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New Advances in the Analysis of Ceramic Materials

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ABSTRACT

This new advance in the theory of constitution in its elaboration of ceramic materials and specifically ceramic bricks is reinforced by this new investigation with SEM, DRX and EDX electron microscopy for brick samples made with materials from two different regions in Paraguay, the Western Region and the Eastern Region. It is intended to simplify the knowledge of the process and therefore help to develop new materials with these techniques using simple raw materials such as clay, sand and silt, and may be some other and new materials that help in the production process of ceramic materials. Analysis of characterization of raw materials, processed and finished materials, physical, chemical and mechanical tests such as compression resistance, bending of small-sized specimens manufactured in the laboratory and in real sizes in the factory were carried out. This continues with the series of investigations in ceramics and showed the stages of the process that involves obtaining the raw material, the elaboration of the ceramic process, storage, drying, wetting, kneading, sintering, and the physical and mechanical testing of the pieces made. The electron microscopy of the samples made at different temperatures in the laboratory compared to each other, lead us to analyze them from the nanoscopic point of view so that it approaches the theoretical knowledge of their formation in their processing and that influence their physical and mechanical properties. analyzed in this study.

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Introduction

This work seeks to state a theory about the mechanism of clay adhesion to sand and those that make up ceramic products, especially brick, in addition to silt inclusions. Sintering is the heat treatment of a powder, in this case ceramic, at a temperature lower than the melting temperature of the mixture to increase the strength and resistance of the piece by creating strong bonds between the particles. In this process it transforms the powder into a compact and consistent powder. The structures of ceramics are generally crystalline and can be bonded ionically, covalently, or have both types of bonds at the same time. Ionic structures are packed very tightly and can be stable or non-stable. Silicates are very important for the ceramic industry, based on their structure on tetrahedra (SiO4)-4, and there are several ways in which these can be combined. In sintering, the pores found between the initial particles (which shrink the component) are removed, combining with growth coupled with a strong bond between adjacent particles. For this to occur there must be a mechanism to transport matter and an energy source must be available to activate and sustain this transport of matter. It is achieved by diffusion and viscous flow and with heat as a source of energy to maintain the transport of the material. "The term clay not only has mineralogical connotations, but also particle size; in this sense, all fractions with a grain size less than 2 mm are considered clays. According to this, all phyllosilicates can be considered true clays if they are within said size range, even minerals not belonging to the phyllosilicate group (quartz, feldspars, etc.) can be considered clay particles when they are included in a clay sediment and their sizes do not exceed 2 mm. Clays are essential constituents of a large part of the soils and sediments because they are, for the most part, final products of the weathering of silicates that, formed at higher pressures and temperatures, hydrolyze in the exogenous environment. Clays, like the rest of the phyllosilicates, have a structure based on the stacking of planes of oxygen and hydroxyl ions.

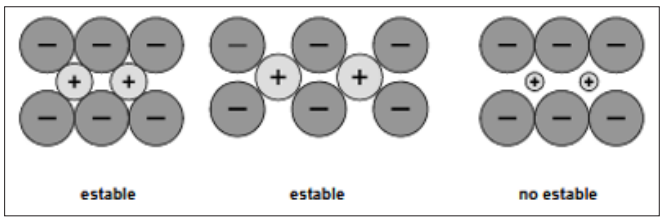


Figure 1: Ionic Stable and Unstable Structures

Methodology

Samples were obtained from ceramic industry sites in Chaco and Tobatí. They were used to make specimens of solid ceramic bricks so that they were kneaded in the factory and subjected to dehydration and sintering in muffles in the laboratory, contrasted with those obtained in the factory. Samples were processed with interrupted cooking at certain temperatures from 500ºC and every 100ºC up to a temperature of 1000ºC. It was observed in a metallographic microscope, the absorption and mechanical resistance were analyzed, the main parameters to determine the category and quality of the brick and the intermediate temperature states. Dimensions and density of said materials were taken. The interpretation was based on previous work in the area, the literature consulted. The elements that make up the bricks were analyzed using XRD, EDX and scanning electron microscopy.

The preparation of the samples was carried out at the INTN. The raw materials used by the ceramic industries of Tobatí and Bajo Chaco in our country were dried and ground. After natural and artificial drying at 110ºC, the samples were ground by passing the No. 40 sieve and moistened with cte. at 5% for the raw materials, it was kneaded by hand passing the No. 8 sieve with a 2.36 mm opening, they were placed in a mold and pressed at 155 kg/cm². Infrared spectrophotometry tests were carried out on the raw material and XRD on the raw materials. Tests were also carried out on texture analysis and classification of the raw material by decantation, Bouyoucos, and natural humidity analysis.

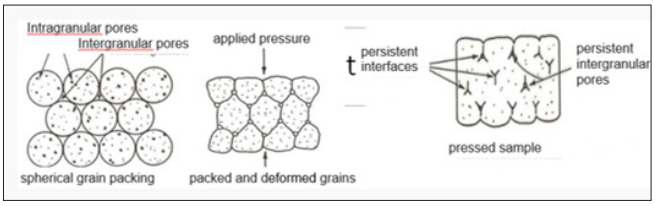


Figure 2: Densification of Ceramic Grains during Manufacturing

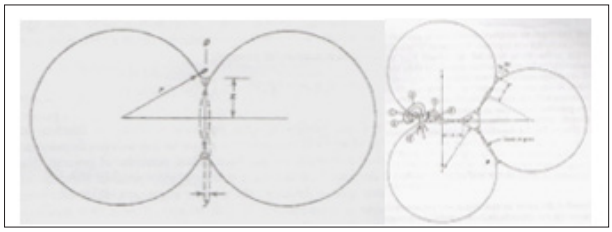


Figure 3: Models for the Transport of the Material during Sintering, having a Contact Area x, an Interface and the Center of the Sphere has moved a Distance, in the Solid State these Transformations Occur.

Analysis Results

Table 1: Component Oxides of the Analyzed Clays (Own Elaboration)

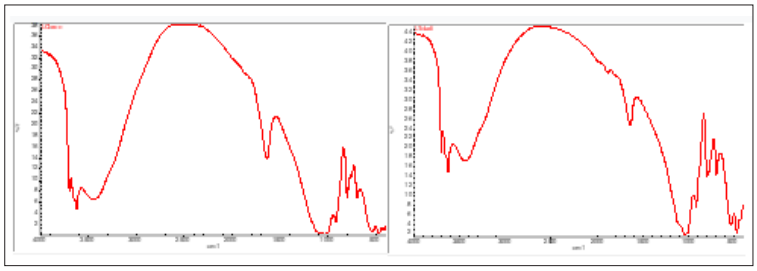
Element	M1 Chaco Mud	M2 Hovaré Tobatí	M3 Tobatí Mud	M4 Kaolin Tobatí	M5. Tobatí 90%Mud+5%Kaolín+5%Hovaré
CaO	0,65	0,17	1,16	0,55	1,076
SiO_2	67,03	75,01	73,93	58,32	73,204
Al_2O_3	17,69	13,50	13,04	25,08	13,665
Fe_2O_3	6,25	5,43	3,99	4,61	4,093
SO_3	0,07	0,07	0,09	0,08	0,089
K_2O	0,85	0,22	2,25	0,62	2,067

In table 2, the important content of SiO_2 can be seen, confirmed by the XRD, in addition the contents of Al_2O_3 and Fe_2O_3 together are important but do not exceed 24% in the finally analyzed bricks that are a product of the raw materials M_1, M_3 and M_5

Table 2: Results of Soil Texture Classification by Decantation or Bouyoucos

	SOIL CLAIFYING RESULTS BY BOUYOUCOS			
M. Raw Material	Clay (%)	Silt (%)	Sand (%)	Type of Soil
M1.Chaco Mud	30,00	60,00	10,00	Loamy Clayey Silt
M2.Tobatí Hovare	38,31	17,78	43,91	Silty Clay
M3.Tobatí Mud	32,48	6,00	61,52	Sand silty clay
M4.Tobatí Kaolin	30,48	24,00	45,52	Sand silty clay
M5. Tobatí	32,67	7,49	59,84	Sand silty clay

The Spectrophotometry results are as follows:



Figures 4 and 5: Spectrophotometry of Chaco Mud M_1 and Tobatí Mud M_3

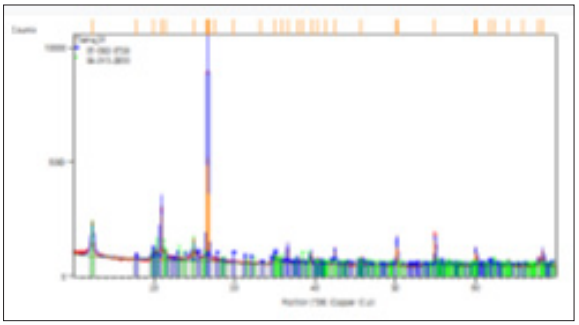


Figure 6: X-Ray Diffractometry (XRD) Results of Sample M1 from Chaco

Table 3: X-Ray Diffractometry (XRD) Results of Sample M1 from Chaco

Pattern List Visible	Ref.Code	Score	Compound	Displ.[°2θ]	Scale Fac.	Chem.
Name			**Formula**			
*	01-082-3726	43	Potassium Sodium Calcium Magnesium Aluminum Iron Silicon Oxide Hydroxide	0,000	0,042	(K0.936 Na0.06 Ca0.01) (Al1.83 Fe0.16 Mg0.01) (Si3.1 Al0.9) O10 (O H)$_2$
*	04-013-2830	41	Aluminum Silicate Hydroxide	0,000	0,134	Al_2 Si_2 O_5 (OH)$_4$

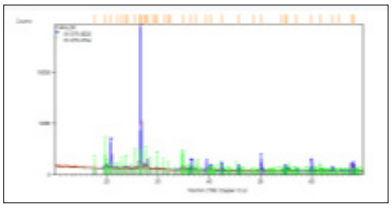


Figure 7: X-ray Diffractometry (XRD) Results of Sample M3 from Tobatí

Table 4: X-Ray Diffractometry (XRD) Results of Sample M2 from Tobatí

Pattern List Visible	Ref.Code	Score	Compound Name	Displ.[°2θ]	Scale Fac.	Chem. Formula
*	01-075-8320	76	Silicon Oxide	0,000	0,957	Si O_2
*	01-070-3754	29	Potassium Aluminum Silicate Hydroxide	0,000	0,200	K (Al_4 Si_2 O9 (O H)$_3$)

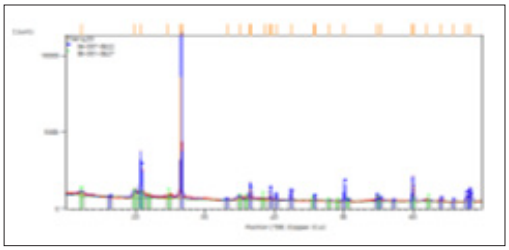


Figure 8: X-Ray Diffractometry (XRD) Results of Sample M3 from Tobatí

Table 5: X-Ray Diffractometry (XRD) Results of Sample M3 from Tobatí

Pattern List Visible	Ref.Code	Score	Compound Name	Displ.[°2θ]	Scale Fac.	Chem. Formula
*	04-007-0522	91	Silicon Oxide	0,000	0,948	$Si O_2$
*	00-001-052	44	Aluminum Silicate Hydroxide	0,000	0,047	$Al_2 Si_2 O_5$
7				(O H)4		

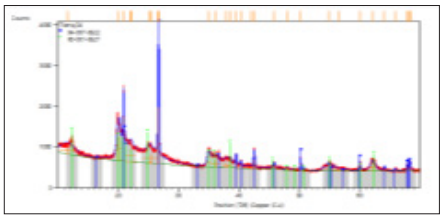


Figure 9: X-Ray Diffractometry (XRD) Results of Sample M4 from Tobatí

Table 6: X-Ray Diffractometry (XRD) Results of Sample M4 from Tobatí

Pattern List Visible	Ref.Code	Score	Compound Name	Displ.[°2θ]	Scale Fac.	Chem. Formula
*	04-007-0522	76	Silicon Oxide	0,000	1,025	$Si O_2$
*	00-001-0527	59	Aluminum Silicate Hidroxide	0,000	0,189	$Al_2 Si_2 O_5 (O H)_4$

Tobatí Sample M_5 combines 90% of Sample M_3 + 5% of M_2 + 5% of M_4

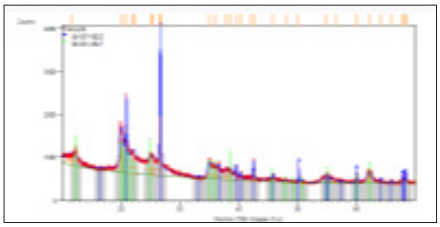


Figure 10: Images of Mechanical Tests on Manufactured Samples and Bricks

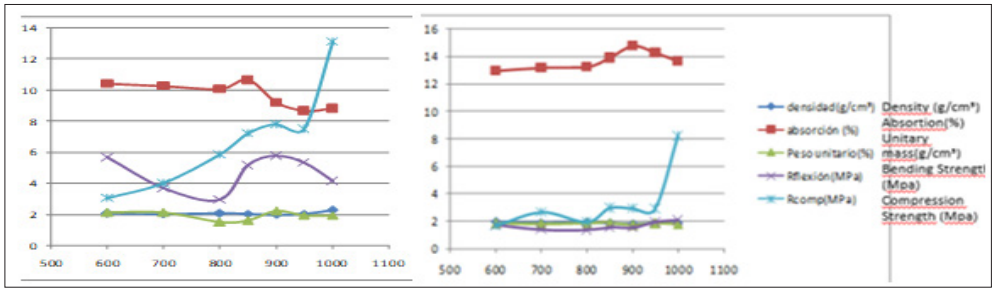
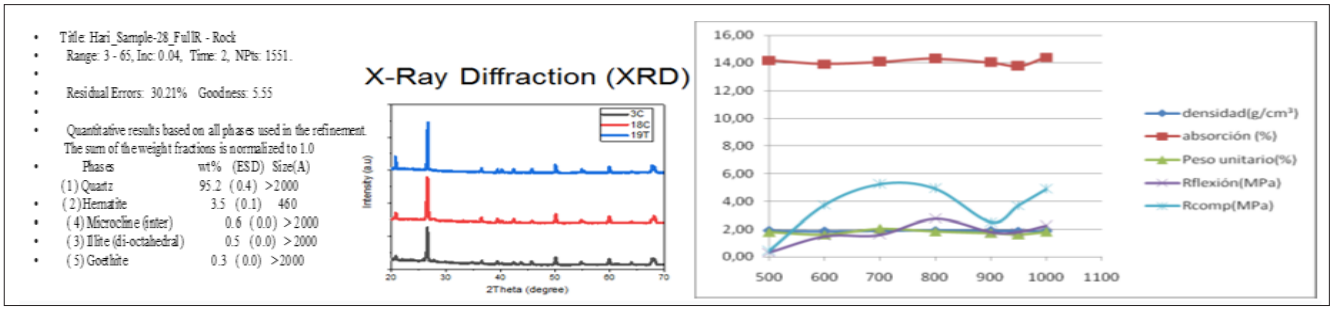


Figure 11: Physical and Mechanical Results on Reduced-Scale Bricks from Chaco M1 Mud and Tobatí M3 Mud. At the Temperature of 900ºC, a Reasonable Density and Absorption was Achieved, the same for Flexural and Compressive Strength in Sample M1 from Chaco. At a Temperature of 1000ºC, a Reasonable Result of Absorption, Flexural and Compression Resistance was Achieved



Figures 12 and 13: Results of Full-Scale Brick Bending and Compression Tests in (Kg/Cm2) Except Poisson's Ratio (U) Which s Dimensionless

In relation to Tobatí's M5 brick, the bending and compression resistance at 750ºC increases, but energy efficiency improves with a decrease in compression resistance with respect to M3, but sufficient in accordance with what the market needs to " load-bearing exposed brick for a single floor." In other properties of M5 there are no important variations during its process, which shows that the densification during kneading and the granulometric distribution favor its resistance for category C of the NP. However, on a natural scale it has been shown to be Category B. A new trend of increasing resistance to compression and bending towards 1000ºC is also recorded. Finally, the results of compression, bending and cutting of bricks used in construction were contrasted with a Reference brick currently sold on the market.

Table 7: Results of Full-Scale Brick Bending and Compression Tests in (Kg/Cm2) Except Poisson's Ratio (U) Which s Dimensionless

Bricks	Bending Strength (kg/cm2)	E (kg/cm2)	Shear strength (kg/cm2)	G (kg/cm2)	u	Compression Strength (kg/cm2)
M1C	59.78	3890	15.94	1298	0.5	110.6
M5T	41.61	5557	10.49	1854	0.5	78.5
Ref	19.72	6113	4.82	2038	0.5	98.6

Bricks sizes in cm: 1C: 23,2x11,5x6; M5: 26x13,2x5,5 y Ref: 23,3x11x5,2

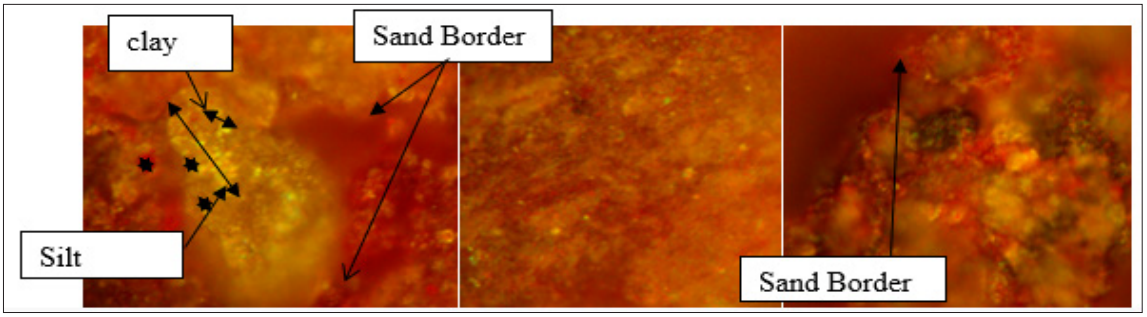
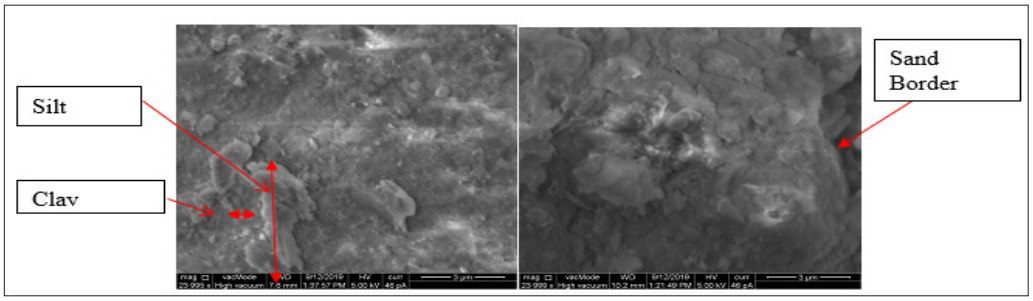


Figure 14 to 16: The Results of Sample M5 from Tobatí at 700ºC, Product of the Mixture of 3 Raw Materials Selected from that Locality (100X)



Figures 17 and 18: SEM Microscopy, Images from the University of Tulsa, Oklahoma

The series of small grains can be observed magnified 23,895 times by electron microscopy between cavities and protuberances, taking into account the original sizes of the original components such as: sands of 0.06 to 2 mm, silts of 0.002 to 0.06 mm and Clays of less than 0.002 mm that were somewhat compacted in their original size and forming larger grains after reducing the vacuum and increasing the grains with temperature.

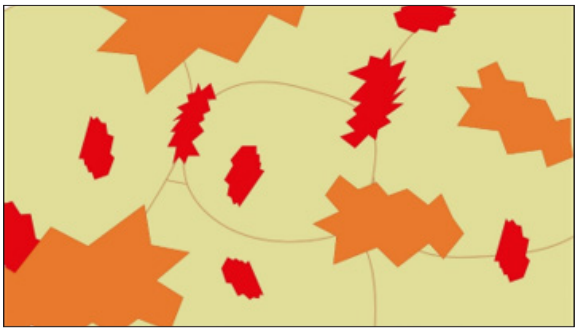


Figure 19: Model of Final Adhesion after Cooling from Sintering

When the sand grains are fractured or broken, they separate due to the resistance they provide to the ceramic brick. The clays (some silts) that agglomerate and are cooked to the sand grains break first, leaving the particles crumbled into powder in a pointed shape when prepared for electron microscopy.

Conclusion

The theoretical statement, which involves the manufacturing process, is finally like this:

In a first stage of wetting and kneading, the sand receives clay and silt from its interior and incrustations on its exterior. When compacted, these mix more intimately, forming fundamental grains of different sizes. During cooking, these grains grow due to thermodynamics, causing expansions of the fundamental grains that join with their similar ones, vibrating, welding and sewing together, maintaining the difference in their sizes, reducing or almost eliminating voids and forming microscopic and nanoscopic grains [1-15].

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