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Effect of wet grinding on the physical-mechanical and structural properties of extruded ceramic tiles

Efecto de la molienda vía húmeda en las propiedades físico-mecánicas y estructurales de baldosas cerámicas extruidas

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Abstract:

Regionally, ceramic companies are limited to characterizing raw materials, without taking into account the grinding methods and forming techniques, which will influence the final properties of the finished product. This project evaluated the integral benefits of an extruded clay raw material within the hive furnace firing stage, after having carried out the conversion in the preparation method. The viability of industrially producing extruded ceramic floor and wall tiles was established, using wet milling, and the advantages of this milling were shown, achieving an integral benefit, among the best use of resources, the increase in quality and a maximum of product profitability.

Keywords: Grinding, extrusion, hive furnace, oxidant, reducing.

Resumen

Regionalmente, las empresas cerámicas se limitan a realizar la caracterización de las materias primas, sin tener en cuenta los métodos de molienda y técnicas de conformado, las cuales tendrán influencia en las propiedades finales del producto terminado. Este artículo, evalúa los beneficios integrales de una materia prima arcillosa extruida dentro de la etapa de cocción en horno colmena, despues de haber realizado la reconversión en el método de preparación. Se estableció la viabilidad de producir industrialmente pavimentos y revestimientos cerámicos extruidos, utilizando la molienda en húmedo, y se muestra las ventajas de esta molienda, logrando un beneficio integral, entre el mejor aprovechamiento de los recursos, el incremento en la calidad y un máximo de rentabilidad de los productos.

Palabras claves: Molienda, extrusión, horno colmena, oxidante, reductora

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Introductión

The ceramic sector has an important weight in the economy of Norte de Santander, especially in the Cúcuta Metropolitan Area, being one of the most representative economic sectors, as it is not only relevant in the generation of jobs but also is key for the economic development of the region. This is corroborated by the percentage share that it has in GDP, contributing around 11.1% [1]-[3]. This sector has long stood out in the construction market at a regional, national and international level for the quality of its materials, in addition, it has been characterized by the use of techniques such as pressing and extrusion to shape the different products, the latter technique being the most widely used, showed by the fact that 90% of companies use this process to produce ceramic flooring and masonry products, compared to 9% of companies engaged in pottery and the remaining 1% corresponds to a single company that works pressing forming, which uses wet grinding for its preparation stage [4]-[6].

Despite the fact that the percentage is low in this process, it is worth mentioning that in the region the only company that uses pressing as shaped, covers around the 90% of regional exports and is part of the four largest ceramic companies in Colombia which exports 80% of construction materials [7]-[8]. This could be mainly attributed to the technical and aesthetic quality that its products acquire in the manufacturing process and one might think that these results are inherent to the process, in which that uses atomized powders and a compaction pressure in the preparation stage which has an essential influence on the improvement of these properties. Even the bibliography, mentions in various investigations carried out in the region, that this type of process benefits the manufacture of ceramic materials [9]-[12].

However, the group of companies that make up the other 10%, which are mostly engaged in extrusion processes, and struggle daily to export their products abroad are being affected because the quality of their products has deteriorated markedly as time goes by and they have gradually lost the prestige they have gained. Most attribute these problems to the manufacturing processes themselves and to the poor controls that are exerted on each stage of the process, others differ that the low quality of the extruded products compared to the pressed products, is mainly due to the processes carried out in the stages of raw material preparation, being more effective in pressing processes where wet milling is used [13]-[14]. For this reason, this research aims to demonstrate that, by wet grinding at the preparation stage in extrusion processes, and by achieving a reconversion at this stage, structural and mechanical properties can be improved and the market not only at the level of potential national but international.

This reconversion in the preparation stage can generate variable profitability in extrusion companies, as it is known that the wet process has higher costs, among these are energy costs with increases of up to 20%, equipment costs and operating costs, but at the same time, it stands out that wet grinding improves subsequent processes, mainly drying and firing processes, because in this process the resulting particles are finer, generating a greater specific area, which helps to reduce firing temperatures, increase the firing density, which achieves less porous and higher resistance products, as opposed to dry milling, which produces thicker particles, difficult to process, a lower specific area, materials more porous and less resistant, in addition to the materials formed by dry grinding, require a firing temperature higher. Likewise, from a technological point of view, the fact of not achieving finer grain sizes means that the products manufactured are not those indicated for gresifiable processes. Furthermore, this type of grinding is not suitable for when it is required to grind hard pastes composed of different materials [14]-[15].

Although apparently this reconversion is not economically viable, and has the aforementioned limitations, and research on the subject is almost nil. With this work, the physical-mechanical and structural behavior of an extruded and milled ceramic product in the wet process was evaluated, and it was determined not only the technological benefits that the reconversion in the raw material preparation stage entails, but also that it is economically sustainable for this type of industrial process. Managing to establish, the viability of industrially producing extruded ceramic pavements and coatings through wet grinding, and achieving an integral benefit, between the best use of resources, the increase in quality and maximum profitability of products.

Materials and Methods

For the development of the investigation, several experimental processes were carried out, the first part, the preparation of the raw material, initially consisted of sampling the clay material, this was obtained directly from the mine, located in the municipality of Villa del Rosario (Norte de Santander - Colombia). The clay belongs to the Guayabo formation, it is of illite - kaolinitic typology, reddish in color and of medium plasticity texture.

Sampling was carried out on one of the mine fronts, it was done in a zigzag shape, using a grid drawn on the front plane and its extraction and collection points were made in an ascending path. With the collected material, the moisture content was taken in an Ohaus MB45 thermobalance, to determine the drying time. With the humidity data, the drying process was carried out, this was carried out in a Gabbrielli Technology brand stove at a temperature of 110±5 ºC for 24 h until obtain a humidity of less than 10%. After the material was dried, a manual pre-crushing was carried out, bringing it to an ASTM 5 mesh through particle size. With the homogeneous material, it was separated by quartering using the cone stacking technique, until obtaining a quantity of 50 kg for each one of the millings (wet and dry), as well as material for X-ray diffraction analysis, which was performed using a Bruker model D8 Advance powder diffractometer with DaVinci geometry, and a quantitative analysis of oxides performed by fluorescence X-ray, using a Panalytica Axios equipment. Dry grinding (MS) was carried out until obtaining a passing particle size of ASTM 10, this was carried out in a fixed head hammer mill, which allows materials with a hardness of up to 7 on the Mohs scale, it has a capacity of production up to 150 kg/h, a speed range between 50 to 6000 rpm and reaches an average particle size of 40 μm (depending on the material). The wet process (MH), was performed in an Alsing mill, with a volume of 100 liters, a speed range of 23 rpm, it has a production of up to 275 kg/h and reaches an average particle size of 74 µm (depending on the material). The two Reinaltech brand teams were on a semi-industrial scale, with the aim of simulating the processes carried out in regional companies.

For the wet process, the slip necessary to carry out the investigation was obtained. It should be borne in mind that for obtaining this material, regionally used parameters were taken into account, such as industrial waste, made in ASTM 230 mesh, which ranged from 3% to 9%; the density, evaluated by means of a pycnometer and with ranges between 1.60 g/cm3 to 1.80 g/cm3 and the viscosity by means of Ford cup No. 4, in a range of 18 s to 25 s, all these ranges are according to the material used which was solely clay. The slip resulting from this stage was deposited in metal trays and dried at a temperature of 110± 5 ºC for 24 h. The dry slip was manually ground in a grinder, normally used to grind coffee; and it was sieved by ASTM 10 mesh. After the two millings, each material was sampled again, 5 kg of each milling were taken and controls were performed, such as a retentate on a sieve in ASTM 230 mesh, a plasticity index by the method of Casagrande and a granulometry by hydrometer 152 H.

Subsequently, after having carried out the characterization of the samples, the process of forming them was developed, which was carried out by means of the extrusion process. The material of each grinding was wetted, using the plasticity index data. After wetting MS and MH, they were left to stand for homogenization for 15 h, in order to guarantee uniform humidity throughout the plastic mass. After 15 h, the forming process was started using the extruder without vacuum. Prismatic specimens in the form of a cladding type of 25 cm × 7 cm × 1 cm were molded. With the shaped specimens, dimensions (length, width and thickness) and mass were measured, they were dried at room temperature for 24 h; they were then brought to the drying oven for a period of 12 h at a temperature of 60±5 ºC. Finally, the temperature was gradually increased by 10 °C/h until reaching a temperature of 110±5 ºC, maintaining it for 24 h. The specimens were dried, dimensions and mass were measured again, one specimen was taken from each grinding and a dilatometric analysis was performed, this was carried out in a DIL 402 EP dilatometer from Netzsch, at a heating rate of 10 ºC/min.

The remaining specimens were taken to the cooking process in a hive oven, following the pre-established parameters and frequently used in this production stage by the company Pisos y Enchapes Margres. This firing stage was carried out at two atmospheres, a firing in an oxidizing atmosphere with a time of 40 h until reaching final temperatures of 950 °C, 1000 °C and 1050 °C, maintaining the final temperature for 4 hours, with the aim to clean any coal deposit and avoid any defect, then the cooling starts. The other firing was carried out in a reducing atmosphere, with a time of 45 h until reaching final temperatures of 975 °C, 1025 °C and 1100 °C, in this firing the reduction time for each temperature was 8 h, 9 h and 10 h respectively, the heating process that was carried out to achieve this atmosphere was basically similar to the oxidizing atmosphere until reaching 900 °C, from then onwards work is started with 3/4 of the closed register in order to start to cover the oven but without losing print run, this is maintained until 975 °C, then the air supply is reduced until it reaches 1050 °C, finally for the temperature of 1100 °C, the same procedure is followed, but after passing 1050 °C, air is no longer injected, feeding the combustion only with coal, in this reduction process, the pants are kept closed, the burners only open to add coal and it is controlled that no black smoke is generated to guarantee the reaction of the carbon monoxide with the clay, finally, the cooling process begins. After firing, dimensions and mass were measured again and the respective analyzes were carried out for the development of the investigation.

The technological aspects of linear shrinkage, mass loss and densification were determined in triplicate using dimensions and mass. In the same way, the technological properties, also in triplicate, were analyzed according to national standards. The structural property was carried out by means of the water absorption test, which evaluates open porosity, a property that is used for the classification of tiles and product specifications. This was developed using the boiling method, leaving the specimens impregnated in boiling water for a time of 2 h, according to NTC 4321-3. The mechanical resistance was carried out by means of the test of resistance to bending at three points, using a pressure press (Crometro) model Flexi-1000 LX-650, from the firm Gabbrielli Technologhy, by applying a loading speed. 1±0.2 N/mm2 per second, according to NTC 4321-4. For each test, 5 specimens were used for each furnace temperature. The quantity of specimens was defined according to the Robert O. Kuehl methodology, taking into account factors such as the furnace atmosphere, the firing temperatures and the types of grinding; it was carried out in this way, since there are no previous studies that talk about this particular topic, therefore, there was no bibliographic information that allowed obtaining more accurate results [16].

Methods

The raw material was prepared for the different characterization tests set out above according to national and international standards NTC 1776, NTC 2401, NTC 3674, NTC 4630, NTC 919, NTC 4321-3, NTC 4321-4 and ASTM D422.

Results and Discussion

As can be seen in Figure 1, the XRD details that the material has its main peaks associated with kaolinite (K) and quartz (Q), the main structures of clay materials. A high intensity of the quartz peaks can be observed, indicating the existence of free silica, which can affect the reactions that occur during preheating. This is confirmed by the high value of 7.20, in the molar ratio (SiO2/Al2O3) and the low loss on ignition (LOI), as shown in Table 1. This is rare in clays from the region, especially in the of the Guayabo formation, since the minerals outcropped there, have excellent properties, the main reason why most companies are located in this area.

Figure 1. DRX of clay raw material (Q. Quartz; K. Kaolinite; H: Hematite; M. Muscovite). Source. Xamtec.

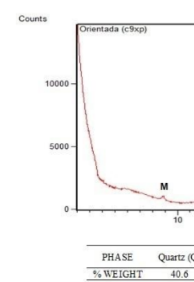

Table 1 shows a low percentage of Al2O3, results that do not agree with previous studies carried out on clay materials from the Guayabo formation in the region, where there is a higher percentage of alumina, as reported by Gelves et al., Diez Contreras et al. y Mora-bastos et al. [12]-[13] y [17]. It also shows a high percentage of silica, being above the average ranges for a clay used in this type of process, which should be between 45% and 55%, as described by Fernández Abajo (2000) "In clay for bricks , the SiO2 content is usually between 45% to 55% " [18]. This suggests the existence of one or more laminar minerals and that of free quartz in variable proportions, which confirms the evaluation in the DRX.

Table 1. Chemical composition of the clayey raw material (FRX). Source. Xamtec.

Oxide	SiO2	Al2O3	Fe2O3	K2O	TiO2	MgO	Na2O	CaO	LoI	SiO/AlO
Mass%	69,80	16.46	4.60	1.43	0.88	0.51	0.13	0.27	5.73	7.20

The Fe2O3 content, foresee the low presence of free iron oxides, in addition, it can be stated that the characteristic color of this material after firing in an oxidizing atmosphere will be a light red, this due to 0.18% of the Fe2O3/Al2O3 ratio, and in a reducing atmosphere it will be beige due to this ratio and its percentage of Ti2O. It is worth noting the presence of micaceous minerals by the data previously evaluated and by the percentage of K2O (1.43%) [19].

According to Table 2, it can be detailed that grinding affects the intrinsic characteristics of the material, as it is observed, in the particles smaller than 63 μm of MH these are reduced by 22.9% compared to MS. If we analyze the wet densification, we can see decreases of up to 10.3% in MH to MS, this is possible because, in MS, the particles are more homogeneous, which generates better packing when they are pushed transversely by the extruder, that is, that for each unit of volume, more particles are accommodated. This homogeneity is attributed to the fact that dry grinding provides 82% of particles less than 500 μm, while wet grinding, the percentage is reduced to 28%, allowing for better performance at the time of extrusion [18]. However, if we analyze the dry density, the behavior changes at this stage, MH increases by 5.9% for MS, this is mainly because wet grinding does not generate homogeneity in particle size, but if a greater uniformity, which allows to increase the specific area and therefore the densification. Although in theory this can be problematic in processes such as drying and firing, due to the elimination of water and other gases as a result of internal reactions, the important thing is that this densification would help to avoid common defects such as deformations in the gauges, or the formation of cracks, frequent problems in regional companies [12], [20].

In figure 2, presents the particle size distribution, the figure shows MH with a higher amount of clay fraction, 37.3%. This is the reason for its increase in plasticity as shown in Table 2. By contrast, MS has the least amount of clay minerals, 30.6%. MH yields a clayey soil texture, and a ceramic aptitude

Table II. Dry and wet technological aspects of MS and MH

Specimens	Retentate on a sieve in ASTM 230	Plasticity index (%)	Wet density (g/cm3)	Dry density (g/cm3)	Dry linear shrinka-ge(%)	Dry mass losses (%)
MS	(%) 10,5±0,2	18,4±0,1	2,036±0,2	2,049±0,5	5,4±0,1	14,8±0,3
MH	8,1±0,4	20,7±0,3	1,826±0,1	2,170±0,4	8,5±0,1	17,8±0,2

ability according to Winkler, for making horizontal-boring bricks and thin-walled pieces. While MS, it yields an equally clayey texture, but with a ceramic aptitude for making shingles, upright brick and tile. This indicates that the preparation process affects the physical characteristics of the material, and although its texture is clayey, a common feature in the clays of the region, by modifying the grinding method, it is possible to theoretically modify the final use of the raw materials. After testing the Casagrande method (Figure 3), it can be inferred that the samples have a medium-high plasticity, since as observed, the MS sample showed a plasticity index of 18.4% and MH of 20.7 %, generating an increase of 12.5%, which indicates that the MH sample has a higher percentage of fine particles, confirming the evaluation of the retentate on the sieve. Despite their plasticity, they will have an acceptable extrusion in both cases, an important aspect when it comes to forming the material.

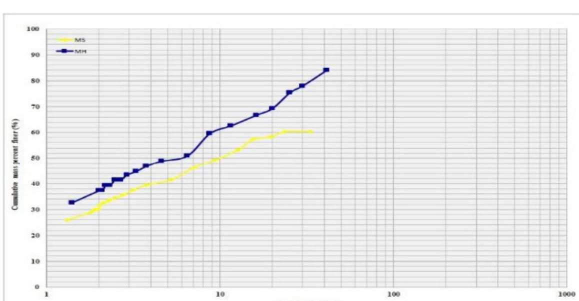

Figure 2. Particle size distribution of MS y MH.

After forming, the behavior of the materials and the effect generated by the preparation process on the internal structure and the packaging of the particles in the specimens were evaluated. As you know, drying is an operation that mainly consists of evaporating the water contained in a molded ceramic product. In the region, 90% of companies use natural drying, not only because of the low cost of the process but also because the Cúcuta Metropolitan Area lends itself to this type of drying, since the average temperature is 30±5 °C, on not so hot days. Reason why, it is necessary that the material preparation processes are the most demanding and thus ensure that the product does not generate problems at this stage. Therefore, technological aspects such as densification, shrinkages and loss of mass must be controlled to achieve an optimal product before entering it for firing. [18] y [21]-[22].

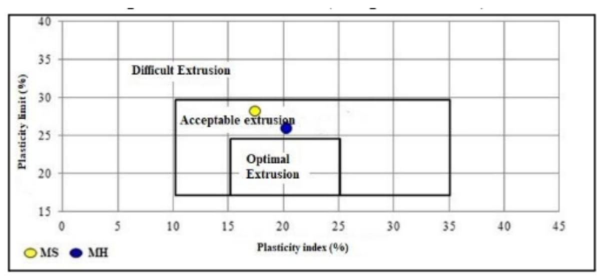

Figure 3. Extrusion forecast (Casagrande method).

In Table 2, the behavior of the specimens after the drying stage was analyzed, as it is observed, MH increases the dry density of the material compared to MS by 5.9%, as it was mentioned already, generating a completely different behavior from the wet density. In the same way, it can be analyzed that the increase in the density from wet to dry is greater in MH (18.84%) than in MS (0.64%). This is because, in shaping, the packing of the particles depends on the size, but in the drying, the variables that affect the process depend fundamentally on the movement of the water molecules within the clay mass and at the speed of migration to the surface, therefore, in the drying, the uniformity of the particle is more important than the size [23]-[24].

For this reason, MH increases the dry density of the material, since it generates greater uniformity of particles, better packaging and better heat transfer in the structure of the material, improving the drying process and reducing time at this stage, this was verified on the duration of the process, where MS lasted 48 h in this process and MH 32 h, reducing drying time by 33.3%, which would generate greater profitability at this stage of the process. This is confirmed by the increase in the dry contraction of MH with respect to MS, which was 57.2%, which indicates that in MH there was greater evaporation of water, greater contact between particles and better drying, increasing the shrinkage. This is important, since drying times will be reduced and it will be guaranteed that the piece enters the oven with the adequate humidity for the firing process, generating less fuel consumption (Coal), less contamination and shorter firing times, mainly in the initial preheating temperatures, where the hygroscopic water of the products evaporates, which is one of the most problematic phases in the firing in hive furance, important aspects in the production processes of regional companies. Another important aspect in the reconversion of the preparation process is that observed in the loss of mass, an increase of 20.3% is observed, confirming a better transfer of heat from the surface of the piece to the center of it.

In the same way as in the wet and dry conditions, in firing, aspects and technological properties of the material were also evaluated. A dilatometric analysis was performed to observe the behavior of MS and MH during firing, densification, linear shrinkage, mass losses, as well as technological properties were evaluated. In figure 4, the thermodilatometric behavior of MS and MH is evaluated.

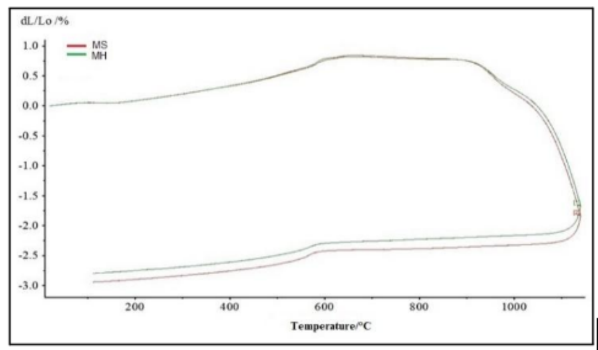

Figure 4. Dilatometric curve. Source. Mintec Ceramic Ltda.

You can see several details to highlight; a low intensity endothermic peak between 100 °C and 200 °C, which is part of the removal of hygroscopic water from the material, it should be noted that this peak is small in both curves, which can be deduced that this clay is poorly rehydratable and will not present tempering problems. However, in the MH curve a reduction of 64.1% is observed in the coefficient of expansion at this temperature, confirming the aforementioned, that wet grinding improves the drying phase and consequently improves the evaporation of this hygroscopic water, improving and accelerating the initial stages of preheating. In the same way, another peak is observed between 500 °C and 600 °C, which refers to the allotropic transformation of quartz (α-β), in the two curves they are presented at the same temperature (582 °C). But in MH, it generates a 30.4% reduction in the coefficient, which will reduce the formation of structural defects and better control at this stage. Another endothermic peak is observed, between 900 °C and 1000 °C, which is part of the decomposition of volatile materials and possibly the beginning of sintering. Finally, it can be observed, a last peak in the cooling, between 500 °C and 600 °C, which refers to the allotropic transformation of quartz (β-α), and as it was seen in the preheating, MH reduces the coefficient dilation. It is noteworthy, that in the MS and MH preheating stage, the total expansion of the two curves is below 1.0%, which are recommended values for clays traditionally used for the manufacture of structural ceramic products, which must be between 1.1% to 1.2% [25].

Table III shows the density values, for MH they increase in both atmospheres, since there is a significant increase in all temperatures. However, it can be seen that the samples reach their maximum densification value at average temperatures, decreasing with the continuous increase in temperature. Although the reduction is not significant, this behavior must be controlled, since the technological properties of the product may be affected. Within the increases it can be detailed that in the oxidizing atmosphere they reach 12.6%, before the decrease, while in the reducing atmosphere the increases are moderate up to 4.9%. This decrease can be attributed to the low percentage of Al2O3 and the percentage of K2O observed in the FRX, which generates the possibility of forming a considerable amount of liquid phase at a relatively lower firing temperature, causing the aforementioned effect, as observed in figure 5.

Table III. Technological aspects of MS and MH in firing.

Specimens	Atmosphere	Temperature	Firing density	Linear shrinkage in firing	Mass Losses in firing
		°C	g/cm³	%	%
MS	OXIDIZING	950	1.796±0,2	0.27±1,2	6.14±1,6
		1000	1.815±0,1	1.08±1,4	6.11±1,7
		1050	1.959±0,1	1.64±0,7	6.03±1,0
	REDUCING	975	1.783±0,3	0.52±0,2	6.28±1,5
		1025	1.948±0,4	2.75±1,5	6.54±0,9
		1100	1.869±0,2	3.32±0,1	6.66±0,6
MH	OXIDIZING	950	1.988±0,2	0.96±0,8	6.51±0,2
		1000	2.043±0,1	1.09±0,6	6.35±1,6
		1050	1.996±0,1	4.36±0,2	6.47±0,5
	REDUCING	975	1.832±0,1	2.45±0,3	6.64±0,2
		1025	2.044±0,1	3.89±1,4	6.88±0,1
		1100	1.970±0,4	3.96±1,1	6.72±0,8

In linear shrinkage, changes in high temperatures can be highlighted, punctually in MH samples, confirming that the sintering mechanism is generated in a better way, which guarantees that the heating of the piece is complete when applying this method of preparation. This can be confirmed with the fact that, as there is a better heat transfer, the internal reactions in the material are complete, generating greater densification and at the same time a greater contraction, as observed in Table 3 . Losses of weight show typical values of clay minerals, with percentages that do not exceed 7%, since the bibliography references that for this type of materials the losses must be between 6% and 12%, below 5% denotes a predominance of inert compounds and between 15% to 20% or more, is caused by the decomposition of calcium carbonate with evolution of CO2. However, it is necessary to highlight the homogeneity in the mass losses, both in MS and in MH, with increases between 3% to 10% as temperature increases [26]-[27].

Finally, the results obtained in the technological properties were compared using the NTC 919 standard, a standard that evaluates coating products for export. This was done, to achieve an equivalence at the regulatory level, with the necessary parameters in terms of water absorption and mechanical resistance, properties normally evaluated in the region for construction products, and thus, demonstrate the advantages and improvements generated in the products when carrying out the conversion in the preparation method, in addition to analyzing if there are changes in regulatory groups between the MH products for MS, as this would help to increase the commercial and technical value of the tiles. As observed in figure 5, the behavior of MH, shows a decrease in its porosity, observed at all temperatures. It is detailed, decreases of up to 7.3% in an oxidizing atmosphere and up to 10.7% in a reducing atmosphere, which shows better heat transfer in the internal structure of the products, facilitating better development in the formation of liquid phase , and giving rise to the sintering of the particles, favoring a less porous structure [28]-[30]. In addition, better control is generated during preheating, achieving better sintering, and consequently, better vitrification and gresification, which improves the efficiency of the firing process, especially in rudimentary furnaces and as problematic as hive furnaces, and increases mechanical properties as will be seen later in figure 6 [31]-[32].

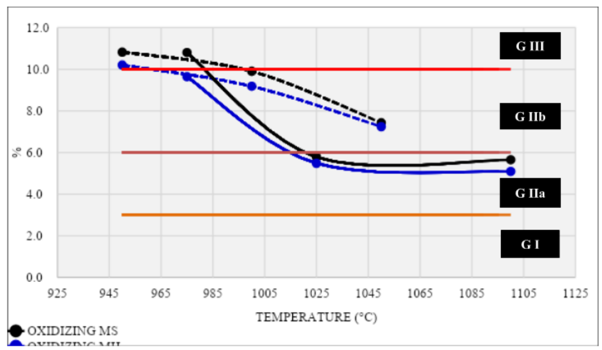

Figure 5. Open porosity of MS and MH.

Likewise, this feature helps to identify the nature of the ceramic body. In the case of coatings, either on the floor or on the wall, the value shown in this property sets, through the mentioned groups, the parameters that these products must meet in the other technological properties, such as mechanical resistance, stain resistance, frost resistance, tribology, among others, these properties ares necessary not only to evaluate the performance of the material against different environments, but also are requirements for the export of ceramic tiles [33]-[35]. For this reason, the evaluation and analysis of this property is of utmost importance because from this result the product will be classified and improvements will be observed. As observed in figure 5, it is detailed that MH and MS are located in the same groups, except in temperatures of 975 °C and 1000 °C, where it is observed that wet grinding improves the technical quality of the same, passing from the AIIb group to AIIa at the two temperatures, which will show an increase in the resistance and durability of the materials, in addition to a higher commercial value. It is noteworthy that, although in most temperatures it does not change groups, it is worth noting the fact that the porosity of the material is reduced, which results in a better microstructure and durability, in reference to the dry grinding processes traditionally used in the region.

In figure 6, mechanical resistance is presented, an increasing trend is observed in the oxidizing atmosphere, the atmosphere most used in regional companies, showing increases of up to 32.9% in MH, compared to 25.2% of MS. Another important aspect to evaluate is that in the reducing atmosphere, a decrease in mechanical resistance is shown as the temperature increases, this suggests that the permanence of the products at each of the temperatures possibly generates small microstructural defects, which causes a decrease in mechanical resistance, despite the reduction in porosity, this is confirmed because the effect is observed in both MS and MH [28], [32].

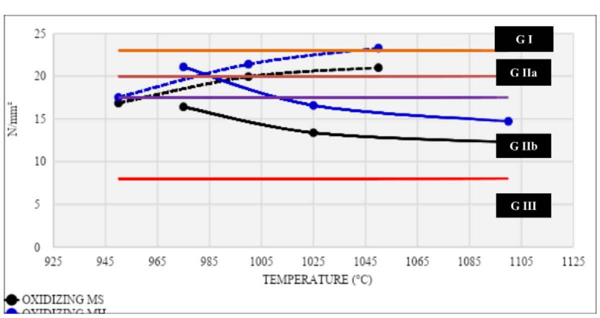

Figure 6. Mechanical resistance of MS and MH

Figure 6 shows significant increases in all temperatures, allowing group change. The most relevant changes that are evaluated, and that allow us to conclude that wet grinding is an ideal preparation for extruded processes, is in the oxidizing atmosphere, where the most relevant changes are shown, which occur at all temperatures. Thus, changes are observed from the AIII group to the AIIb group at 950 °C; from group AIIb to group AIIa at 1000 °C and the most significant change is from group AIIa to group AI at 1050 °C, confirming that this type of grinding not only reduces porosity, but also increases technical quality, achieving improvements not only at lower temperatures but generating increases in the commercial value of the pieces, which makes this type of construction materials more competitive in international markets. This is supported that the evaluated products generate changes to groups of higher technical requirements, therefore, the products will be commercially more competitive.

However, it should be mentioned that in the reducing atmosphere the only group change is achieved at 975 °C, going from the AIII group to the AIIa group. Despite the fact that it is the only change, it stands out that it is achieved at temperatures below 1000 °C. In fact, although mechanical resistance is reduced at medium and high temperatures, it should be noted that MH specimens are still above MS, which have been prepared using traditional methods.

Conclusions

The study of the thermal and technological behavior of an extruded ceramic product through the reconversion of the preparation method by wet grinding, and the effects that occurred during the hive furance firing process, has led to the following conclusions:

Clay is a raw material that presents crystalline structures associated with kaolinite and quartz, with the presence of micaceous minerals, it has free silica, which can affect the preheating stage. The alumina content is lower than expected compared to other clays in the region and the iron oxide generated light colors in the products after firing.

Wet grinding reduced the particles smaller than 63 microns by 22.9%, after the stage of preparation of the plastic mass for extrusion, achieved better packaging of the material by increasing the dry density and it was demonstrated that this process of Grinding improves heat transfer in the material structure, going from 48 h of drying in the MS samples to 32 h in the MH samples, decreasing the drying time by 33.3%.

Regardless of the process variables in the manufacture of a ceramic product by extrusion, it has been proven that the reconversion in the preparation of the raw material not only improves the process window, but also increases the structural and mechanical characteristics. This confirms the close relationship between the preparation of the raw material and its behavior during the manufacturing process.

When comparing the results of water absorption and mechanical resistance in wet grinding, with traditional methods of dry grinding, decreases of up to 7.3% in an oxidizing atmosphere and up to 10.7% in a reducing atmosphere are evident, in addition to increases in mechanical resistance up to 32.9%, which showed higher technical values and therefore higher commercial values

Finally, it can be seen that wet milling is an ideal form of preparation for extruded processes since it generates significant increases in all the properties evaluated, mainly in the mechanical property, evidencing changes to groups of greater technical demand and higher value. commercial, as changed from group AIII to AIIb at 950 ºC; from group AIIb to AIIa at 1000 ºC and from group AIIa to AI at 1050 ºC, all in the oxidizing atmosphere, an atmosphere frequently used in the cooking stages of regional companies.

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