Clays for Brick Manufacturing in Actopan, Hidalgo: Physical, Chemical and Mineralogical Characterization¹

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Samples of clays from Actopan, Hidalgo employed in brick manufacturing are physical, chemical and mineralogical characterized. Transmitted polarized light microscopy showed a uniform particle size with grain morphology characteristic of euhedral crystals with quartz, feldspars, nontronite, and iron oxides particles. Scanning Electron Microscopy revealed 75 μm to 90 μm wide subhedral structures formed by particles from 2.0 μm to 5.0 μm ; and rombohedrales forms 40 μm wide, 70 μm long, constituted of silicon, aluminum, iron, titanium, calcium, minor amounts of potassium, magnesium, and sodium. Minerals such as quartz, albite, cristobalite, calcium and Hematite phases were recognized by X-Ray Diffraction technique. Chemical analysis by atomic emission spectrometry with Inductively Coupled Plasma confirmed this mineralogy composition while laser granulometry method found the same particle size. Grain size analysis determined submicrometric dimensions, and multimodal type curves, that can be interpreted as the mixing of two or more different mineral phases in each sample.

Keywords: Clays, Mineralogical characterization, Optical microscopy, Scanning Electron Microscopy (SEM), X-ray diffraction.

1. Introduction

A topic of great technological relevance is the optimization of the natural resources to produce diverse industrial materials, because it promotes the competitiveness in domestic and international markets. To do this, alternative technologies that can modify materials from natural beds in the elaboration of products with optimum improved properties for specific applications are required. One of the most popular materials are clays that constitute 25% of Earth's crust¹⁻³. Since the dawn of humanity, clays have been used in tablets for writing, construction bricks, monuments, pottery, containers, ceramics, the mud for drilling of wells that act as a lubricant, sealant, vehicles for waste removal, the binder in foundry sands, in civil engineering works, as filter media and bleaching, for catalysts, as a plastic paste, etc.2-3. In particular, clays are of high commercial interest in the construction industry as brick elements, and there is a continuous effort to modify its constituents looking for cheaper, lighter, non-igneous, and in general better materials^{1,4}. To modify the natural materials is required to know in detail its structural characteristics, chemical composition and properties⁵⁻⁷, which can be achieved using modern analytical techniques such as polarized light microscopy, Scanning Electron Microscopy, X-Ray Diffraction, Infrared Spectroscopy, among others⁸⁻¹¹. For the specific case of brick manufacturing, it is important not only to characterize the microstructural characteristics of the clays, but also its macrostructural behavior¹²⁻¹⁴.

Clays can be defined based on its size, this includes all particles with diameter less than 4 µm^{2,3}. They can also be described by its composition, comprising hydrated silicates of aluminum belonging to the groups of kaolin, and illites. Alternative, they can be characterized by its petrography, as sedimentary rocks formed from one or several minerals, rich in hydrated silicates of aluminum, iron magnesium, hydrated alumina, or ferric oxide, with a predominance of colloidal particles. Almost all clays are silicates with foliated structure from the Muscovites group, have crystal structure and are negative biaxial. Clays have an excellent plasticity when they are hydrated, are very rigid when burned at a high temperature, and brittle in dry state. In nature, they often appear as mixtures of several clay minerals with abundant impurities and pigmentation. The clay minerals are compounds formed by hydrated aluminum silicates, resulting of the hydroxylation

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of the feldspars. Clays consist mainly of two structures: a tetrahedral one, which is comprised of four silicon atoms and other octahedral unit formed by six hydroxyls surrounding aluminum or magnesium atoms. Usually, both structures are combined to form sheets. If some of the environment water is adsorbed, it is linked to the crystal lattice. On the other hand, the form of hydroxyl OH can enter the constitution of this same lattice. The crystal building is presented in the form of flakes; whose tiny dimensions causes an enormous complexity in its study.

Characterization of clays is difficult because the structures that constitute them are chemically very similar⁸⁻¹¹. Due to this are required different analytical methods for a detailed description. Clays are not consolidated or little hardened, for example, natural lands contain an excess of particles of sizes lower than 1/256 m in diameter. Moreover, 10% of mineral clays are shaped by other materials such as feldspars, biotite, quartz, pyroxene, zirconium, zeolites, and carbonates (ankerite, calcite, and dolomite).

In Actopan, Hidalgo, México; it is common to manufacture red bricks from the sandy material of the region. This is the natural material most used for construction. This material is not well characterized, it is suspected that it is composed of clays with a high content on feldspars and silico-alumina, thus with a higher SiO₂ proportion than Al₂O₃. To understand the properties of clays used for this brick manufacturing, in this paper different clay samples of Actopan, Hidalgo are physical, chemical and mineralogical characterized using transmitted polarized light microscopy (TPLM), Scanning Electron Microscopy (SEM), chemical analysis by atomic emission spectrometry with Inductively Coupled Plasma (ICP), and X-Ray Diffraction (XRD).

2. Materials and analytical methods

2.1. Location and geological setting

The clay samples come from Actopan, Hidalgo, Mexico. Actopan is located in the south-central part of the province of Hidalgo, a state in the central section of Mexico. Its geographic coordinates are: 20°16′06.71" North latitude and 98°56′40.38" West longitude (see Figure 1). The samples correspond to the andesitic domain, which includes thorns formations and group Pachuca, of the Oligocene age. They are made up of lavas, breccia and tuffs of andesitic-dacitic composition with some rhyolitic interdigitations (Zumate formation) in Mount Pachuca-Real, calc-alkaline affinity and the rhyolitic domain consisting of formations: Don Guiño, unit rhyolitic and rhyolite knives, age Oligocene-Pliocene. It consists of rhyolite-rhyodacite with structure of Tuff, Ignimbrite and gap, calc-alkaline character with alkaline trend¹⁵.

Sampling was carried out in the slopes of the gully (in this case, anthropogenic), in the form of channel with a

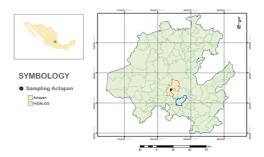




Figure 1. Plane location of the sampling area in Actopan, Hidalgo, Mexico.

length from 1.00 m to 1.50 m by 0.30 m of thick and small wells with a depth of 0.25 m to 0.50 m, with weight of 3.0 Kg (see Figure 2). The samples were taken with a plastic shovel, labeled and stored in plastic bags for transportation and subsequent preparation for the different analyses carried out. Reference of the sampling point was performed using a geographic locator satellite (GPS). The sampling was randomly so that clay's samples were representative, considering mainly their physical characteristics as color megascopic: alteration, hardness, texture, particle size, among others (Figure 2).





Figure 2. Sampling clay site in the form of channel.

2.2. Mineralogical characterization

Mineralogical characterization of the samples was performed by polarized light microscopy in thin films with transmitted light (anhydrous), scanning electron microscopy (SEM), and X-rays diffraction (XRD). Through this study, SEM determined primary mineral soil species (mineral associations of clay), as well as products of alteration (secondary mineral associations and/or products of weathering). Mayor, minor and signs of minerals' crystalline phases were determined by an X-ray diffractometer BRUKER® model D8 Advance

Da Vinci. This equipment of simultaneous detection works in the range of 20-110 degrees. For optical polarization was used an X-ray of copper tube Observations by OMP BX41 (UAEH) model from the Microscope Olympus® brand. SEM JEOL® model JSM -6610LV coupled to the SEM X-Ray Microanalysis was employed for the SEM observations.

2.3. Sample preparation

Clay samples were placed in plastic containers (basins) distributing them across the surface of the container, drying them at room temperature. Then, each sample was homogenized and a representative sample was drifted by quartering.

In the microscopically analysis by OMP and SEM, thin films were obtained by anhydrous method (intertexture relations). Homogenized and sieved to -200 mesh fractions were observed with light transmitted in nicoles for the OMP and they were cross and parallel. For the SEM, an amount of particles from clay samples were placed in a sample carrier with carbide tape and the sample was covered with gold to make them more conductive.

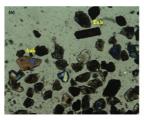
Quantitative chemical analysis of traces of elements was done by an old spectrometry coupled induction plasma (ICP), taking part of the sample that was sifted to -100 mesh. For the X-Ray Diffraction (XRD), 10 g of the sample were used and sieved to -200 mesh.

For grain size analysis (Ro-tap and laser), 100 g of sample was taken and held a screening in a set of 5 screens (# 120, 200, 325, 400, 500, - 500) for the mechanical particle size analysis (Ro-tap), which shows the proportion in particle size and 1 g for particle size laser that allows determine the size distribution of particles suspended in a liquid medium.

3. Results and discussion

3.1. Transmitted Polarized Light Microscopy (TPLM)

Transmitted polarized light microscopy allows defining the textural relationships and associations of the mineral phases for metallic minerals, mainly oxides and hydroxides of iron (limonite, hematite, among others) and non-metallic minerals (silicate)¹⁶. Using this technique, it is shown that the examined clay samples have a not homogeneous particle size, with grain morphology from euhedral to anhedral according to the degree of alteration that present (see Figure 3a). The mineralogical composition detected by this technique determined that tested clay samples have abundant quartz (Figure 3b), feldspar (plagioclase Anorthite-albite solid series), chlorite and oxides of iron (Figure 4). Feldspar particles did not present morphology alteration from euhedral to subhedral type, as their alteration growths, particles appeared rounded up becoming anhedral usually associated with variable amounts of iron oxides and chlorite. Iron content is generally very abundant and turns from a dark grey to black (opaque minerals), this mineral sometimes exhibits an oxidation penetrative way.



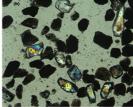


Figure 3. Transmitted polarized light microscopy photomicrographs showing particles euhedral (a), anhedral (b) and quartz. The micrographs are cross nicoles, objective 4X, and field horizontal 2.00 mm photograph.



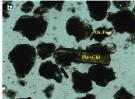


Figure 4. Transmitted polarized light microscopy photomicrographs showing (Feld) feldspar and plagioclase (Pla), chlorite (chl) and (Ox-Fe) iron oxides. The micrographs are cross nicoles, objective 10 X, and field horizontal 0.98 mm photograph.

The source of these banks of detrital material can be felsic volcanic rock (a qualitatively interpretation) that due to its abundant content of feldspar can led to clays. Present chlorite alteration is derived from a hydrothermal process occurred in the rock, not from a type of alteration arising in the material bank. The presence of iron oxides is evidence that rocks carriers of this material have a preferentially composition intermediate to basic (basaltic-andesitic type). These correspond to the domain andesitic composition are andesite basalt to rhyodacite, texture afanitic to porphyric with interstratifications of tuff and gaps in spill with poorly consolidated horizons, levels rhyolitic known as training Zumate¹⁷ and bodies andesitic to latitic in the form of dikes. Its affinity is calc-alkaline and his age has only been determined in the region of Zimapan that corresponding to the Oligocene (38 to 27 ma)18. To a lesser extent are felsic (of type rhyolitic-dacitic) justifying the presence of feldspar and quartz which correspond to the rhyolitic domain, which is composed of massive rhyolite and ignimbrites and some break-through horizons, evidence of small domes (San Clemente). This domain has a calc-alkaline character; however, there is evidence of an alkaline influence shown by the high content of alkali to the North of Ixmiquilpan. It contains mineralization of Ti that can demonstrate a high magmatic differentiation. The age assigned by dating (K/ Ar) ranges from 38 to 27 ma¹⁸, which corresponds to the

Oligocene, however other authors stablished the age until the Pliocene¹⁵.

3.2. Chemical analysis (ICP)

Table 1 presents the results of the chemical analysis by atomic emission spectrometry with inductively coupled plasma (ICP) of five clay samples from two brick factories in the same area (Figure 1). The major elements calculated based on oxides have very heterogeneous concentrations of Al₂O₃, CaO, K₂O, MgO, MnO, FeO, Na₂O, TiO₂, and SiO₂. It should be noted that concentrations of SiO₂ and CaO are slightly larger in ACT-4 and ACT-5 samples. These chemical analyses do not mark a zoning relative to their values in concentration (Figure 5) allowing it to interpret the rock origin type. The chemical analysis shall be used to assess the quantities of Fe, Al and Si, which are the elements that help or affect the manufacture of bricks (plasticity) based on the percentage used.

The great amount of FeO, as Hematite Fe₂O₂, limonite FeO(OH)·nH2O, and goethite FeO(OH) reflex the presence of oxides and hydroxides in the clay samples. These products of alteration (salts efluorescents) are similar to those that occur in waste caused by mining activity¹⁹. They correspond to a hydrothermal alteration from subsequent (source rock) in situ (supergene alteration) to the originated by meteoric leaching. Only, TiO, is achieved by determining fields compatible with different areas of ACT1, ACT2, ACT3, ACT4, and ACT5 sampling, so Ti can be an indirect liner (see Figure 6). Regarding the trace elements such as Pb, Zn, Cu, Cr, Co, V and Ba, these arise in heterogeneous and low concentrations ranging from tens to hundreds of mg/Kg (Table 2). Samples have significant values of Pb, Zn and Cu, which are characteristic elements of primary mineral phases, product of some mineral ore leached and eroded. This is mainly due to the process of hydrolysis and alteration as the silicified, among others, of the present metal mineralization, which correspond to the surrounding areas of the mining district of Pachuca-Real on the mount, in which the mineralization is low sulphidation epithermal. Therefore, these associated factors of weathering thus also give the formation of secondary and even tertiary mineral phases.

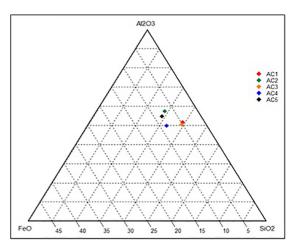


Figure 5. Ternary diagram of FeO, SiO₂, and Al₂O₃ concentrations without zoning in incompatible fields in the two areas.

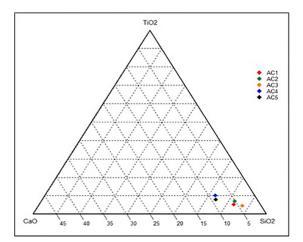


Figure 6. Ternary diagram of CaO, TiO₂ and SiO₂ concentrations with zoning in compatible fields in the two areas.

3.3. X-ray diffraction (XRD)

The most representative clay samples of the area of Actopan, Hidalgo, exhibit the crystalline mineral phases given in Table 3. X-Ray Diffraction (XRD) found quartz (SiO₂) and albite (NaAlSi₃O₈) as the majority crystalline mineral phases, which confirm the nature of the outcropping rocks that influence in the region and which correspond to the

Table 1. Chemical analysis of clays from Actopan, Hidalgo based on oxides by atomic emission spectrometry with inductively coupled plasma (ICP).

SAMPLE	Al ₂ O ₃ %	CaO %	K ₂ O %	MgO %	MnO %	FeO %	Na ₂ O %	TiO ₂ %	SiO ₂ %
ACT 1	17.75	3.02	2.46	1.44	0.06	3.27	2.62	1.35	47.86
ACT 2	18.52	2.35	1.50	1.92	0.08	4.57	1.70	1.59	41.45
ACT 3	15.90	1.94	2.00	1.34	0.07	3.29	0.00	1.09	43.78
ACT 4	14.83	3.90	1.21	1.89	0.10	5.12	0.00	2.30	39.70
ACT 5	15.76	3.83	1.08	1.99	0.10	4.79	2.80	1.69	37.07
$Average \pm SD$	17 ± 2	3.0 ± 0.9	$1.6\pm\!0.6$	1.7 ± 0.3	0.08 ± 0.02	4.2 ± 0.9	1 ± 1	1.6 ± 0.5	42 ± 4

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SAMPLE	PB (mg/kg)	Zn (mg/kg)	CU (mg/kg)	CR (mg/kg)	Co (mg/kg)	V (mg/kg)	BA (mg/kg)
ACT 1	11.43	32.79	344.76	32.79	6.95	44.21	398.91
ACT 2	11.38	73.23	287.98	71.50	11.38	62.59	394.85
ACT 3	15.22	41.92	337.57	31.94	7.24	36.68	337.08
ACT 4	6.97	48.53	369.09	151.57	21.90	104.28	272.03
ACT 5	12.18	50.20	282.55	132.95	15.90	73.81	246.52
Average ± SD	11 ± 3	49 ± 15	324 ± 38	84 ± 56	13 ± 6	64 ± 27	330 ± 70

Table 2. Chemical element tracing analysis by ICP of clays from Actopan, Hidalgo.

andesitic domain, both the rhyolitic. It should be noted that the abundant content of quartz determined by this technique on samples, suggests another source of material that can be derived from the levels of sand ledge interspersed in the volcanic horizons of Plio-Quaternary in the region¹⁵. These horizons may correspond to a continental flysch in the area.

Certain minority mineral phases are similar and consist of albite and cristobalite and it was only determined at the Na_{0.3}Fe₃+2(Si,Al)₄O₁₀(OH)₂·nH₂O, and Hematite, Fe₂O₃ as mineral in the form of evidence phase (Figure 7). In the case of cristobalite, this occurs as a Polymorph of quartz, while the other species belong to the family of the phyllosilicates, sometimes hydrated or ferric and are the product of the meteoric or hydrothermal alteration of feldspars and pyroxenes and institutions constituting the rocks containing them (hosting rock).

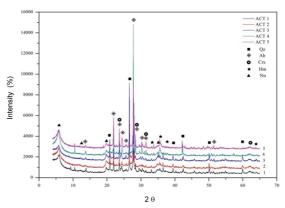


Figure 7. X-ray Diffraction patterns in the clay samples.

Table 3. X-Ray Diffraction (XRD) of the clay samples of Actopan, Hidalgo.

Sample	Mineralogy	Majority phase	Minority phase	Indication
	Quartz	X		
	Albite		X	
ACT-1	Cristobalite		X	
	Nontronite		X	
	Hematite			X
	Quartz	X		
ACT-2	Albite		X	
AC1-2	Hematite			X
	Nontronite		X	
	Quartz	X		
ACT 2	Albite		X	
ACT-3	Nontronite		X	
	Hematite			X
	Albite	X		
	Quartz		X	
ACT-4	Cristobalite			X
	Nontronite			X
	Hematite			X
	Albite	X		
A CIT 5	Cristobalite		X	
ACT-5	Nontronite			X
	Hematite			X

Table 4	SEM nunctue	I quantitative ch	emical analy	cie in crystale	of clave from	Actonan

Element	ACT1-B	ACT1-D	ACT5-E	ACT5-D	ACT2-D	ACT2-C	ACT3-A	ACT3-B
		W % of the microanalysis						
О	33.58	33.01	41.79	52.86	57.78	35.00	52.59	45.69
Na	0.52	-	-	0.39	-	-	-	-
Mg	0.54	0.88	1.11	1.74	0.77	3.38	0.81	2.24
Al	3.60	5.23	4.54	7.16	4.26	3.68	6.44	8.70
Si	8.39	9.22	11.90	16.79	18.51	14.00	19.20	20.61
K	0.45	0.55	0.46	0.44	0.24	0.41	1.30	1.15
Ca	2.07	0.50	1.07	1.12	0.37	1.19	1.05	2.09
Ti	14.42	3.70	-	0.24	-	-	-	-
Fe	21.35	36.65	3.33	3.56	2.06	14.87	2.90	4.59

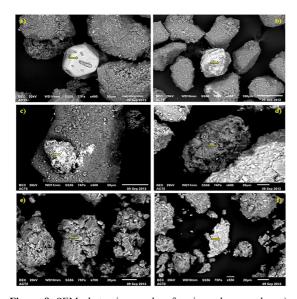


Figure 8. SEM photomicrographs of various clay samples. a) and b) subhedral morphologies; (c), (d) and e) morphology detail and variation in size of silicate particles; f) rhombohedral structure corresponding to dolomite. Composition determined by microanalysis (table 4).

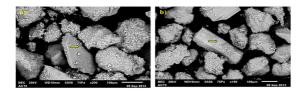


Figure 9. SEM photomicrographs with secondary electrons: a) and b) morphologies and structures laminar type, subhedral and silicates (in feldspars) euhedral, composition is determined by its respective microanalysis (table 4).

Table 5. Modal values obtained by laser diffraction particle size distribution curves

Sample	Modal values (µm)			
ACT1	185, 20, 5, 1			
ACT2	185, 23, 1			
ACT3	103, 223, 1, 20			
ACT4	203, 100, 1			
ACT5	103, 88, 1			

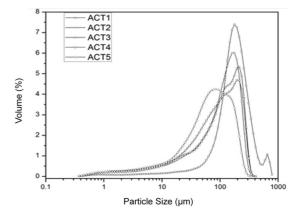


Figure 10. Particle size distribution of the clays obtained by laser diffraction.

3.4. Scanning Electron Microscopy (SEM)

SEM of the clay samples from the area of Actopan, Hidalgo, produced the results reported in Table 4 and Figure 8. Different morphologies appear in Figure 8, like the subhedral structure of 75 µm wide and 90 µm long consisting of titanite (CaTiSiO₅) (shown in the center of Figs. 8a and 8b), and the clumps that have anhedral forms of the order of 100 µm wide and 150 µm long (surrounding the surface particles smaller than 5.0 µm) mainly composed of Ti, Fe, O, Na, Mg, Al, Si, K, and Ca. In Figures 8 c, d, e, are shown with greater detail structures of the order of 40 µm wide and 80 μm long, with particles from 2.0 μm to 5.0 μm. In the center of Figure 8f can be seen a rhombohedral structure of 40 µm wide and 70 µm long, that can correspond to a dolomite [CaMg(CO₃)₂]. It should be noted that the determination of the mineral species in the clay from the microanalysis was difficult due to the weak stoichiometry contaminated by the presence of significant amounts of impurities like Na, Mg, K, and Ca, which correspond to complex groups of silicates (phyllosilicates and tectosilicates mainly).

In Figures 9a and 9b appear anhedral structures of 150 μ m wide and 200 μ m long, which correspond to small size particles (5.0 μ m in diameter). In the center of Figure 9a is observed a subhedral structure, presenting a flat face

(laminar structure) of 300 μm long and 120 μm wide (with a thickness of 50.0 μm), while in the center of Figure 9b are presented one euhedral structure of 75 μm width, 300 μm long, and 25 μm thick, with a flat surface face. In both cases, these structures belong to plagioclase, which are a subgroup of alkali feldspars that constitute a solid solution, an isomorphic series, between albite (NaAlSi₃O₈) and Anorthite (CaAl,Si₃O₆).

3.5. Determination of particle size

Grain size analysis is the mechanical procedure that separates the constituent particles of an aggregate per sizes. The retained mass is expressed as a percentage of the total sample and the retained percentage expressed both partial and accumulated in a grading curve, the percentage of material passing each screen in relation to their diameter. With this method is determined the textural class from each of the samples, thus allowing a classification for each zone. For the area Actopan-Arenal (ACT-ARE), it was determined a sandy texture to sandy clay. Table 5 and Figure 10 show the results of particle size for the clay samples, determined by laser diffraction. All samples have submicrometric size and all the curves are multimodal type, i.e., presented two or more modal values, interpreting it as a mixture of two or more different mineral phases in each sample.

4. Conclusions

Clays from the region of Actopan consist of a mineralogy composed of quartz, feldspar, plagioclase, nontronite, and iron oxides. As XRD found, the principal mineral phases are quartz, and albite. Microanalysis showed different morphologies ranging from subhedral to anhedral, mainly of feldspars. The composition is complex (phyllosilicates and tectosilicates) due to the presence of impurities in the crystalline structure. Moreover, some particles correspond to secondary and tertiary products and are alterations caused by the factors of weathering association. Laser diffraction confirms that the samples are submicrometric in size and have curves of multimodal type, which can be interpreted as the mixture of two or more mineral phases. Knowledge of the characteristics of this type of material will allow to modify them for obtaining better properties for brick manufacturing.

5. Acknowledgements

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