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NEW CONTRIBUTIONS REGARDING THE MECHANISM OF ADHESION AND STRENGTH IN TRADITIONAL CERAMICS

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Abstract: The research project will allow to understand the operation of physical mechanism of clay adherence to sand during the process of kneading, drying and firing in manufacturing ceramics, in order to lead to a contribution to the theory that explains the interactions of both phases and how they work to achieve mechanical properties and little absorption, important requirements for the quality of these products, some of them structural. Raw material is obtained from deposits that are used to manufacture ceramics in the Western (Paraguayan Chaco) and Eastern Regions of Paraguay and with these materials half brick test tubes both of solid and hollow ceramic bricks submitted to drying and firing temperature processes interrupting said process according to proposed temperature each 150° C up to 750° and from then on each 50° up to 900° or 1200°C according to the mineralogic composition of raw material, in order to continue studying macroscopically and microscopically and validate the theoretical contribution that we seek to verify in the aforementioned theory. Using techniques such as XRD, SEM, among others. Besides physical density tests, unit weight, bending and compression in semi-pressed solid bricks of small scale sizes in order to facilitate investigation. In this study at least three different types of clays will be chosen, some combined to form other ceramic products and take advantage of this variation for the conclusions of this theoretical study.
Keywords: Mechanical properties, adherence mechanism, raw material, final product.

INTRODUCTION

The term "clay" not only has mineralogical connotations but also particle size, in this sense all fractions with a smaller grain size than 2 mm are considered clays. According to this all phyllosilicates can be considered true clays if they are within said size range, including minerals not belonging to the group of phyllosilicates (quartz, feldspars, etc) can be considered clayey particles when they are included in a clayey sediment and their sizes do not exceed 2 mm.

METHODOLOGY

Samples were obtained from deposits used by ceramic industries from the Chaco and Tobatí. These samples were used to elaborate test pieces of solid ceramic bricks so that they are slapped in the factory and subjected to dehydration and sintering in laboratory muffles contrasted with those obtained in the factory. It had a process in which samples were taken with interrupted cooking at certain temperatures, for example, at temperatures of 500º C and every 100º C up to temperatures normally used in each of these industries with their respective raw materials. We proceeded to observe with magnifying glass and metallographic microscope in order to observe the changes during the process, the absorption and mechanical resistance to compression were also analyzed, 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 difficulties of the previous treatment of polishing were solved with a hand sander and sandpaper of several graduations and the metallographic microscope suitable for observation which was managed with other laboratories. The interpretation was based on previous work in the area, literature, theories of thermodynamics and thermokinetics of gases in the cooking oven, as well as taking account of the current compositions for the manufacture of quality products for export.

The elements, minerals and crystallography that make up the bricks were analyzed by x-ray diffractometry.

Photos 1 to 6 Preparation of samples was done at the INTN and FIUNA.

RESULTS

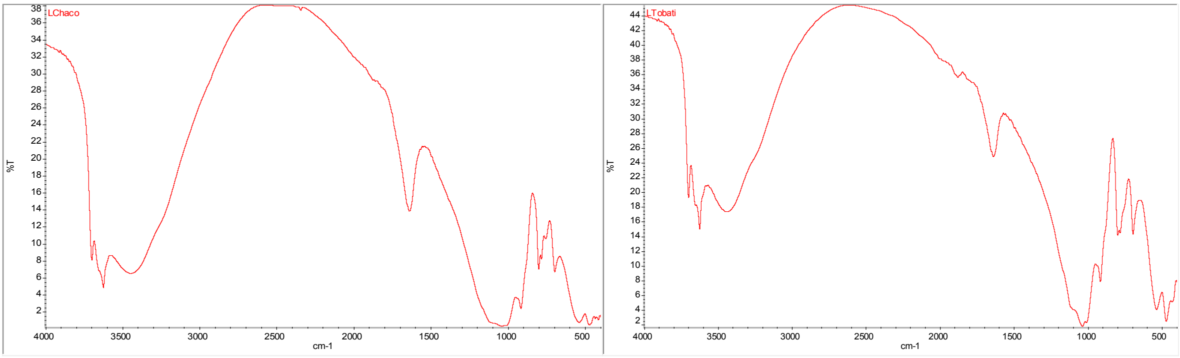
Elemento	M1 Lodo Chaco	M2 Hovaré Tobatí	M3 Lodo Tobatí	M4 Caolín Tobatí	M5. Tobatí 90% lodo+5%Caolín+5%Hovaré
CaO	0,65	0,17	1,16	0,55	1,076
SiO2	67,03	75,01	73,93	58,32	73,204
Al2O3	17,69	13,50	13,04	25,08	13,665
Fe2O3	6,25	5,43	3,99	4,61	4,093
SO3	0,07	0,07	0,09	0,08	0,089
K2O	0,85	0,22	2,25	0,62	2,067

Table 1: Component oxides of different clays analyzed (own elaboration XRD, 2018).

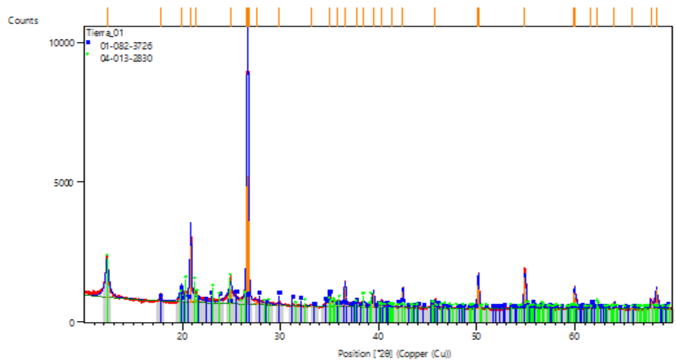
	RESULTADOS DE CLASIFICACIÓN DE SUELOS POR BOUYOUCOS			
M. Materia prima	Arcilla (%)	Limo (%)	Arena (%)	Clase de suelo
M1.Lodo Chaco	30,00	60,00	10,00	Franco Arcillo limosa
M2.Hovare Tobatí	38,31	17,78	43,91	Arcilla limosa
M3.Lodo Tobatí	32,48	6,00	61,52	Arena Arcillo limosa
M4.Caolin Tobatí	30,48	24,00	45,52	Arena Arcillo limosa
M5. Tobatí	32,67	7,49	59,84	Arena Arcillo limosa

Table 2: Results of soils texture classification by decantation or Bouyoucos.

Spectrophotometry results are the following:

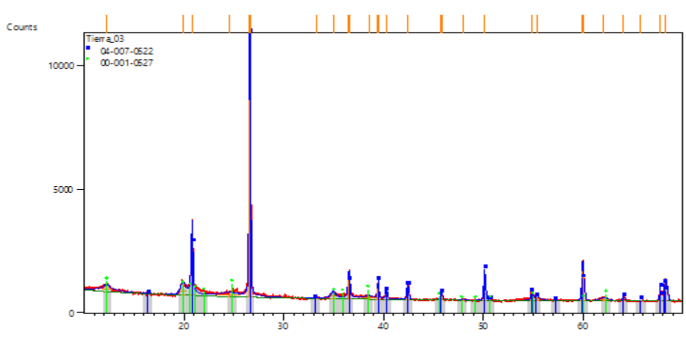


Graph 1: Infrared Spectrophotometry of Mud Chaco M1& M3 from Tobatí.



PatternListVisible Name	Ref.Code	Score	Compound Name	Displ.[°2θ]	ScaleFac.	Chem. 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	AluminumSilicateHydroxide	0,000	0,134	Al2 Si2 O5 (O H)4

Graph 3 and Table 3: X-Ray Diffractometry Results (XRD) of sample M1 from the Chaco.



PatternListVisible	Ref.Code	Score	CompoundName	Displ.[°2θ]	ScaleFac.	Chem. Formula
*	04-007-0522	91	Silicon Oxide	0,000	0,948	Si O2
*	00-001-0527	44	Aluminum Silicate Hydroxide	0,000	0,047	Al2 Si2 O5 (O H)4

Graph 4 and Table 4 X-Ray Diffractometry Results (DRX) of sample M3 from Tobatí.

ANALYSIS OF RESULTS

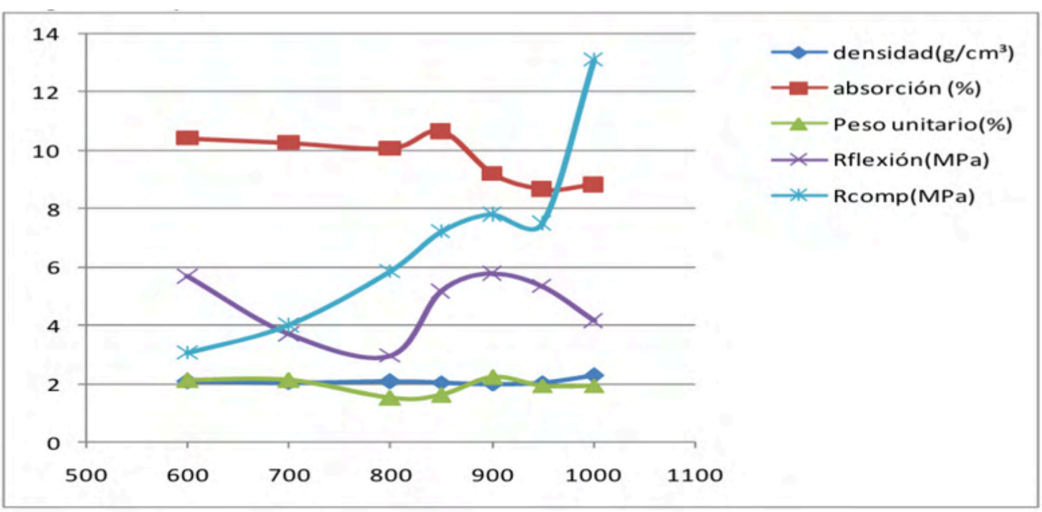
QUALITATIVE AND CUANTITATIVE ANALYSIS:

Photos 7 to 12: Bending tests, test pieces of bricks in press of 10kN capacity and full-scale bricks for compression and their corresponding press tests with a scale of 10,000 kgf. (own elaboration, Plastic Technology Lab at FIUNA and Construction Materials Lab., FIUNA).

Muestra	M1	Lodo Chaco	Rflexión a 500ºC=3,26 Mpa				
Propiedad/tempºC	600	700	800	850	900	950	1000
densidad(g/cm³)	2,07	2,05	2,07	2,06	2,01	2,06	2,28
absorción (%)	10,43	10,27	10,08	10,65	9,19	8,67	8,83
Peso unitario(%)	2,12	2,12	1,52	1,61	2,23	1,94	1,94
Rflexión(MPa)	5,68	3,69	2,95	5,17	5,78	5,34	4,15
Rcomp(MPa)	3,065	4,01	5,86	7,22	7,81	7,505	13,115

Table 5: Physical and mechanical results in reduced-scale bricks from Chaco mud M1,

Note: Bricks left in the oven until it cooled were over sintered and their mechanical properties decreased.



Graphic 5: Physical and mechanical results in reduced-scale bricks from Chaco Mud M1.

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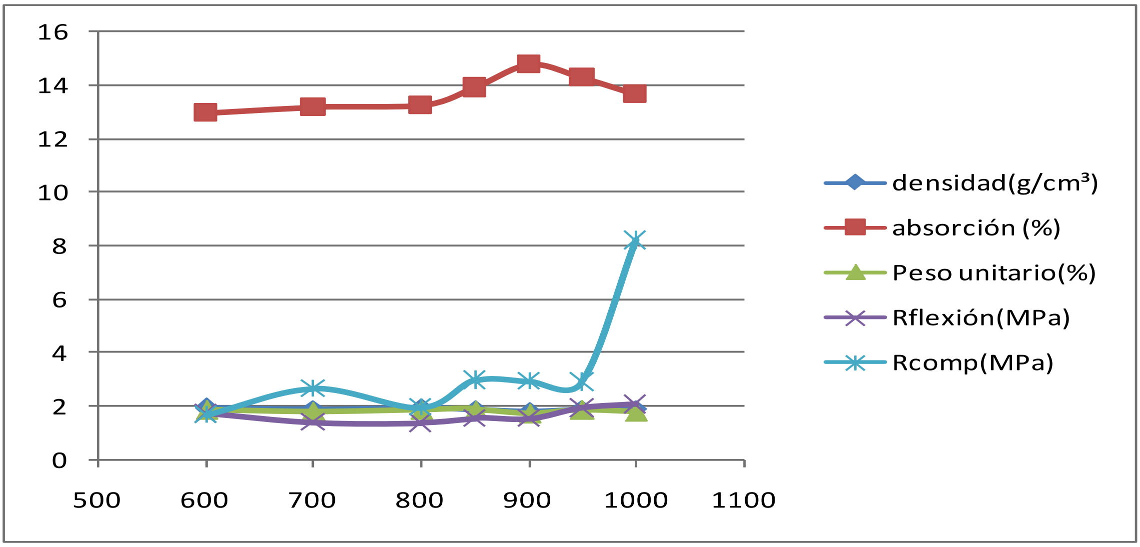
Muestra	M3	Lodo Tobatí	R flexión a 500ºC=1,29 Mpa				
Propiedad/tempºC	600	700	800	850	900	950	1000
densidad(g/cm³)	1,93	1,92	1,96	1,90	1,84	1,90	1,9
absorción (%)	12,95	13,19	13,27	13,91	14,78	14,29	13,68
Peso unitario(%)	1,87	1,81	1,87	1,91	1,74	1,86	1,79
Rflexión(MPa)	1,74	1,41	1,39	1,56	1,54	1,93	2,07
Rcomp(MPa)	1,695	2,65	1,975	2,99	2,97	2,92	8,21

Table 6: Physical and mechanical results in reduced-scale bricks from Tobatí Mud M3.

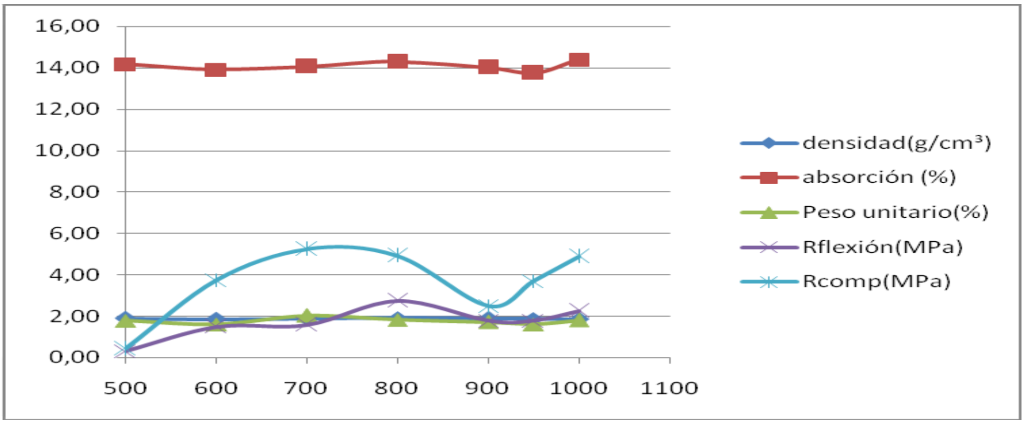


Graph 6: Physical and mechanical results in reduced-scale bricks from Tobatí Mud M3.

At 1000ºC a reasonable result for absorption, bending and compression strengths was obtained.

Muestra	M5	Tobatí	90%Lodo+5%Caolín+5%caolín				
Propiedad/tempºC	500	600	700	800	900	950	1000
densidad(g/cm³)	1,90	1,86	1,88	1,92	1,90	1,88	1,86
absorción (%)	14,17	13,91	14,05	14,29	14,02	13,75	14,40
Peso unitario(%)	1,79	1,61	2,03	1,83	1,72	1,61	1,80
Rflexión(MPa)	0,31	1,49	1,58	2,74	1,79	1,79	2,25
Rcomp(MPa)	0,44	3,74	5,24	4,92	2,50	3,695	4,885

Table 7. Physical and mechanical results in reduced-scale bricks of sample M5 fromTobatí.



Graph 7: Physical and mechanical results in reduced-scale bricks of sample M5 from Tobatí.

Compresión en ladrillos

Deflex mm	Carga M1 C (kgf)	Carga M5 T (kg)	Carga Ref (kg)
0	0	0	0
0,02	1000	220	300
0,03	1750	335	450
0,04	2500	450	600
0,05	3250	600	800
0,06	5000	750	1000
0,07	6000	975	1250
0,08	7000	1200	1500
0,09	8000	1800	2250
0,1	9000	2400	3000
0,125	10000	3750	4000
0,15	10400	5100	5000
0,175	10800	6450	6220
0,2	11200	7800	7440
0,225	11600	8300	8000
0,25	12000	8600	8500
0,275	12500	9000	9000
0,3	13000	9300	10200
0,4	14500	10200	12000
0,5		11400	12360

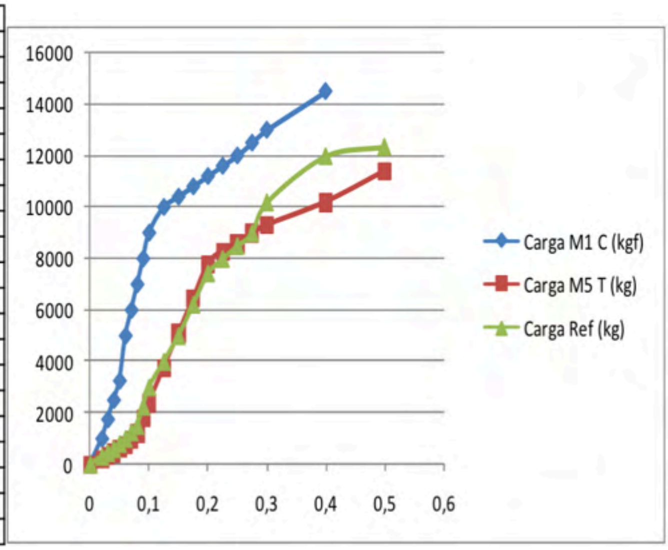
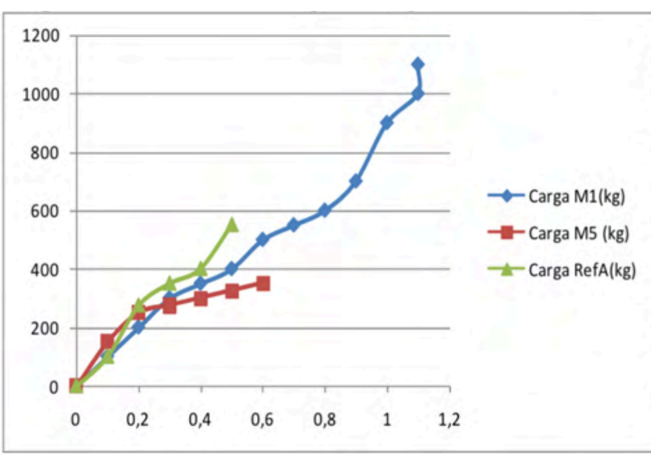


Table 8 & Graph 8: Compression load (kgf) versus deformation (mm) for construction bricks.



Deflex mm	Carga M1(kg)	Carga M5 (kg)	Carga RefA(kg)
0	0	0	0
0,1	100	150	100
0,2	200	250	275
0,3	300	275	350
0,4	350	300	400
0,5	400	325	550
0,6	500	350	
0,7	550		
0,8	600		
0,9	700		
1,0	900		
1,1	1000		
1,1	1100		

Graph 9 & Table 9: Bending load (kgf) versus Deformation (mm) in samples of bricks.

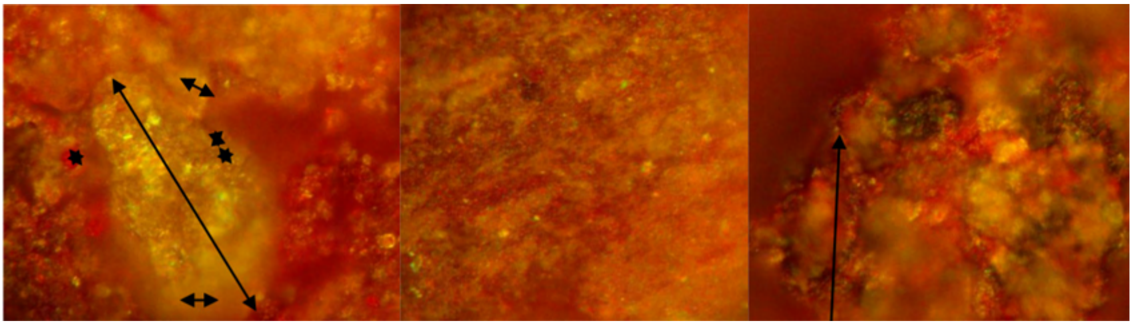
Ladrillos	Rotura a flexión	E	Rotura por corte	G	U	Resistencia a compresión
M1C	59.78	3890	15.94	1298	0.5	110.6
M5T	41.61	5557	10,49	1854	0.5	78.5
Ref A 3 agujeros	19.72	6113	4,82	2038	0.5	98.6

Table 10: Results of bending and compression tests of full-scale bricks in (kg/cm2).

THEORETICAL MODEL OF ADHERENCE DETACHED DURING CERAMIC BRICK PROCESS

Seen in the metallographic microscope later and noticed when making the bricks on a reduced scale of approximately 6x2x1 cm, that when sand is moistened it swells together with water and silts and clays also participate, they are kneaded and then compacted being incorporated as part of the internal structure of the sand and also remaining on the outside of the largest and swollen grain (the sand). Subsequently, these positions are consolidated in the oven at the sintering temperature.

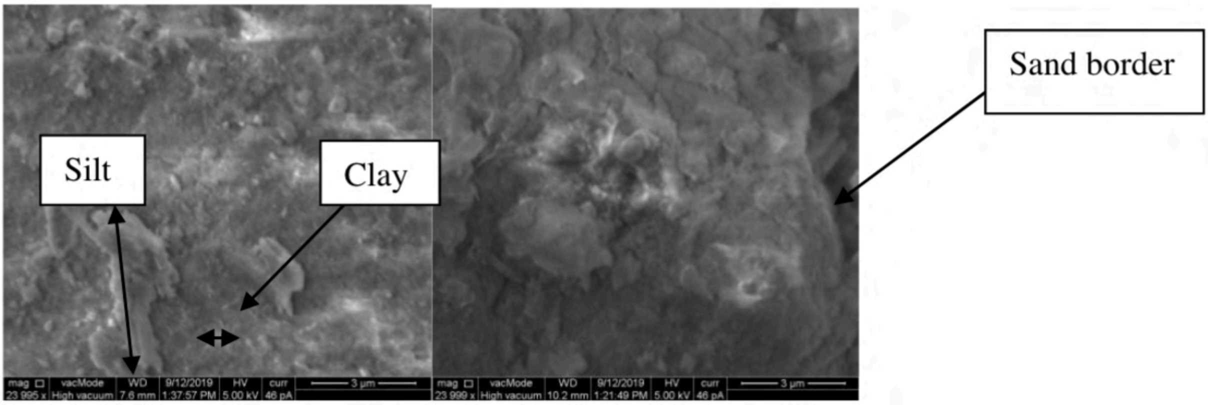
Graph 10:Adherence model based on size of components. (note mainly the clays pricking and being embedded in sands and silts. Elaborated by the author, 2018).



Photos 13 to 15; Samples of M5 bricks from the area of Tobatí, you can observe at 700, 800, and 1000º C temperatures similar behaviour but at different temperatures than those of of M1 bricks (100X).

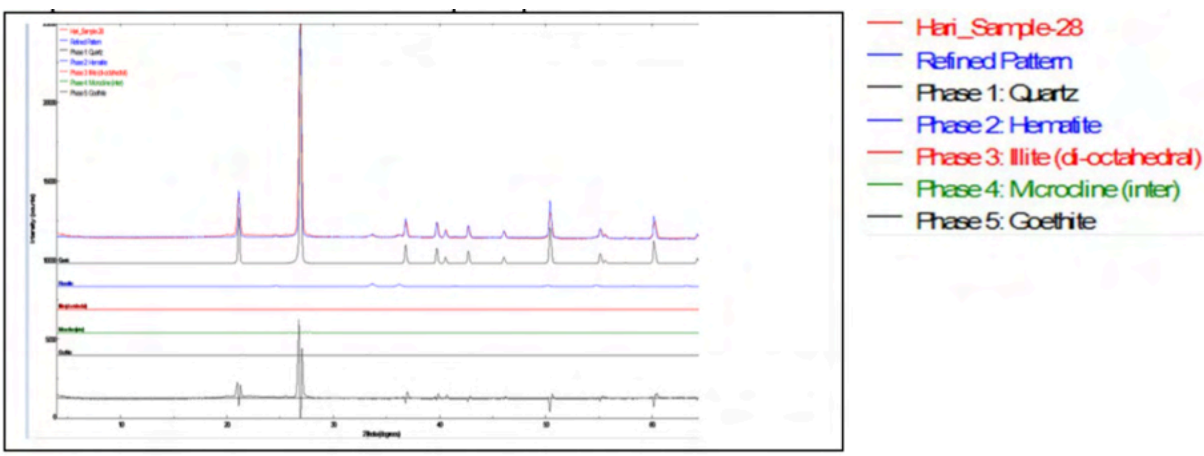
(In photo 45 it´s shown sand, silt and clay sizes. Grain and Sand borders are nearly confused in photo 47)

Figures 22 and 23: Elemental structures of Kaolinite and Illite.



Photos 16 & 17: Results from Tobatí M5 sintered at 700ºC, observed with SEM at Tulsa University, Oklahoma, USA.

You can observe the serie of small grains increased from 12.000 to 50.000 times by electron microscopy between cavities and bumps, tanking into account the original sizes of original components suchas as: 0,06 to 2 mm sands, 0,002 to 0,06 mm silts and less than 0,002 mm clays which were somewhat compacted int their original measurement and forming bigger grains after reducing the void and increasing the grains with the temperature.

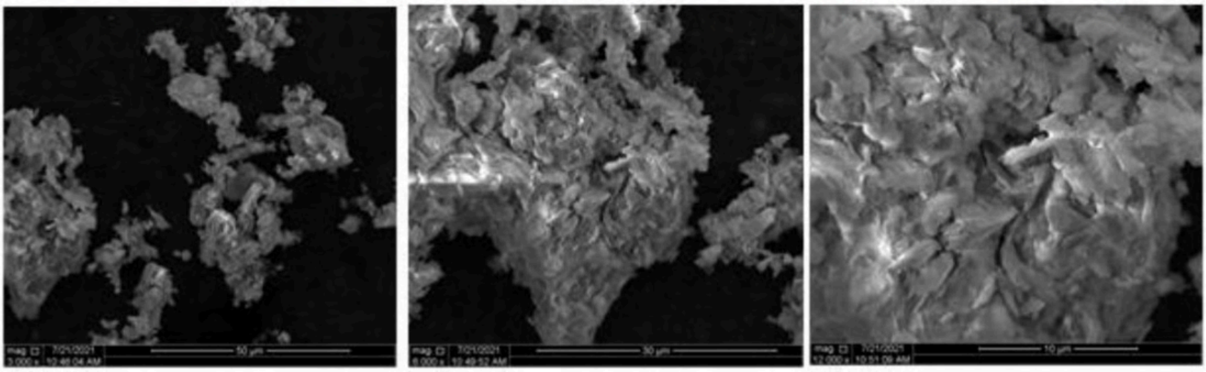


- Title: Hari_Sample-28_FullR - Rock
- Range: 3 - 65, Inc: 0.04, Time: 2, NPts: 1551.
- Residual Errors: 30.21% Goodness: 5.55
- Quantitative results based on all phases used in the refinement.
- The sum of the weight fractions is normalized to 1.0
- Phases wt% (ESD) Size(A)
- (1) Quartz 95.2 (0.4) > 2000
- (2) Hematite 3.5 (0.1) 460
- (4) Microcline (inter) 0.6 (0.0) > 2000
- (3) Illite (di-octahedral) 0.5 (0.0) > 2000
- (5) Goethite 0.3 (0.0) > 2000

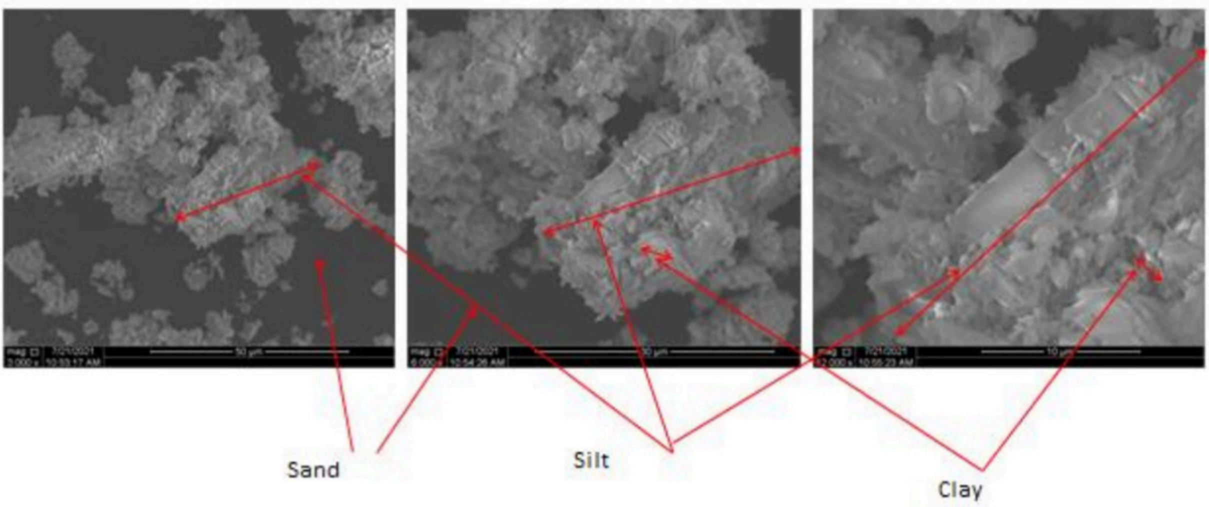
3C and 18C bricks from Chaco Mud
19 T brick from Tobatí Mud
Scale 1:5 aprox

Graph 11: XRD results in Tobatí samples up to 700 ºC

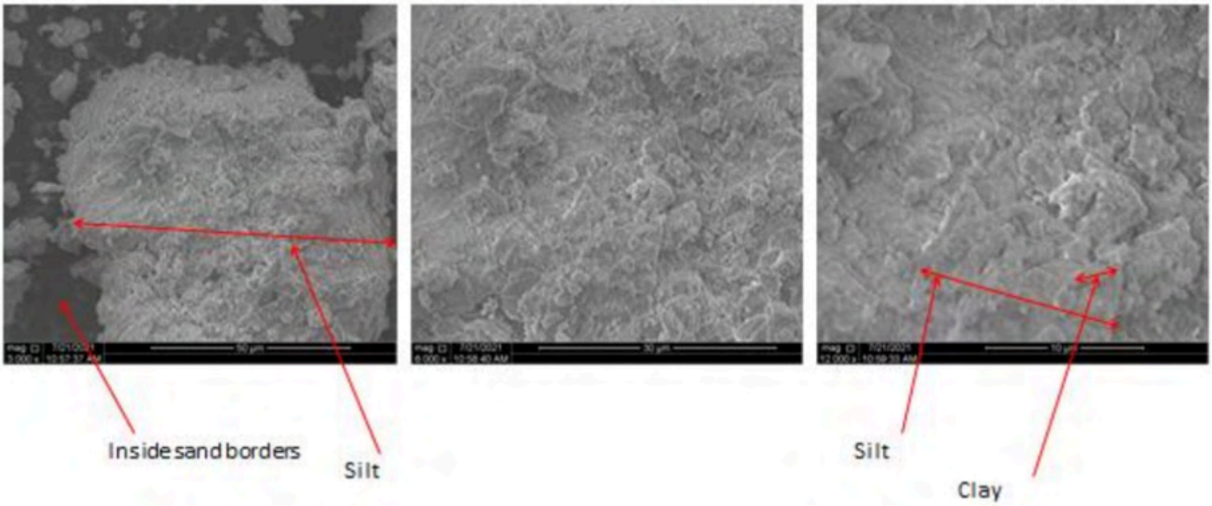
SEM images of Sample # 3C (Densifying)



SEM images of Sample # 18C



SEM images of Sample # 19T



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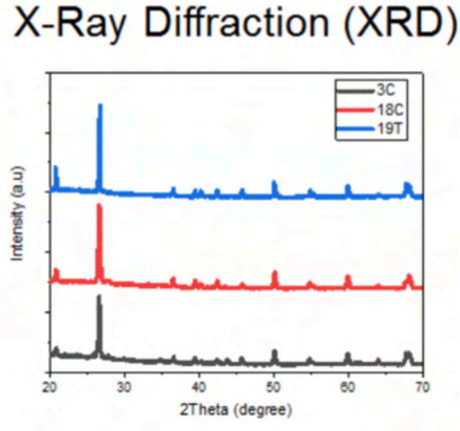
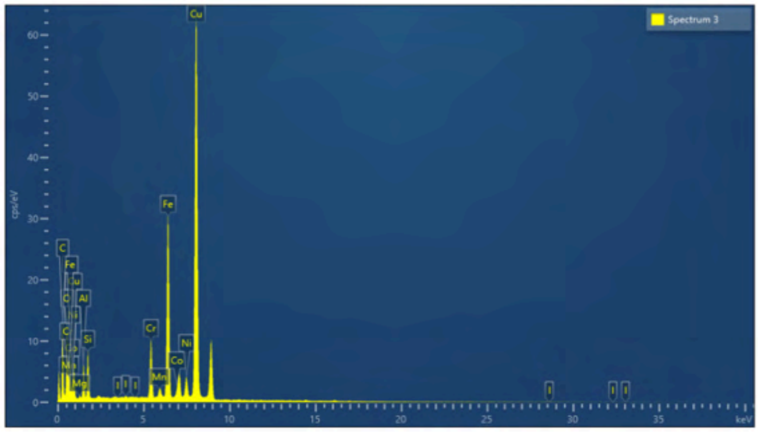
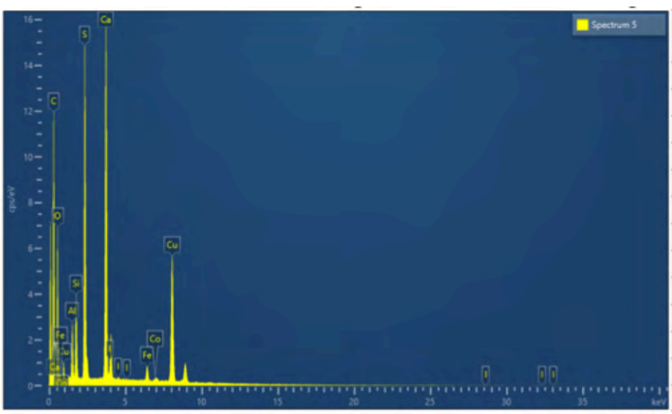




Three peaks at 2θ values of 44.585, 64.980, and 82.297 deg corresponding to (110), (200), and (211) planes of iron were observed and compared with the standard powder diffraction.



Spectrum Label	Spectrum 3	Spectrum Label	Spectrum 5
C	9.15	C	29.31
O	3.62	O	7.55
Mg	0.13	Al	2.63
Al	1.67	Si	3.89
Si	2.44	S	17.26
Cr	5.21	Ca	22.14
Mn	0.30	Fe	1.41
Fe	19.30	Co	0.24
Co	0.84	Cu	15.56
Ni	2.47	I	0.00
Cu	53.91	Total	100.00
I	0.96		
Total	100.00		

CONCLUSION

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

"In a first stage of wetting and kneading, the sand receives the contribution in its interior and incrustations in its exterior of clays and silts, when compacting these they mix more intimately forming fundamental grains of different sizes. During cooking, these grains grow due to thermodynamics, causing expansions of the fundamental grains that unite with their similar ones, welding and sewing together, maintaining the difference in their sizes, reducing or almost eliminating the voids and forming larger microscopic and nanoscopic grains" .

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