averaging the values of the five samples of each composition.

The porosity of the samples was determined according to the ASTM C20-0 Standard. To do this, small pieces of the brick are cut using a precision diamond saw, taking samples from the central and external regions of the bricks. This is due to a greater macroscopically presence of pores observed in the internal zone than in the superficial one, and the standard yields a single porosity value, so this procedure is carried out in order to avoid errors in the determinations.

Optical microscopy analyses were used to observe the microstructure of the bricks, the presence of cracks or micro cracks inside the material, and the size, shape and distribution of the present pores. The preparation of the samples for OM observation is carried out firstly by embedding in resin, extracting the air by means of a vacuum pump, to allow the resin to enter the exposed pores. This allows the next stage (polishing) to be performed correctly, avoiding shelling during this process. The specimens were polished using a two-plate automatic polisher (Praxis brand) with water-based SiC paper of different grain sizes (400 to 1500), and the final finish was performed with polishing cloths and 6μm and 1μm diamond pastes.

The flexural test was carried out with a universal testing machine on scale samples, according to the relationships established in the ASTM C67-03a Standard. The main objective of this study is to analyze the mechanical behaviour of these materials, when subjected to a pure bending stress. In this way, the modulus of rupture (MOR) of the bricks was determined.

3. Results and discussion

3.1. Raw materials characterization

Table 2 presents the chemical analysis of the used clay, expressed as percentage of the elements, without taking into account in this calculation the carbon and oxygen content determined in the samples, which could be part of organic matter, or inorganic compound (carbonates).

The determined composition of the biomass is 70.4% C and 29.6% O.

Table 2. EDS analysis of the used clay.

Element	Mg	Al	Si	K	Ca	Fe
Clay composition [%]	4.5	26.7	58.4	5.7	1.5	3.2

The weight loss on ignition of the clay was 1.12% and for pistachio shells was 98.2%.

The X-ray patterns of pistachio shells is presented in Figure 1. Four reflection peaks are observed at values of 16.7, 21.7, 34.7 and 44.4, which have been assigned to microcrystalline cellulose. Other authors, in work done with sisal fibres (Benítez-Guerrero et al., 2014), have obtained similar spectra. Mtibe et al. (2015) have also observed these reflection peaks in cellulose and cellulose nanofibers extracted from residues of the corn industry.

The X-ray pattern of the clay shows the presence of iron oxide in the hematite structure (Fe_2O_3), silicon oxide in the quartz structure (SiO_2), potassium feldspar ($xAlSiO_8$), iron (Fe) and aluminium oxide (Al_2O_3). The diffractogram have been analysed taking into account the PCPDFWIN - International Centre for Diffraction Data - 2000 database.

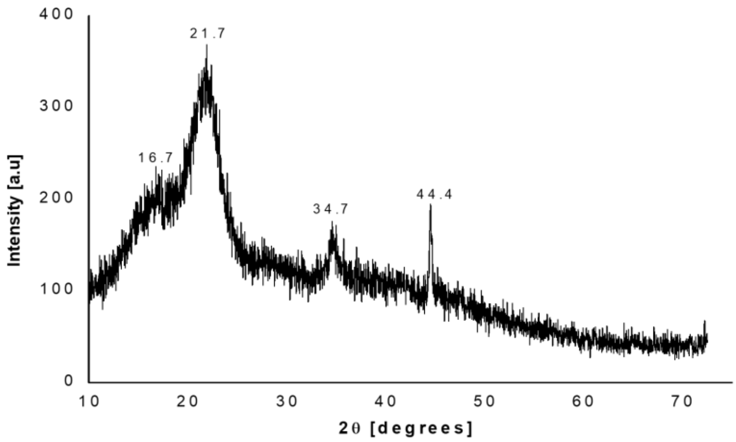

Fig 1. XRD of grounded pistachio shells.

The result of the DTA-TGA test is shown in Figure 2. The presence of two exothermic peaks in the DTA at 335°C and 450°C is observed. The latter is a wide peak that covers approximately 130°C (from 370°C to 500°C). The TGA curve presents a first gentle slope of weight loss up to approximately 227°C, and then two other zones in the ranges 227°C-270°C and 280°C-460°C. These two zones would indicate different reactions probably corresponding to the combustion of hemicellulose, cellulose and lignin, with their corresponding weight losses. These compounds are present in the vast majority of agro industrial biomass (Manals Cutiño et al., 2011).

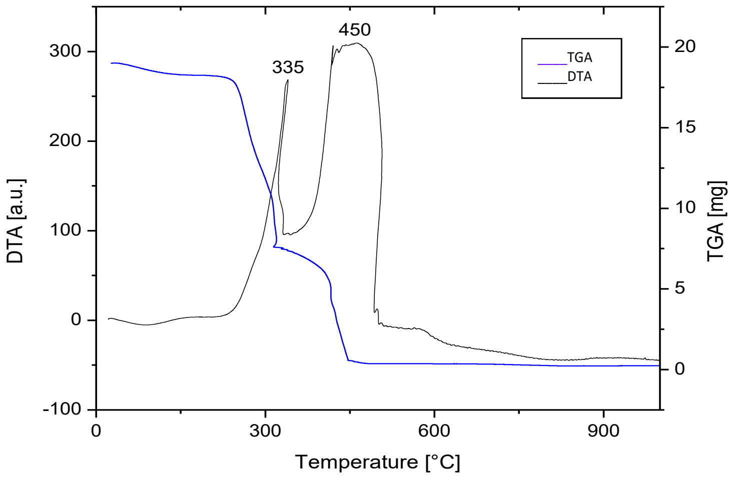

Fig 2. DTA-TGA of pistachio shells.

The clay sample shows a typical DTA-TGA curve for this type of material, with continuous weight loss between 200°C and 700°C. This can be assigned to various reactions, up to 400°C a slow endothermic dihydroxylation reaction of the clay and up to 700°C the combustion of the organic material present. In the 850°C-950°C range, a peak in the DTA curve without weight loss was recorded. This can be assigned to the phase transformation reaction of the alumino-silicates present in the sample, which is a reversible reaction.

3.2. Ceramic bricks production

Figure 3 shows the results of the particle size distribution of the materials used to make the ceramic bricks. It can be seen that more than 50% of the clay has a particle size greater than 250μm and the rest is evenly distributed among the sizes of the selected sieves. In the case of pistachio shells, approximately 75% of the sample has sizes greater than 500μm. This granulometric distribution is adequate to achieve a good homogenization of the mixtures of both materials at the beginning of the production of the bricks.

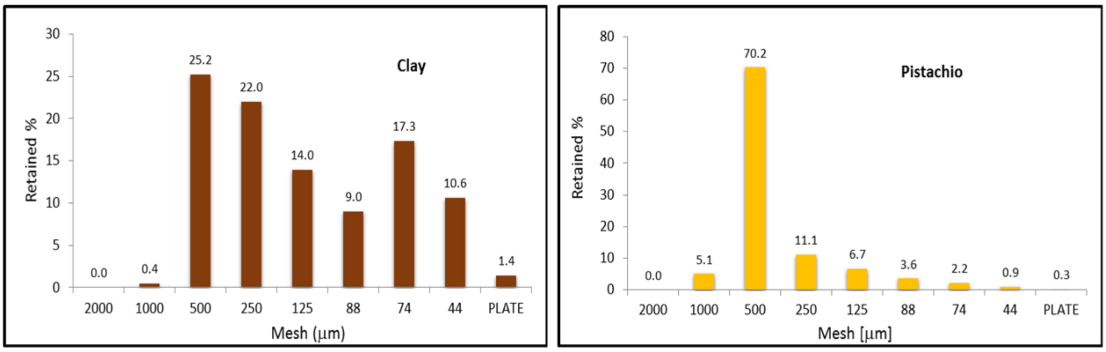

Fig 3. Particle size distribution of the used raw materials.

Figure 4 shows the aspect of the green pieces obtained after the drying process at room temperature. It is possible to appreciate that as the amount of biomass added increases, the surfaces of the bricks appear rougher, and the edges and corners appear less defined.

As mentioned above, all sintered samples have been produced at 950°C. The obtained compacts are also shown in Figure 4. A homogeneous reddish colour in all samples can be observed. In addition, a greater porosity in the bricks is notable as the amount of incorporated residue increases. In the samples with higher residue contents (15% and 20%), slight shattering of the structure can be observed on edges and corners. Similar results have been obtained in previous works, in the production of porous ceramic bricks using other residual biomass such as corn cobs (Quaranta et al., 2023) and peach pits (Quaranta et al., 2020). Martín-Morales et al. (2018), analyzing the behavior of ceramic bricks obtained with aggregates of various biomasses (rice husks, almond shells, olive pits, olive wood and olive pruning) have determined that the best macroscopic characteristics and properties were obtained for biomass proportions by volume between 7.5% and 15%.

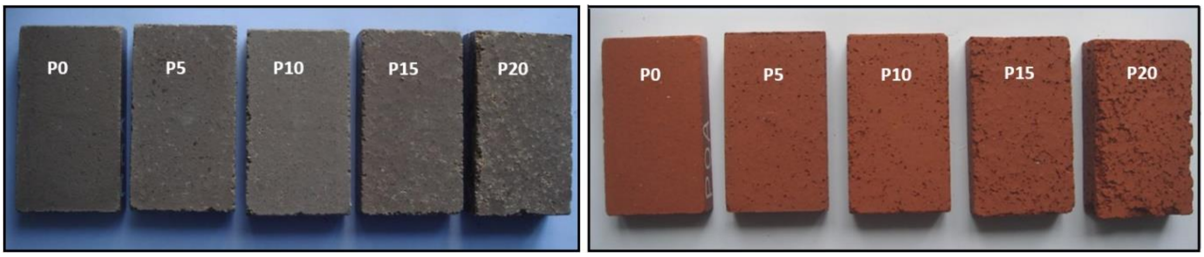

Fig 4. Green bricks (left) and bricks obtained at 950°C (right).

The PVV values of the analysed samples indicate that a contraction occurred in all of them, which increases as the residue content increases with a maximum value of 7.9% for the sample with 20% residual biomass incorporated, as can be seen in Table 3. Negative values indicate shrinkage processes.

Table 3. Properties of the ceramic bricks.

Element	P0	P5	P10	P15	P20
PPV [%]	-6.6	-7.2	-7.3	-7.5	-7.9
LOI [%]	-9.4	-13.9	-17.2	-20.1	-24.1
Porosity [%]	19.2	28.3	29.4	33.2	37.0
MOR [MPa]	9.1	6.2	6.1	4.3	5.3

The values of weight loss on ignition (LOI) of the compacts are also presented in Table 3. The results show that the higher the residue added, the higher the value of this parameter is, as expected, since the added biomass combusts inside the brick during firing, with the consequent weight loss. Negative values indicate the weight percentage of the samples that has decreased after heat treatment.

Table 3 also shows the porosity values of these fired compact samples. It can be seen that as the residue content increases, the porosity of the bricks also increases, being the maximum value recorded 37 %. This has a relationship with the LOI results mentioned above.

However, this increase in porosity does not present a direct proportional relationship, since as the weight loss increases due to biomass combustion, the porosity also increases but in minor proportions, taking into account the P/LOI values.

In Table 4 the P/LOI values are presented, as well as the following relations: P/PVV and LOI/PVV. These calculations have been made because it has been observed that for samples produced with the addition of biomass, all these values are related by the expression (1). Although this expression is not fulfilled for the case of the reference bricks, without added biomass.

$$P/PVV - P/LOI \cong LOI/PVV \quad (1)$$

Table 4. Relationship between Porosity, LOI and PVV.

Property \ Sample	P0	P5	P10	P15	P20
P [%]	19.2	28.3	29.4	33.2	37.0
LOI [%]	9.4	13.9	17.2	20.1	24.1
PVV [%]	6.6	7.2	7.3	7.5	7.9
P/LOI	2.0	2.0	1.7	1.7	1.5
P/PVV	2.9	3.9	4.0	4.4	4.7
LOI/PVV	1.4	1.9	2.4	2.7	3.1
(P/PVV - P/LOI)	0.9	1.9	2.3	2.7	3.2

The mechanical properties of the compacts were evaluated by testing the flexural strength. Table 3 shows the Modulus of Rupture (MOR) of each of the analysed samples. It can be seen that the inclusion of pistachio shells in the samples produces a decrease in flexural strength, with respect to the clay reference sample. As the percentage of residue in the sample increases, the flexural strength decreases, with the exception of the sample with 20% incorporated residue, which has a slightly higher MOR than P15 sample.

It is known that a general reduction of the mechanical properties in these kind of materials can be caused by the presence of pores, as well as other microscopic imperfections such as surface or internal cracks, which act as notches or stress points. In the case of ceramic bricks, MOR values are influenced by diverse parameters such as porosity values, permanent volumetric variation, weight loss on ignition and presence of superficial or internal microcracks. Depending on these parameters and their combinations, more or less resistant matrices can be obtained. In the case of these samples these combinations may have occurred leading to a slight higher resistance value for the sample P20 compared with P15. However, the P20 macroscopic characteristics observed, in particular the edges and corners shelling, indicate that at these percentages of residue, the quality of the products is not good.

In Argentina, although there are regulations that establish how to perform the determinations of this mechanical property, there is no required market value of flexural strength. For this reason, the values obtained in this work are compared with the value established for ASTM C410-60 standard, for industrial floor bricks, where the required MOR value is 5.2 MPa.

Optical microscopy analysis was used to study the internal structure of these bricks, in particular to determine the shape and size of the formed pores, and the presence of internal cracks. Figure 5 presents images taken from different samples using the same 100X magnification. The difference in the shape and size of the pores in samples that have important proportions of residue, compared with the P0 sample, without residue, is remarkably observed.

As can be seen in the Figure 5, in the bricks without biomass aggregate, P0, a homogeneously distributed porosity is observed, with rounded pores of small sizes that do not exceed 0.1 mm. Samples P5 and P10 present a homogeneous distribution of pores with various sizes that reach values of 0.4 mm, although the most abundant are 0.3 mm pores. In these samples, the pores present irregular shapes, similar to those observed in the particles of the ground pistachio shells, as can be seen in the image shown in Figure 6.

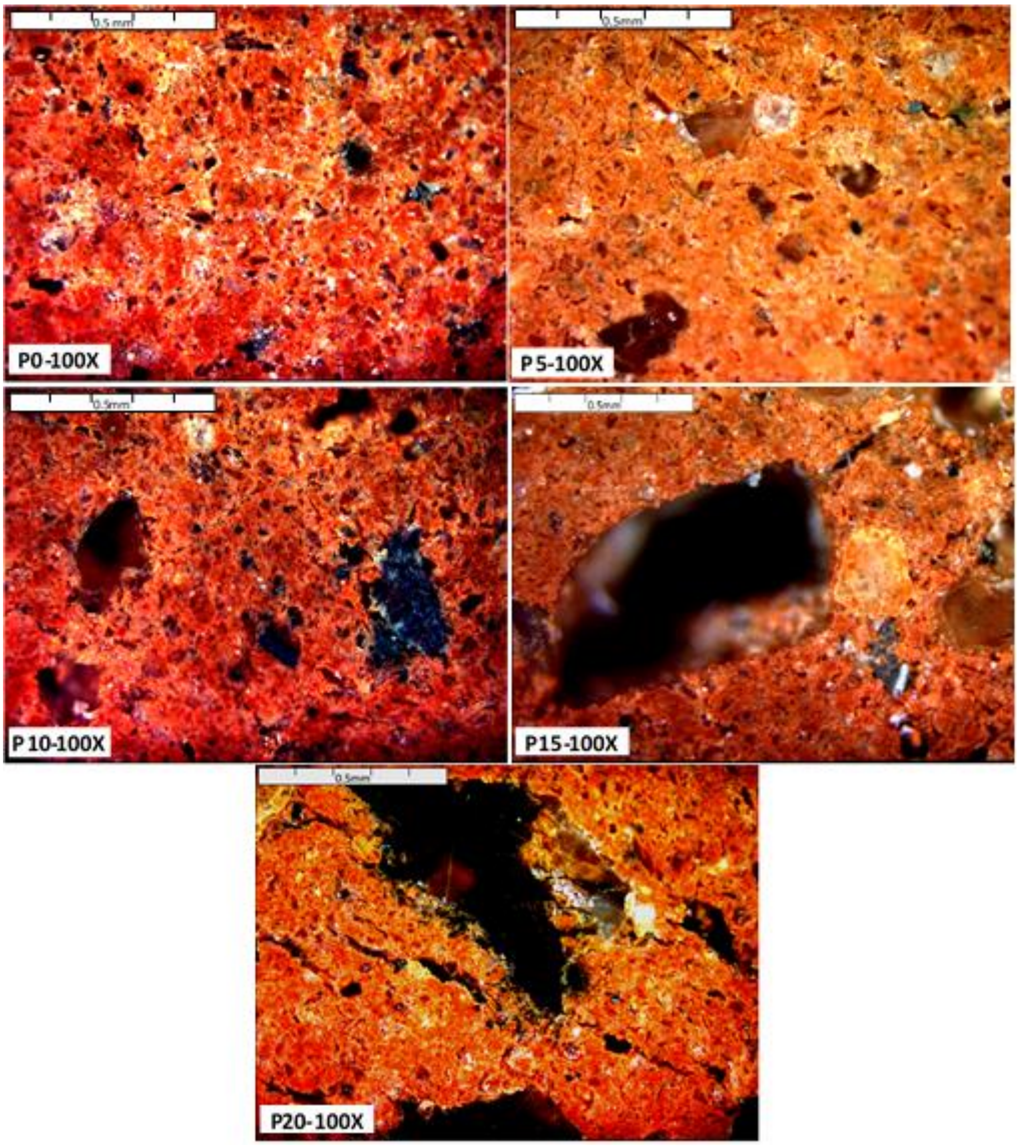

Fig 5. OM images of the samples with different contents of pistachio shells.

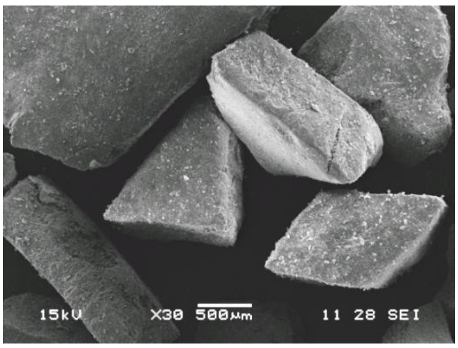

Fig 6. SEM image of ground pistachio shells.

Taking into account that approximately 70% of the ground biomass is constituted of 0.5 mm particles, and that the most abundant pore size observed is 0.3 mm, it can be inferred that during brick firing, the biomass particles that combust generate pores of sizes corresponding to 60% of the size of the particles that give rise to them.

In the samples with higher percentage of pistachio shells, P15 and P20, the pores size is greater than the particles size used for this residue, observing pores of up to 2 mm. The shape of these pores is irregular and elongated compared with those observed in P5 and P10. This would be indicating that during mixing with the clay, and due to the high percentage of residue added, the biomass particles agglomerate, distributed in a non-uniform way, generating pores corresponding to these agglomerates during the firing process.

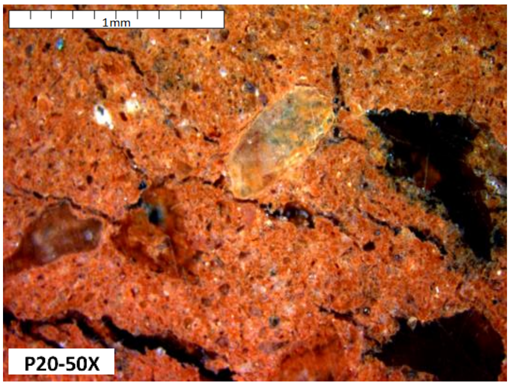

Fig. 7. SEM image showing microcracks presence in P20 sample.

It is also possible to observe in sample P20, the presence of numerous internal microcracks in the structure of the brick matrix, which would explain the increase in flexural strength observed in relation to P15 sample, because these microcracks may initially be acting as stress absorbers. In sample P15 microcracks can also be observed in the matrix, although they are smaller and in less quantity. Figure 7 shows a micrograph of P20 where these microcraks can be observed.

Some studies on pore-forming materials in ceramics found in the literature (Feng et al, 2013; Sarikaya & Dogan, 2013), mention the relationship between the shape of the aggregated particles and the pores formed, but they are generally organic compounds produced by chemical synthesis, with well-defined homogeneous characteristics, and not from residual materials as in this case. Studies such as the one in this work where the pore former is a ground biomass, that is, where the particles have a preferential shape according to their structure, have not been found in the literature.

The pistachio shell biomass used in this work is considered to be of medium density. Pelozo et al. (2022) have carried out a comparative study of ceramic bricks produced with 10% volume aggregates of different biomasses (corncob, peanut shells, pistachio shells, olive pits, etc.), using similar particle sizes, the same heat treatment, and general experimental conditions. The results indicated that the porosity of the samples is directly related to the density of the biomass used, and therefore, the other properties of the bricks are also influenced by this parameter.

Taking into account the obtained results, for pistachio shells under the conditions described in this research, the proportion of added biomass that ensures the use of the greatest amount possible while maintaining the macroscopic characteristics and properties of the ceramic products within those market requirements was 10% by volume.

4. Conclusions

Based on the analysis of the results obtained during the development of this study, and taking into account the characterizations of the used materials, it is possible to conclude that the use of pistachio shells, as a pore former in the ceramic industry is feasible.

The proportion of added biomass that generates the best characteristics and properties in the final product is 10%. At this percentage of aggregate, significant quantities of residual materials are used, and also ceramic pieces with very good macroscopic and microscopic characteristics are obtained, with porosity and flexural strength values that meet market requirements for this type of product.

A detailed analysis of the shape and size of the formed pores has been carried out using an optical microscope. It has been observed that for contents of 5 and 10% biomass, the shape of the pores is similar to the shape of the particles of ground pistachio shells, while for the higher contents (15 and 20%), it is much longer. On the other hand, the size of the pores at lower contents is around 60% of the original size of the particles, and for the higher percentages of biomass, they are much larger. This would indicate that in this case of higher contents, agglomerates of particles are formed and when combusted, lead to that porosity characteristics.

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