Effect of Kaolin-Quartz-Feldspar Mixing Ratio on Density, Water Absorption, and Strength in Porcelain Ceramics

Cinthya Alvarado, MSc[1], Abigail Huaccha, BS[2], Iván Vásquez Alfaro, BS[3], Hernán Alvarado-Quintana, PhD[4]
[1]Universidad Privada del Norte, Perú, cinthya.alvarado@upn.edu.pe
[2]Universidad Nacional de Trujillo, Perú, ahuaccha@unitru.edu.pe
[3]Universidad Nacional de Trujillo, Perú, ivasqueza@unitru.edu.pe
[4]Universidad Nacional de Trujillo, Perú, halvarado@unitru.edu.pe

Abstract*– The objective of this research was to find the optimum mixing ratio of kaolin-quartz-feldspar concerning the possibility of obtaining porcelain ceramics with the highest strength and density and the lowest water absorption. Statistica 12 software was used to optimize the mix design using the simplex centroid method. Ten compositions were prepared according to the mentioned design, formed by extrusion in the form of cylindrical specimens in triplicate, after sintering the specimens in a gas oven at a temperature of 1280°C, their apparent bulk density and water absorption properties were determined using an immersion method and diametral compressive strength in a 50 kN load press at a speed of 1 mm/min. Contour plots were constructed to determine the optimal mixing ratio for the three investigated properties. From the analysis of these graphs, it was concluded that the triaxial paste with the highest resistance and density and the lowest water absorption is that of 75% kaolin, 5% quartz, and 20% feldspar. Finally, a dilatometric study was carried out for a mixture with that mixing ratio from room temperature to 1300°C to monitor its linear thermal expansion with temperature.

Keywords-- Porcelain; mixing ratio; water absorption; strength; density

I. Introduction

Peru is a very rich country culturally but it is also a country very rich in natural resources. More than thirty types of non-metallic minerals are produced in Peru, these products being used in various sectors such as construction, manufacturing, the chemical sector, agribusiness, energy mining, and the environment. The production of the non-metallic mining subsector was 61.8 million tons, registering an increase of 31.5% compared to 2017 [1, p. 63].

Currently, in our country the production of white body porcelain tile is carried out by Cerámica San Lorenzo and Cerámica Lima (Celima), a Peruvian company that manufactures this product, using various raw materials such as kaolin, kaolinite, quartz, and feldspar which are of local origin. The product is made under very rigorous conditions and requires a high level of technical knowledge in the manufacturing process, in addition to investment in technology and infrastructure, thus managing to export nationally manufactured porcelain tiles, which represents a key milestone for the local industry [2]. However, there is a serious lack of processes that allow obtaining an adequate degree of purity, a high fineness modulus as well as exact control of sintering temperatures; which translates into quality control of both the raw material and the final product. Within the wide range of products marketed by the ceramic industry, the development of porcelain stoneware has been outstanding.

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Porcelain is a ceramic material produced from the mixture of three basic components kaolin (clay), quartz (silica), and feldspar. The clay [$Al_2Si_2O_5(OH)_4$], gives plasticity to the ceramic mix; quartz (SiO_2) maintains the shape of the shaped piece during firing; and feldspar [$K_xNa_{1-x}(AlSi_3)O_8$], serves as flux. These three constituents place porcelain in the phase system [(Na_2O, K_2O)-Al_2O_3-SiO_2)] in terms of oxide constituents, hence the term triaxial porcelain [3]. The distinguishing factor in the properties of different porcelain products is due to the variations in the proportion of said starting materials, the processing, and the firing program adopted. The fired product contains mullite ($3Al2O_3.2SiO_2$) and undissolved quartz crystals (SiO_2) embedded in a continuous glassy phase, originating from feldspar and other low melting impurities in the raw materials. Porcelains represent one of the most complicated ceramic systems due to the complex interrelationship between raw materials, processing routes, and the kinetics of the firing process [4].

The porcelain stoneware tile is a very compact product, completely vitrified and with extremely low porosity [5]. Vitrification indicates a high degree of sintering melting that gives fired porcelains low porosity (sometimes <0.5%) and high glass content (>40%) [6]. The main technological advantages of porcelains are their high mechanical strength, low water absorption, translucency, and durability [7].

Within the wide range of products marketed by the ceramic industry, the development of porcelain stoneware has been outstanding. The word "gres" means that the ceramic mass of the tile is extremely vitreous. In recent years, it has experienced the largest increase in production and sales of all ceramic building materials. The main difference between porcelain and porcelain stoneware lies in their firing program. Thus, porcelain is sintered following a long-term process (24 hours or more) to promote high mullite formation, while porcelain tile is manufactured through a faster sintering cycle (60 – 90 minutes), in which the tiles are inside the oven for no more than 90 minutes [8].

The international standards that regulate the manufacture of ceramic tiles, ISO 13006 (2018), and UNE-EN 14411 (2016), among others, classify tiles based on two parameters: the manufacturing method and water absorption (E).

Combining these two values results in the creation of ten product groups. Table I summarizes this classification [9].

TABLE I
TILE CLASSIFICATION BASED ON WATER ABSORPTION

Manufacturing method	la $E \leq 0.5\%$	lb $0.5\% < E \leq 3\%$	lla $3\% < E \leq 6\%$	llb $6\% < E \leq 10\%$	lll $E > 10\%$
A: Extrusion	Ala	Alb	Alla	Allb	Alll
B: Dry Pressing	Bla	Blb	Blla	Bllb	Blll

For a tile to be considered porcelain tile, it must have a water absorption equal to or less than 0.5% (Ala or Bla groups). Non-porcelain stoneware or simply stoneware can have absorption of up to 6% (groups Alb, Blb, Alla, Blla).

In Peru, there is resistance to producing high-temperature firing white ceramics due to a long pottery tradition or ultimately, due to a lack of knowledge of processes, techniques, and/or adequate parameters to obtain a good product. Although, we have enough raw material in natural deposits as is the case in the province of Huamachuco; there is a great lack in terms of processes that allow obtaining an adequate degree of purity, a high fineness modulus as well as an exact control of sintering temperatures; which translates into quality control of both the raw material and the final product.

The largest energy consumption during the manufacture of porcelain and other ceramics occurs during the firing process. Therefore, work has been done to reduce the total energy consumption by modifying the heating ramps and chemical compositions [7]. Our country does not have porcelain production plants that carry out this advanced technology because we need more technological production equipment and studies in this regard. For this reason, it is necessary to carry out systematized studies on the production of porcelain with local raw materials, in this way this research is intended to analyze, quantify and deduce the appropriate compositions of each component since, depending on the mix design of the components different properties will be obtained and therefore, different types of porcelain. Likewise, it is intended to spread the existing technology and apply it in our country.

The mixing ratio of the materials plays an important and decisive role in the quality of the final product; however, there is a need to better understand the relationship between the properties of porcelain tiles and the starting composition of raw materials and their processing to improve their processability and performance [10].

There are publications on this topic, which are detailed below:

Gültekin, Topates, and Kurama investigated a porcelain tile that was sintered at nine different temperatures between 1150 and 1230 °C. The investigation showed that the amount of the amorphous phase and the degree of densification show good compatibility with each other; up to 1200 ° C gradually increase. The pore size increases up to 1170 °C. [11]

Sokolár, Kersnerová, and Sveda evaluated the sintering behavior of dry-pressed samples made from the mixture of kaolin and three different rock types of feldspar, bone ash, and quartz sand dependent on water absorption and temperature. cooking. The optimal fluxing agent for sintering is bone ash: the mixture with 20% by weight showed a sintering temperature of 1200°C. That's about 50°C lower compared to the fluorine fluxing agent. [12]

Lerdprom, Chinnam, Jayaseelan, and Lee compared porcelain samples processed at heating rates of 5, 15, and 30°C/min with samples processed under direct sintering. Direct sintering reduced the total processing time by ~50% and also reduced the sintering temperature from 1200°C to 1175°C, suggesting that the direct sintering method provides benefits meaning that it could replace sintering in the future. conventional porcelain processing. [7]

Kivitz, Palm, Heinrich, Blumm, and Kolb investigated the behavior of a potash porcelain stoneware body during the firing process. The optimal firing temperature is reached in the range of 1260º-1280ºC when the open porosity reaches a minimum value and at the same time, the linear contraction is maximum. [13]

Nyongesa and Aduda showed that the effect of mullite and quartz phases on porcelain strength, compressive strength, and porcelain tensile strength decreases as quartz increases. The tensile strength increases and the compressive strength decreases as the mullite increases. [14]

According to this background and being density (porosity) and water absorption the properties on which they are based to classify porcelains, the problem to be addressed in this study was to determine what is the effect of the mixing ratio kaolin/quartz/feldspar on density, water absorption and strength of porcelain ceramics?

II. MATERIAL AND METHODS

A. Material

The raw materials used to prepare porcelain in this study were kaolin (PZ-600), feldspar (SIP M325), and quartz (Granulated M-40). The three raw materials are of Peruvian origin and were supplied by the mining company Aggregates Calcareos S.A. The chemical compositions of the three raw materials used were determined by X-Ray Fluorescence which is summarized below in Table II.

TABLE II
CHEMICAL COMPOSITION OF THE RAW MATERIALS USED (% WEIGHT)

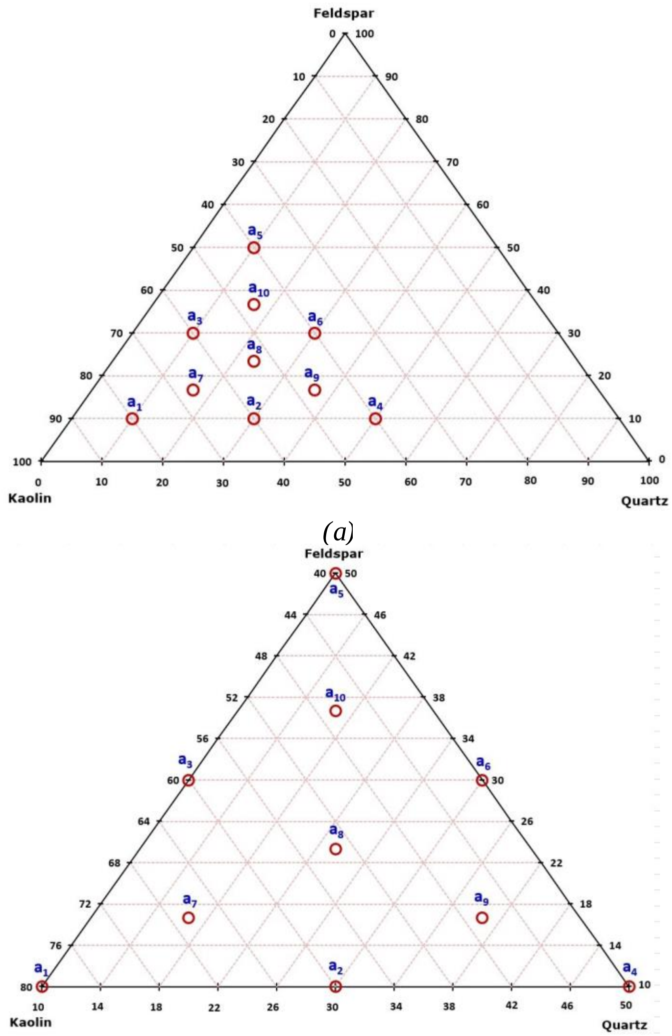
OXIDE CONTENT	KAOLIN	FELDSPAR	QUARTZ
SiO_2	53.60	68.61	97.80
Al_2O_3	31.12	16.42	0.77
Fe_2O_3	0.08	0.12	0.02
CaO	0.10	1.10	0.03
Na_2O	-	2.88	-
K_2O	-	9.95	-
C.L.	12.46	0.14	0.19

Regarding the particle size of the raw materials used, the kaolin presents a d50 of 3.54 µm, determined by sedigraph, it passes almost completely the mesh No. 400 (38 µm) leaving only a residue of 0.00121%. The feldspar passes mesh No. 325

(45 μm) at 98.12%, leaving a residue on this mesh of 1.88%. Finally, the quartz passes mesh No. 40 (425 μm) 93.4%, leaving a residue on the said mesh of 6.6%.

B. Research Design

A unifactorial experimental design was used, with the factor or independent variable: kaolin-quartz-feldspar mixing ratio with ten levels (Fig. 1) and three response or dependent variables: apparent bulk density (g/cm^3), water absorption (%), and Diametral compressive strength (MPa). All the experimental tests were done in triplicate, so the total number of tests per response variable was 30.



(a)

(b)

Fig. 1 Triaxial composition diagrams of the studied formulations. (a) Complete ternary diagram indicating the location of the study region. (b) The ternary diagram focused only on the study region.

C. Process

Specimens with a circular section of 28mm diameter and 8mm height were formed by extrusion, of ten different formulations in triplicate, according to the design shown in Figure 1, in which the percentages of each component in the mixture were varied: the quartz of 10 to 50%, feldspar from 10 to 50% and kaolin from 40 to 80%. The designations of (% Quartz - % Feldspar - % Kaolin), respectively, were: $\mathbf{a_1}$ (10%-10%-80%), $\mathbf{a_2}$ (30%-10%-60%), $\mathbf{a_3}$ (10%- 30%-60%), $\mathbf{a_4}$ (50%-10%-40%), $\mathbf{a_5}$ (10%-50%-40%), $\mathbf{a_6}$ (30%-30%-40%), $\mathbf{a_7}$ (16.67 %-16.67%-66.67%), $\mathbf{a_8}$ (23.33%-23.33%-53.33%), $\mathbf{a_9}$ (36.67%-16.67%-46.67%) and $\mathbf{a_{10}}$ (16.67%-36.67%-46.67%).

Once the specimens were formed, they were dried for 72 hours in the environment and then in an oven at 110 °C for 24 hours. They were then sintered in a gas oven at a maximum temperature of 1280°C, keeping them at said temperatures for 30 minutes. Finally, they were cooled in the same oven for 24 hours.

The water absorption, E (%), and the apparent bulk density, ρ_b (g/cm^3), were determined by the Archimedean liquid displacement method following the procedure described in the ASTM C 373-16 Standard, that is, dried samples at constant mass (D) were placed in distilled water, boiled for 5 hours, and soaked for an additional 24 hours at room temperature. After impregnation, the suspended mass in water (S) and the saturated or soaked mass (M) of each sample were obtained. To obtain the weights stipulated in the aforementioned standard, an analytical balance with a resolution of 0.1 mg was used. The water absorption, E (%), expresses the ratio of the mass of water absorbed to the mass of the dry sample and was calculated using the following formula: $E (\%) = [(M - D)/D] \times 100$ The apparent bulk density, ρ_b (g/cm^3), was calculated according to the following formula: ρ_b (g/cm^3) = D/V, where V (cm3) is the external volume obtained as follows: V= M s.

The diametral compressive strength (DCS) was determined in a Humboldt HM5030 load-bearing press with a load capacity of 50 kN. The measurements were made with a displacement speed of 1 mm/min, with a load applied until the specimen failed, based on the ASTM D3967 standard. The strength (DCS) was calculated using the following formula: $\sigma_f = 2P/\pi LD$ Where: σ_f diametral compressive strength (MPa); P: the force exerted (N); L: thickness (mm); D: diameter (mm).

D. Analysis of data

The data obtained in the previous item were processed with the Statistica 12 Software using the simplex centroid method to obtain the response surfaces, the contour diagrams, and the tracking diagrams of the studied property.

Once the graphs were analyzed, with the statistical data provided by the software, the model that best fit the data was selected. Finally, from the contour graphs, it was determined which is the optimum mixing ratio that has the highest bulk density and strength and the lowest water absorption. A dilatometric test was performed on this formulation to evaluate the evolution of its densification with temperature, for this, the LINSEIS L75H/1600 Dilatometer was used following the procedure established in the ASTM C372 Standard.

III. RESULTS

A. Apparent bulk density results

Figure 2 shows the contour graph of the apparent density at the sintering temperature of 1280°C as a function of the mixture proportions of the investigated porcelains, which were obtained with the cubic model (since it had the largest R^2). The optimal mixtures are in the dark red zone close to the kaolin vertex, while the mixtures with low apparent densities are in the dark green zone.

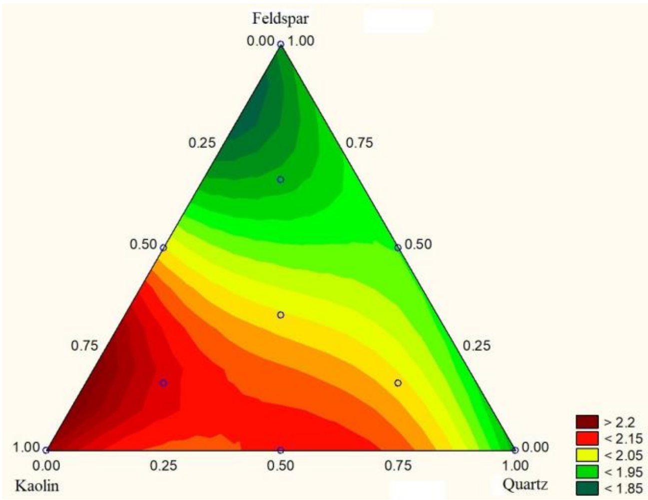


Fig. 2 Contour diagram of the Apparent Bulk Density (g/cm^3) as a function of the Mixing Proportions at the sintering temperature of 1280°C.

Figure 3 shows the tracking of each of the components of the mixture on the Density (gr/cm^3) for the mixture $\mathbf{a_{10}}$: 53.33%K-25.33%Q25.33%F at the sintering temperature of 1280° c. Kaolin, being the largest component in percentage in the mixture, increases the apparent density contrary to the contribution of quartz, which, when increased, decreases the apparent density, however, in a range of 10% to 35%, it maintains the density at 2.05 gr/cm^3. Feldspar from 80% tends to decrease the bulk density.

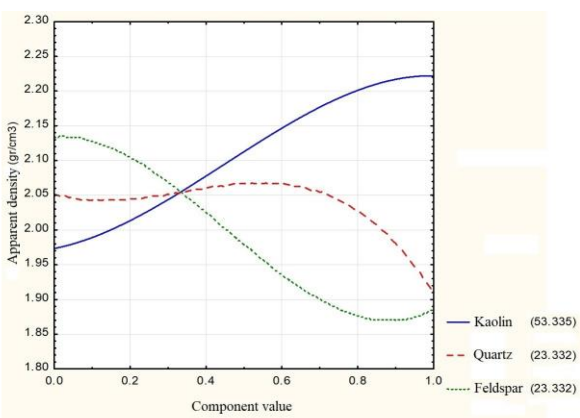


Fig. 3 Trace of the Apparent Bulk Density (g/cm^3) depending on the mixture proportions at the sintering temperature of 1280°C taking as reference the mixture $\mathbf{a_{10}}$.

B. Water absorption results

Figure 4 shows the contour graph of the water absorption at the sintering temperature of 1280°C as a function of the mixture proportions of the investigated porcelains, which were obtained with the cubic model (>R^2). This figure shows the highest water absorptions in dark red and the lowest water absorptions in dark green, which is what is sought in this research.

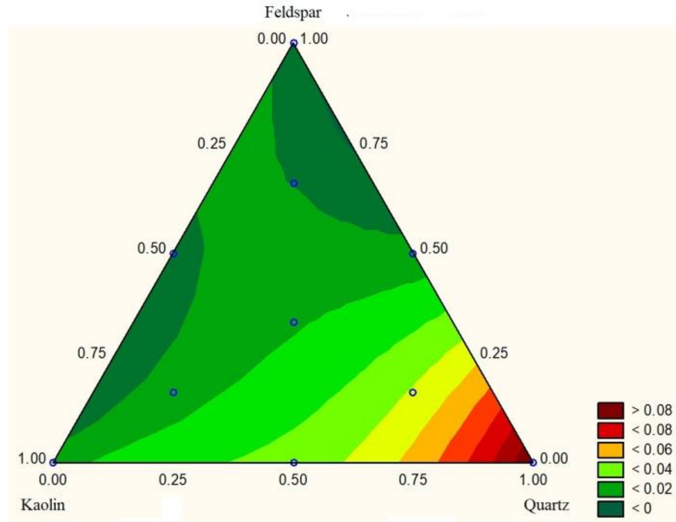


Fig. 4 Contour diagram of the Water Absorptions (%) as a function of the Mixing Proportions at the sintering temperature of 1280°C.

Figure 5 shows the tracking of each of the components of the mixture on the water absorption (%) for the mixture $\mathbf{a_{10}}$: 53.33%K-25.33%Q25.33%F at the sintering temperature of 1280°C. It is observed that as the quartz increases, the absorption increases; creating porosity in the mixture which is not desirable in porcelain. It should be noted that the effect of kaolin is almost horizontal, that is, it maintains the mixture; however, by increasing the amount of feldspar in the mixture, the absorption decreases, thus becoming the component that improves the mixture and therefore the properties of the porcelain.

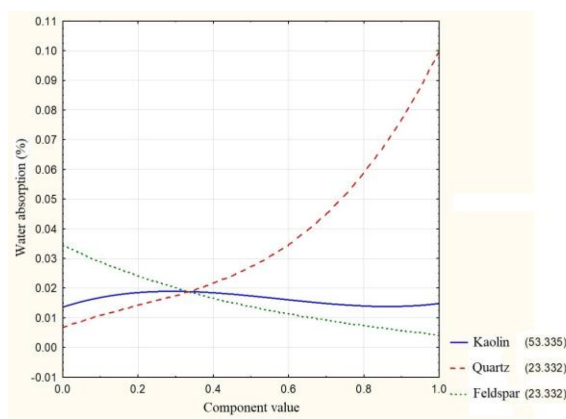


Fig. 5 Trace of the Water Absorption (%) depending on the mixture proportions at the sintering temperature of 1280°C taking as reference the mixture $\mathbf{a_{10}}$.

C. Diametral Compressive Strength results

Figure 6 shows the contour graph of the diametral compressive strength (MPa) at the sintering temperature of 1280°C as a function of the mixture proportions of the investigated porcelains, which were obtained with the cubic model (>R^2). The dark green areas indicate the mixtures with lower resistance to diametral compression, which is not what we are looking for. On the other hand, the dark red areas show the optimal mixtures, that is, those that will generate porcelains with greater resistance.

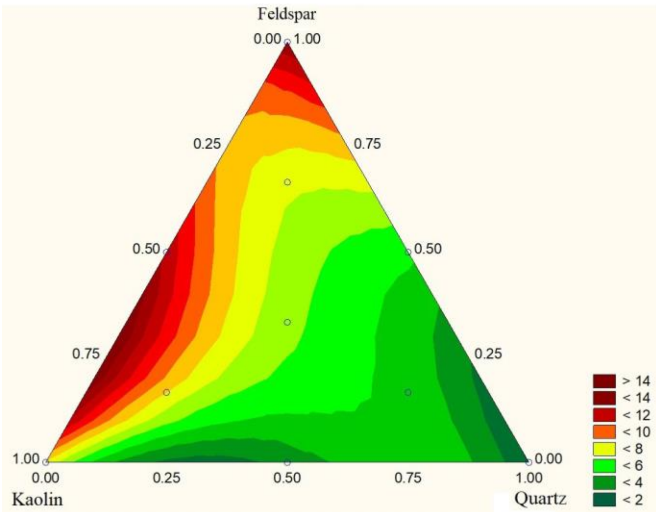


Fig. 6 Contour diagram of the Diametral Compressive Strength (MPa) as a function of the Mixing Proportions at the sintering temperature of 1280°C

Figure 7 shows the tracking of each of the components of the mixture on the Diametral Compressive Strength (MPa) for the mixture **a_{10}**: 53.33%K-25.33%Q25.33%F at the sintering temperature of 1280°C. It is observed that both the addition of feldspar and kaolin increase the diametral compressive strength, but this increase is more pronounced in feldspar, especially at low (<20%) and high increases (>80%). On the other hand, the addition of quartz decreases the diametral compressive strength in all ranges.

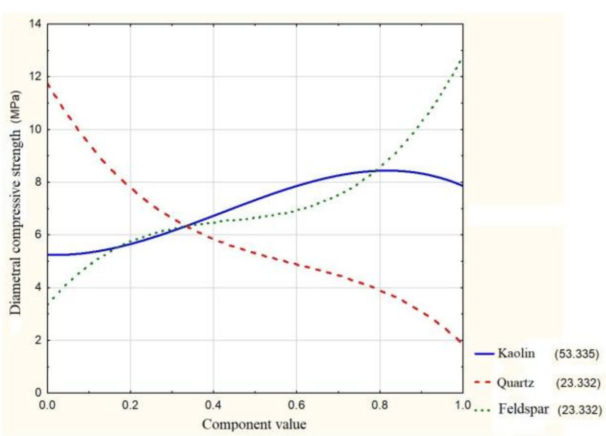


Fig. 7 Trace of the Diametral Compressive Strength (MPa) depending on the mixture proportions at the sintering temperature of 1280°C taking as reference the mixture **a_{10}**.

D. Dilatometric analyses

From the analysis of figures 2, 4, and 6 it was determined that porcelain with 75% Kaolin, 5% Quartz, and 20% Feldspar would have the best results. A cylindrical specimen was prepared with said composition and with the initial dimensions presented in Table III. A dilatometry test was then performed. Table III shows that both dimensions and mass have shrunk and vitrified. Figure 8 shows the dilatometric curve of this porcelain. In the first section from room temperature to approximately 500°C, a slight expansion is observed attributed to the expansion due to heating of the components, then there is a slight contraction between the temperatures of 500°C-850°C which corresponds to the initial stage of sintering Likewise, the contraction observed in the section between 850° and 1050°C can be attributed to the beginning of sintering with the presence of liquid phase [15] in the last section between 1050° and 1225°C, the greatest contraction occurs, attributed to the secondary (intermediate) stage of sintering, where the formation of necks grows, pore channels are formed and a high contraction is produced, this it is a stage in which, as a result of a higher capillarity pressure, a greater dissolution-reprecipitation of particles is promoted, which increases the speed of densification [16]. Finally, the section from 1225° to 1300°C produces a very small contraction attributed to the tertiary (final) stage of sintering where according to reference [15].

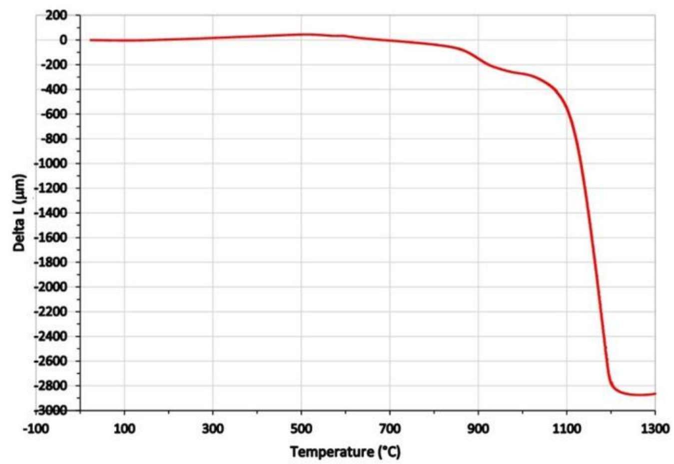


Fig. 8 Dilatometric curve of porcelain with 75%K, 5%Q, and 20%F.

TABLE III
DATA OF THE PORCELAIN SPECIMENS BEFORE AND AFTER CARRYING OUT THE DILATOMETRY TEST

	INITIAL	FINAL
LENGTH	22.9 mm	20 mm
DIAMETER	10 mm	9.01mm
MASS	2.6632 g	2.4228 g

IV. DISCUSSION

According to Figure 5, the effect of the components at 1280°C on the absorption of porcelain is shown, it is between 0.09% and 0.35%, which is a fairly acceptable value. These water absorption results obtained agree with the Reference

[17], when investigating the behavior of a body of potassium porcelain stoneware during the sintering process, determined that the optimum sintering temperature is reached in the range of 1260º-1280ºC when the open porosity reaches a minimum value and at the same time the linear contraction is maximum. Water absorption decreases with increasing sintering temperature, this is because the addition of fluxes (feldspars) to the paste causes the material to reach a liquid state when cooked, thus most of the pores initially present are covered, which reduces the penetration of water. According to Reference [18], the total porosity represents a trend similar to linear shrinkage, which initially increases (due to the decrease in open porosity) with the increase in sintering temperature until reaching a maximum value and then decreases due to the increase in closed porosity (called swelling). In Fig. 7, feldspar from 40% tends to markedly increase the resistance to diametral compression, however, quartz decreases it. This is related to the Reference [14] the effect of mullite and the phases of quartz on the strength of triaxial porcelain from Kenya, quartz-feldspar-kaolin has been investigated. It was found that both the compressive and tensile strength of porcelain decrease with high quartz content, and circumferential cracks around quartz grains may constitute fracture initiation defects.

V. CONCLUSION

A sintered triaxial porcelain was obtained at a temperature of 1280°C with an apparent density of 2.15 g/cm^3 and water absorption of 0.99% with a diametral compressive strength of 7.02 MPa. With a mixture dosage of 53.33%K-23.33%Q23.33%F.

The increase in feldspar and kaolin in the mixture increases bulk density, diametral compressive strength and decreases water absorption, while the increase in quartz decreases bulk density and increases water absorption.

The optimal mixtures would be the mixtures: 75% kaolin, 5% quartz, and 20% feldspar.

The mathematical model that best correlates the experimental data at the sintering temperature of 1280°C is the cubic model, whose equations are:

For V = Apparent Bulk Density:

$v=+0.0147x+0.099y+0.004z-0.091xy-0.011xz-0.153yz+0.2075xyz+0.086xy(xy)-0.089xz(xz)+0.$

For V = Water Absorption:

$V=+0.0147x+0.099y+0.004z-0.091xy-0.011xz-0.153yz+0.207xyz+0.0861xy(xy)-0.0897xz(xz)+0.$

In both cases: x = %Kaolin, y = %Quartz and z = %Feldspar.

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