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BENEFITS OF USING MORINGA OLEIFERA ASH AS A RAW MATERIAL IN THE MANUFACTURE OF CERAMIC BUILDING MATERIALS

BENEFICIOS DE LA UTILIZACIÓN DE CENIZA DE MORINGA OLEIFERA COMO MATERIA PRIMA EN LA FABRICACIÓN DE MATERIALES CERÁMICOS DE CONSTRUCCIÓN

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Abstract: The purpose of this work has been to explore how beneficial is the use of moringa ash as a substitute material for clay in the manufacture of ceramics for masonry. For this, ceramic pastes with 0%, 4.5% and 9.0% ash were formed, which were moistened and subsequently subjected to a molding process using the extrusion technique. After a process of natural drying of the specimens, they were fired at 800°C, 900°C and 1000°C in a heating oven with electrical resistance. The research process included the characterization of the raw materials using techniques such as X-ray diffraction and fluorescence (XRD/XRF), as well as Fourier Transform Infrared Spectroscopy (FTIR). The characterization of the finished product was carried out by physical-ceramic analysis associated with dimensional changes in the material, density and open porosity, as well as characterization by scanning electron microscopy (SEM) and XRD. The results obtained show that moringa ash is chemically rich in potassium (26.29%) and calcium (26.03%) with a significant amount of sulfur (3.15%) and chlorine (2.20%) being present these elements both in amorphous fraction (80.1%) and crystalline (calcite, arcanite and sylvite). In relation to the ceramics obtained, it was identified that the addition of moringa favors the sintering and vitrification processes in a better way with respect to the formulation with only clay, this as the firing temperature increases and despite the existence of carbonate phases and residual organic matter in the ash. It is possible to obtain a ceramic 11% less heavy than the reference material.

Keywords: Ceramic process, ceramic flux, vitrification, substitute material, mineralogical characterization.

Resumen: La finalidad del presente trabajo ha sido explorar si resulta beneficioso el uso de la ceniza de la moringa como material sustituto de arcilla en la fabricación de cerámicos para mampostería. Para ello se conformaron pastas cerámicas con 0%, 4,5% y 9,0% de ceniza, las cuales fueron humectadas y posteriormente sometidas a proceso de moldeo mediante la técnica de extrusión. Luego de un proceso de secado natural de las probetas se realizó cocción de estas a 800°C, 900°C y 1000°C en un horno de calentamiento con resistencia eléctrica. El proceso de investigación incluyó la caracterización de las materias primas mediante técnicas como la difracción y la fluorescencia de rayos X (DRX/FRX), así como espectroscopia de infrarrojos con transformada de Fourier (FTIR). La caracterización del producto terminado se realizó mediante análisis físico-cerámico asociado a cambios dimensionales en el material, porosidad abierta, así como caracterización mediante microscopia electrónica de barrido (SEM) y DRX. Los resultados obtenidos dejan ver que la ceniza de moringa es químicamente rica en potasio (26,29%) y calcio (26,03%) con una cantidad importante de azufre (3,15%) y cloro (2,20%) estando presente estos elementos tanto en fracción amorfa (80,1%) como cristalina (calcita, arcanita y silvita). Con relación a los cerámicos obtenidos se identificó que la adición de moringa favorece los procesos de sinterización y vitrificación de mejor manera respecto a la formulación con solo arcilla, esto a medida que se incrementa la temperatura de cocción y a pesar de la existencia de fases carbonatos y materia orgánica residual en la ceniza. Se logra obtener un cerámico 11% menos pesado que el material de referencia.

Palabras clave: Proceso cerámico, fundentes, vitrificación, material sustituto, caracterización mineralógica.

1. INTRODUCTION

Moringa oleifera is a species of tree native to India, but it can be found in many parts of the world especially in tropical zone countries, being very common its presence in Latin American territories such as Mexico, Colombia, Ecuador and Venezuela (Abdull, Ibrahim, & Kntayya, 2014; Mahmood, Mugal, & Haq, 2010).

When performing a literature review (Bonal, Rivera, & Bolívar, 2012; Martín et al., 2013; Doménech, Durango, & Ros, 2017; González, 2018) it was found that different authors in their research describe the benefits of moringa. This species is used as a supplement in the feeding of different farm animals, as well as in medicines and in the cleaning of water. Also, the authors show that this plant has a high content of proteins and vitamins in each of its parts so that oil is extracted from the seeds, the trunk is appropriate in the adsorption of heavy metals and in the manufacture of ropes and carpets, seed husks function as the primary material for the production of activated carbon and anionic exchangers. Moreover, moringa is beneficial as fertilizer, cleaning agent, biological fuel, pesticide and is being incorporated as an ingredient within meat products and breads as a preservative and antioxidant additive with very good results without affecting the sensory characteristics of the final product.

Another relevant aspect of the oleiferous moringa is that it can grow in conditions of water insufficiency and is of agile growth (Abdull, Ibrahim, & Kntayya, 2014; Mahmood, Mugal, & Haq, 2010), In addition, it can be sown in almost all tropical, semi-arid and subtropical regions. These characteristics have allowed its cultivation in Colombia in territories such as Bolivar, Tolima, Meta and Antioquia (Castro, 2013), but its presence has also become widespread in the urban areas of cities as often happens in the metropolitan area of Cúcuta (Colombia), where a large number of specimens are evidenced on platforms, parks and inside homes (Gelves et al., 2021).

During the cultivation of moringa is usually pruned at the stage of growth of the species, in order to improve the yields in the production of leaves, a fact that brings the generation of a significant amount of plant residue (Navarro, 2015). Similarly, in urban areas, this type of tree is continuously pruned either for aesthetic reasons or due to the need for maintenance of electrical networks, which also causes a significant amount of plant biomass waste (Gelves et al., 2021).

Many of these pruning residues have no specific applicability, however, their use in residential combustion processes is evidenced, but it could also be used in the generation of thermal energy through combustion in some companies that require heat production for their processes, as some industrial laundries are doing in the city of Cúcuta (Colombia) for steam generation in boilers. The result of this combustion activity is usually the inorganic material commonly known as ash, which also has problems for its final disposition, due to its high basicity.

When reviewing the literature (Abdull, Ibrahim, & Kntayya, 2014; Mahmood, Mugal, & Haq, 2010; Bonal, Rivera, & Bolívar, 2012; Martín et al., 2013; Doménech, Durango, & Ros, 2017; González, 2018; Castro, 2013) Many studies showed that moringa is a very striking species due to the characteristics of each of its parts. However, there was no evidence of information associated with the characterization of their ashes or their possible uses in the industry. For this reason, this research aims to respond to the approach on the probable benefits of the use of moringa oleifera ash as a raw material in the manufacture of ceramic construction materials as a new contribution to knowledge.

2. METODOLOGY

2.1 Materials

Two types of materials were used in this work. The first is a clay mineral traditionally used in the production of ceramics in the metropolitan area of Cúcuta, which comes from the sector La Alejandra municipality of Zulia, Norte de Santander associated with the geological formation Grupo Guayabo (Flórez, Sánchez, & Blanco, 2018). Likewise, moringa ash was used which was obtained from the traditional combustion of pruning residue, which was provided by a company in the city of Cúcuta (Colombia) in charge of this work through the streets of the city. Materials were characterized by DRX, FRX and FTIR in the case of ash.

2.2 Methods

Moringa (tree pruning) material collected was spread and dried outdoors for 15 days. It was also burned outdoors in order to obtain the ashes. Moreover, the clay used as raw material was previously crushed in an industrial hammer mill. Both raw materials were sieved by 2mm mesh before mixing and homogenization.

The materials were chemically characterized by the FRX technique, performed in a sequential 4KW dispersive wavelength X-ray fluorescence spectrometer by Bruker model S8 Tiger and analyzed by means of the Quant-analysis methodExpress and Spectra software; structural characterization was performed using DRX on a Bruker dust diffractometer model D8 Advance with DaVinci geometry at 40kV and 40 mA conditions, with a measurement range between 3,5° and 70° and a counting time per step of 0,6 seconds. In addition to the DRX, FTIR analysis was carried out for the ash, executed on a Shimadzu model UH-50A equipment in the spectral range between 400 and 4,000 cm-1, 48 scan and 4 cm-1 resolution in transmittance mode, using a mixture of potassium bromide (KBr) dry and ash (through mesh 75 µm). On the other hand, morphological aspects were studied using scanning electron microscopy (SEM), in a model JEOL JSM-6490LV. A gold coating system was used by reference sputtering Denton vacuum desk IV.

For the manufacture of the ceramic specimens, three types of paste specified in Table 1 were formulated.

Table 1: Formulation of ceramic pastes

Material	Paste 1	Paste 2	Paste 3
	P1	P2	P3
Clay (%)	100,0	95,5	91,0
Ash (%)	0,0	4,5	9,0
Wetting water (%)	20,0	25,0	28,0

Source: authors' elaboration.

Each of the pastes was formed with tablets 12.0 cm long, 5.0 cm wide and 1.0 cm high, by means of the extrusion technique by means of a manual screw machine without end and vacuum. After this process, the tablets were weighed and left outdoors for 48 hours. They were weighed again and proceeded to dry them in a drying stove of 64 liters of capacity brand Construmetálicas, where they initially remained for three hours at 65°C to then move to a temperature of 120 °C for twelve more

hours. When leaving the stove, the shrinkage and weight loss measurement was performed during drying, following the parameters of ASTM C326-03 (ASTM International, 2003).
The test pieces of each of the three pastes were grouped to be cooked at temperatures 800°C, 900°C and 1000°C. This process was performed in a heating oven with electrical resistance brand Construmetálicas with a capacity of 27 liters, where the tablets went from room temperature to the assigned temperature (800°C, 900°C or 1000°C) by means of three ramps, the first at a speed of 5°C/min to 500 degrees Celsius, the second at a speed of 2°C/min to reach 600 degrees Celsius, and finally, the third ramp again with a speed of 5°C/min to achieve the remaining degrees for the predetermined temperatures.

The cooked tablets were again calculated for shrinkage and weight loss after cooking, according to the guidelines of ASTM C326-03 (ASTM International, 2003). After the cooking process, the percentage of water absorption was determined according to the guidelines of NTC 4321-3 (ICONTEC, 2005). The analyses were completed with the characterization of the finished product using SEM and DRX techniques, making use of the same equipment mentioned above.

3. RESULTS AND CONSLUSIONS

3.1 Characterization of raw materials

This section presents the characterization of the two materials, a process carried out by means of diffraction and fluorescence analysis of rays - X (see Table 2 and Table 3). Similarly, Figure 1 presents the FTIR spectrum of moringa ash.

Table 2: Structural characterization of raw materials (Rietveld refining, % by weight)

Phase	Moringa ash	Arcilla
Arcanite (K_2SO_4)	3,9%	-
Calcite, Mg ($CaCO_3$)	0,9%	-
Calcite ($CaCO_3$)	9,4%	-
Dolomite $CaMg(CO_3)_2$	1,0%	-
Fairchildite $K_2Ca(CO_3)_2$	0,9%	-
Hidroxiapatite, Si $Ca_5(PO_4)_3(OH)$	0,6%	-
Kutnohorite $CaMn^{2+}(CO_3)_2$	0,4%	-
Periclase MgO	0,9%	-
Quartz SiO_2	0,7%	34,3%
Silvite KCl	1,2%	-
Iron and Nickel Phosphate $FePO_4$, $Ni_3(PO_4)_2$	0,1%	-
Kaolinite – 1A $Al_2Si_2O_5(OH)_4$	-	13,4%
Muscovite - 2M1 $KAl_2(Si_3Al)O_{10}(OH,F)_2$	-	6,7%
Illite $(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}$ $[(OH)_2,(H_2O)]$	-	1,7%
Microcline $KAlSi_3O_8$	-	2,9%
Hematite Fe_2O_3	-	0,8%
Amorphous	80,1%	40,2%

Source: authors' elaboration.

Table 3: Chemical characterization (FRX, % weight)

Oxide	Moringa ash	Clay
K_2O	25,34%	1,58%
CaO	29,14%	0,62%
P_2O_5	9,30%	0,20%
MgO	6,02%	0,68%
SO_3	6,30%	0,02%
Cl	1,76%	-
SiO_2	1,02%	62,62%
Na_2O	0,42%	0,20%
Fe_2O_3	0,26%	5,05%
BaO	-	0,04%
ZrO_2	-	0,04%
Al_2O_3	0,20%	20,10%
SrO	0,09%	0,02%
ZnO	0,06%	0,03%
MnO	0,04%	0,03%
CuO	0,02%	0,02%
TiO_2	0,02%	0,89%
Lost calcination	20%	7,84%

Source: authors' elaboration.

From Tables 2 and 3 it can be seen that moringa ash contains a high percentage of potassium, part of this element is present in the crystalline phases of arcanite and silvite, however, it is very likely that most of it is in the amorphous fraction. Something similar happens with calcium, second element with greater presence in the ash, which is associated with the crystalline phases of calcite, dolomite, fairchildite,

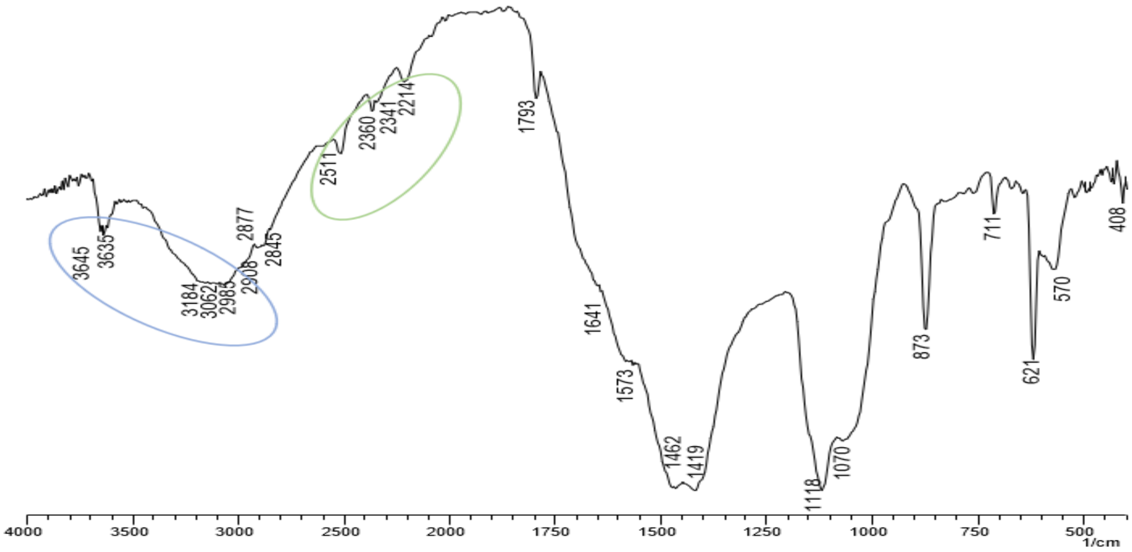

Fig. 1. FTIR spectrum of moringa ash. Source: authors' elaboration.

hydroxyapatite, kutnohorite and in the amorphous phase. The FTIR analysis is complementary to the structural characterization by DRX, it is usually useful to identify amorphous phases and corroborate the existence of crystalline phases. In the spectrum of Figure 1 characteristic calcite bands are evidenced in 1419 cm-1, 1462 cm-1, 1793 cm-1; also, characteristic vibration bands of the arcanite in 621cm^{1} and 1118cm^{1}; hydroxyapatite at 570cm^{1} and 1070cmpositions in accordance with the DRX analysis presented in Table 2 (Chukanov, 2013). Additionally, other phases present in the ash were identified that could not be evidenced by DRX , highlights the portlandite with its characteristic vibration bands in 3645cm^{1} and 428cm^{1}; the vibrations highlighted in green in Figure 1, from 2214 cm^{1} to 2511 cm^{1}, appear to be associated with the presence of residual carbon bonds that are likely to be given by the organic material still present in the ash after combustion is completed, on the basis of their existence as reflected by the losses of calcination shown in Table 3 and the colour of the ash. On the other hand, the vibrations indicated in the blue area (Figure 1) can be associated with the presence of OH groups in the material, the strength of the signal would indicate that there is a considerable value of these links that are different from those of the portlandita, which leads to suggest the presence of other hydroxide compounds within the amorphous phase that were not fully identified. In addition, it is important to highlight the amount of phosphorus, present as iron phosphate and hydroxyapatite; magnesium whose presence can be associated with the crystalline phases of dolomite and periclase; sulfur presented as arcanite and chlorine incorporated as silvite.

According to Fernández (Fernández, 2000) the alkaline oxide content determines the temperature necessary to achieve a certain degree of firing of the part, that is, the desired mechanical strength and porosity". According to the above, in clays whose percentages of alkaline oxides do not reach 1% the firing of the brick is usually between 1.100 and 1.200 ºC; while the clays with high content of Na O and K O the cooking temperature would be within 825 and 850 °C (Fernández, 2000). That is, that potassium seems to be beneficial to lower the cooking temperature of ceramic tablets which could reduce fuel consumption and cooking times, This fact makes interesting the use of moringa ash as a possible substitute material for clay in the manufacture of construction ceramics.

Chlorine (2.20%) and sulphur (3.15%) present in ash are negative aspects of this raw material for use in the ceramic industry. In the case of chlorine, with the increase in temperature for ceramic firing, silvite (KCl) can be broken down by releasing the chlorine present, which could react with water vapor and hydroxyl species from the clays and end up releasing HCl as a contaminant to the atmosphere (Fernández, 2000). With regard to sulfur, its high presence in the ash can bring two effects on the ceramic process, the first of which is the release of this sulfur as a gas at high temperature (formation of SOx), components that upon reaching the atmosphere will end up forming acid rain (Garcés & Hernández, 2004). The second aspect is that the sulfur that is as sulfate in the ash remains in the solid fraction (ceramic) and that, due to the high calcium content in this material, ends up forming calcium sulfate, which is responsible for the appearance of white spots on the surface of the ceramic (plaster efflorescence) even in low concentrations (Cáceres, Molina, & García, 2015; Gazulla et al., 2010). From this result it was considered that only clay should be replaced by ash in ceramic pastes in values lower than 10%.

When observing the characterization data concerning clay, it can be analyzed that, according to Tables 2 and 3, it is a material with high silicon content associated with the presence of crystalline phases of quartz, kaolinite, Muscovite, ilite, microclin and even in the amorphous phase which accounts for 40%. Aluminum is the second element with greater presence in the clay material, this can be found in the same phases mentioned above except quartz. According to the literature a high quartz content decreases the percentage of moisture in the mixture and drying contraction, also increases the porosity of the dry and cooked piece, and improves impact resistance (Fernández, 2000); these characteristics are beneficial in drying, but in the firing process quartz hinders the sintering and vitrification of the part causing inconsistencies in mechanical strength tests; in addition, if the cooking temperatures are not adequate, cracks can be generated inside the material (Verduch, 1974), so it is of special care these variables in clay materials with high quartz content, therefore the addition of moringa ash (with low silicon content) as a substitute would have positive effects on the design of ceramic pastes. As for the results of DRX, the clay material is characterized by having a relevant percentage of kaolinite and muscovite, these materials are beneficial in the ceramic process as they decompose at temperatures below 1000°C and favor the formation of glass phase and crystalline phases beneficial for the mechanical resistance of ceramics such as mullite (Álvarez, Sánchez, & Gelves, 2017), that is why its use should also be privileged and not exceed the substitution of clay for ash which does not possess these phases.

3.2 Physical-ceramic analysis

From the monitoring of the ceramic process, it was possible to establish the percentage of drying shrinkage (% C.S) and firing (% C.C), the percentage of weight loss in drying (% P.P.S) and cooking (% P.P.C), together with the percentage of absorption (% ABS) results shown in Tables 4 and 5.

Table 4: Drying ceramic physical analysis

Paste	%C.S	%P.P.S
P1	8,47	18,10
P2	6,67	21,37
P3	7,22	22,66

Source: authors' elaboration.

Table 5: Physical ceramic analysis in cooking

Temperature	Analysis variable	P1	P2	P3
800°C	%C.C	0,18	0,63	0,45
	%P.P.C	5,63	6,20	7,64
	%ABS	12,72	17,96	20,42
900°C	%C.C	0,18	0,76	0,90
	%P.P.C	6,10	5,79	8,48
	%ABS	12,03	15,28	16,94
1000°C	%C.C	1,05	0,93	1,34
	%P.P.C	6,10	6,80	8,81
	%ABS	10,49	14,01	11,60

Source: authors' elaboration.

According to the information in Table 4, it can be evidenced that the highest percentage of drying contraction is given by tablets made with only clay (P1), this data is interesting starting from the fact that the wetting of the pastes with ash required a higher water content (see Table 1), with which it would be expected more contraction in P1 and P2. One might think, based on the amount of water removed at the drying stage (Table 4), that this substance reacted with ash to form hydrated potassium and carbon compounds. This reduction in shrinkage results in a positive aspect for the use of ash as a substitute material since it is possible to have greater dimensional control (tolerances) during the manufacturing process of the ceramic product on an industrial scale, This could be reflected in the decrease in the number of non-compliant products in references such as floors and/or wall coverings.

In relation to the cooking process a greater contraction is evident in the test pieces made with ash, being more marked when there is greater clay substitution. The explanation for this behavior could be associated with a greater sintering and vitrification of the ceramic product of the higher potassium content in the pastes that acts as a flux element as previously mentioned. This statement is supported by the evolution of open porosity, associated with the measurement of water absorption percentage, where it is noticeable that there is a more marked decrease in the value of this technological property in ash ceramics as the cooking temperature increases. Similarly, this statement is supported by the electron microscopy results presented in section 3.3. At 1000°C cooking temperature, P2 and P3 show significant differences with regard to water absorption, this again highlights the role of potassium as a flux element. When comparing the water absorption of P1 and P3 at this last temperature, very similar values are evident, a fact that could be misinterpreted as not achieving improvements by adding ash, however it is important to note that the pastes with ash presence in this case 9% in P3, have included residual organic matter and have carbonate species that decompose in high temperature, which contributes to the formation of greater porosity with respect to the material with only clay as evidenced in the microphotography in Figure 2. On the residual organic matter present in the ash, it could be thought that, if there is a good oxidation in the oven and there is a good diffusion of oxygen inside the ceramic specimens, energy inputs to the system would be obtained that would make it possible to reduce energy consumption during the cooking stage, thus giving greater relevance to the use of ash as a substitute material.

3.3 Characterization of the finished product

In this section, pastas P1 and P3 were taken as reference, due to the results obtained in the ceramic physical analysis. These materials were characterized by X-ray diffraction and scanning electron microscopy. The results are presented in Tables 6 and Figure 2.

Table 6: Structural characterization finished product ((Rietveld refinement, % by weight)

Fase	**800°C**		**900°C**		**1000°C**	
	P1	**P3**	**P1**	**P3**	**P1**	**P3**
Cuarzo	61,0	56,6	59,8	60,8	63,1	61,5
Moscovita	8,6	10,9	7,4	5,5	2,0	1,4
Hematita	2,9	2,5	2,8	2,8	2,4	2,0
Microclina	-	-	-		<1,0	<1,0
Albita	-	-	-	-	-	1,1
Amorfo	27,5	30,1	29,9	30,9	31,6	33,5

Source: authors' elaboration.

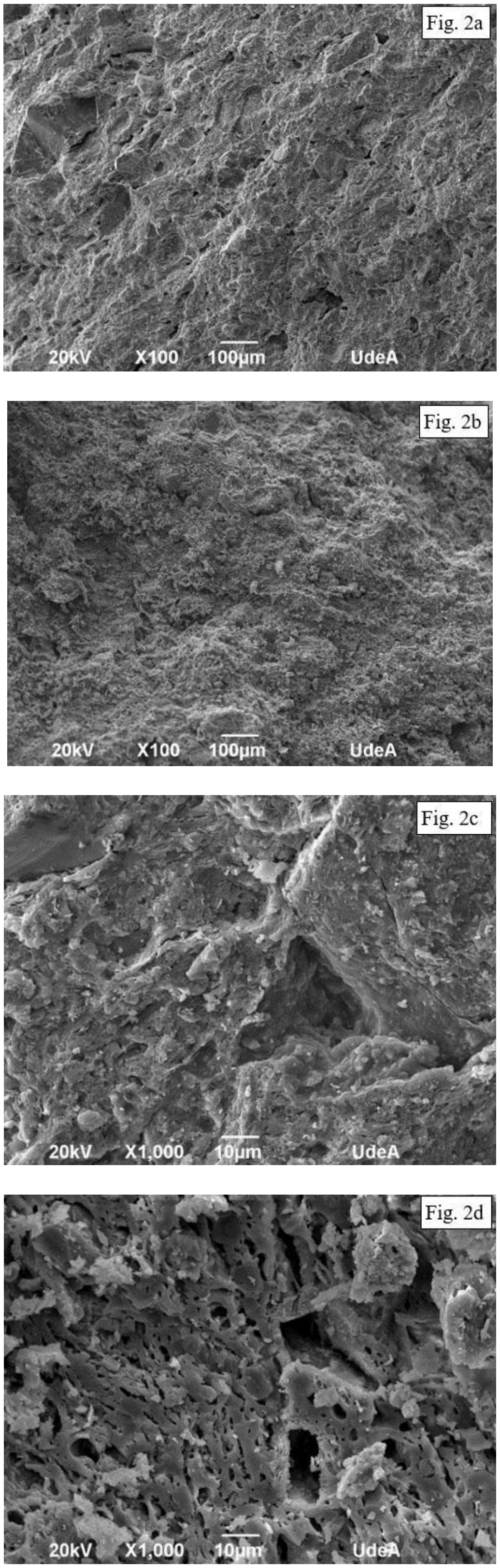

Fig. 2. Scanning electron microscopy finished product. Source: authors' elaboration.

Monitoring the evolution of the microstructure as the temperature increases (Table 6) reveals aspects such as the low reactivity of quartz in the study temperatures, the increase in the amorphous fraction in both materials as the cooking temperature rises, associated with the formation of glass phase by the decomposition of kaolinite and muscovite. Table 6 shows the evolution of this last phase (continuous decrease) with the temperature increase. Amorphous phase formation is slightly higher in P3, which may be due to the effect of potassium as a flux agent. It is also evident that increasing the temperature begins the formation of feldspars especially in the ceramic material with the presence of ash.

With regard to the surface analysis by scanning electron microscopy, the results show significant differences between ceramics made with P1 and P3 at the cooking temperature of 1000°C. At a magnification of only 100X (Figure 1a and Figure 1b) higher porosity is seen in the P1 ceramic, however, increasing the magnification to 1000X ((Figure 1c and Figure 1d) shows another completely different situation. In the case of ceramic P1 (Figure 1c) or clay only, a surface with less pores is visible and vitrification is not so marked, still observing the border between grains in several areas of the image. In the case of ceramics with 9% ash (Figure 1d), there is evidence of a high porosity of varying size (between 1 and 10 micrometers), between a glass matrix that is quite evident to the naked eye (possibly caused by the higher potassium content), with some grains still not sintered. The explanation to this porosity, as already explained, responds to the spaces left by the oxidation of the residual organic matter still present in the moringa ash and to the decomposition of the carbonates identified by DRX that release carbon dioxide.

On the other hand, as an interesting fact it was possible to determine that the tablets with ash are lighter than those of clay only, in the case of tablets with 4.5% of ash it was found that they are 7% less heavy than those with clay only; while tablets with 9% ash are 11.1% lighter, this is probably due to the decomposition of organic matter and carbonates as mentioned above. This can be beneficial because it would be possible to make lighter buildings and allow the transport of more material in freight services between cities.

As a negative aspect it was evidenced that the ceramic specimens made with moringa ash present efflorescence on their surface that, as mentioned above, is caused by the presence of sulfates especially calcium. This would result in limitations on the use of this material in applications where the product needs to be used as a facade or as a ceiling covering, as these stains are considered aesthetic defects and would detract from the commercial value of the finished product.

4. CONCLUSIONS

Moringa ash, due to its high potassium content, has an important role in the manufacture of ceramic construction materials as evidenced in the absorption results and scanning electron microscopy, because they contribute to the sintering and vitrification of ceramics.

The high sulphur content, despite using low ash concentrations in the pastes, generates efflorescence which limits the use of the ceramic product in aesthetic applications; However, the ash would be useful to elaborate elements of enclosures such as blocks and solid bricks which are then coated with filling and paste.

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