

Physicochemical and Mineralogical Characterization of Moroccan Clay of Taza and Its Use in Ceramic Technology

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Abstract: This study concerns the results of Physicochemical and mineralogical characterization of a white clay located in Taza region in Morocco and its use in the ceramics industry. Several techniques were used; in particular X-ray diffraction (XRD), scanning electron microscopy coupled with EDX microanalysis (SEM-EDX), differential thermal and gravimetric analyses (DTA-TGA) and finally infrared Fourier transform (FTIR) and X-ray fluorescence (XRF). The first objective of this work is to put a new line of research that deals with the use of clay in ceramic technology. The second objective was to develop gels of oxides of high purity from these clays. we can say that the white clay of Taza has the same characteristics of clays used in the ceramics industry (medium heat loss, low shrinkage, good flexural strength and good behavior in plasticity), this white clay Taza adding 0.57% sodium carbonate is sufficient to have a good deflocculation and the viscosity is minimum corresponds to the stability of the slip, in his introduction to a formula of slip was successful with a rate of 35 to 45%. The SEM-EDX, X-ray, chemical analysis and Infrared spectroscopy demonstrated and allowed us to identify the different minerals that make up the white clay, compared with the available data, we identified illite and kaolinite as clay minerals, other minerals present as impurities major are quartz, calcite, dolomite and feldspar. These results show the important features to justify its use in the ceramic industry.

Keywords: Clay, Ceramic, DTA, FX, XRD, FTIR

1. Introduction

Today, the use of clays, including those that are rich in SiO_2 and Al_2O_3 , is experiencing a boom in new construction, ceramics and crafts, pharmaceuticals foundry and pottery. Aluminosilicate bricks are used in the coating of blast furnaces, refining furnaces and kilns in many laboratory ovens. Also called for the development of ceramic materials that are harder than ceramic, earthenware and traditional as well as the crowns of quartz based on alumina. The clay that is the subject of this work is known as "Taza white Clay" consisting essentially of illite and kaolinite, in its natural state. Most clay deposits in this region of northeastern Morocco are heterogeneous and are composed of some smectites mixed with illite and /or kaolinite and other impurities [1]. In the liquid state, mud clay is defined as a water-clay suspension, the origin of the use of sludge is

probably the drilling of oil wells [2]. It allows, due to its rheological properties in order to respond to numerous requests for drilling, such as the stability of the structure (the impregnation of the land and make a cake filter to limit the wall) and spoil disposal [2-3]. In Morocco, earth clays are mainly used for manufacturing traditional and modern construction materials (bricks, tiles, sanitary...) and for pottery. The basic structure of layer silicates and all silicates is the ion SiO_4^{4-} , where the silicon occupies tetrahedral sites. The aluminium ion (Al^{3+}) can substitute for Si^{4+} , but it is generally located in the octahedral sheet.

2. Materials and Methods

2.1. Clay Material

The Liassic aquifer Taza corridor consists of dolomites and limestones Middle Lias Upper Lias. It occupies an area of

1500 km². It is based on an impermeable substratum consisting of Triassic clay-dolerite. It ennoie from south to north below the Miocene marl formations of southern Rif furrow. Its boundaries are formed by the accident southern Rif tight north, Palaeozoic flush with the massive Tazekka and buttonholes in the Middle Atlas Causse. It consists of shales sometimes crossed by quartzite beds and siliceous veins. The physico-chemical analysis shows that this clay is illite.

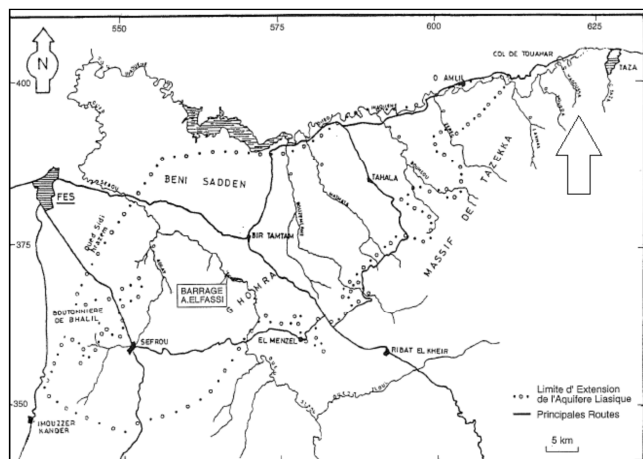


Figure 1. Location map of the city of White Clay of Taza in Morocco.

2.2. Methods

The raw clay and its fine fraction (less than 2 microns in diameter), which is isolated by sedimentation following the experimental procedure [4], were studied using X-ray diffraction (XRD), thermal analysis (DTA and TG), infrared spectroscopy (IR), X-ray fluorescence (XRF), scanning electron microscopy (SEM-EDX). As the mineral composition is variable associated with clay, it seemed necessary to XRD analysis. Spectrometric analysis by SEM, SEM-EDX was performed at the Laboratory of Materials Chemistry IFM, University of Turin (Italy) This is a scanning electron microscope to detect chemical elements, ray analysis X was performed by a diffractometer (45kv, 40mA whose technical characteristics are: Configuration type PW3064, PW3050/60 type goniometer, rotating sample holder (spinner) type PW3064, using either a copper or the anticathode cobalt, the thermograms were carried out by operating a XPERT-PRO under the following conditions: heating rate = 10°C / min, sample weight = 40 mg, atmosphere: air. The Fourier transform infrared (FTIR) samples were obtained on a spectrometer with a DTGS detector and a KBr beam splitter, the technique of pressed KBr disk (1 mg sample and 200 mg of KBr) was used, the spectra were recorded in the region of 4000 - 400 cm⁻¹.

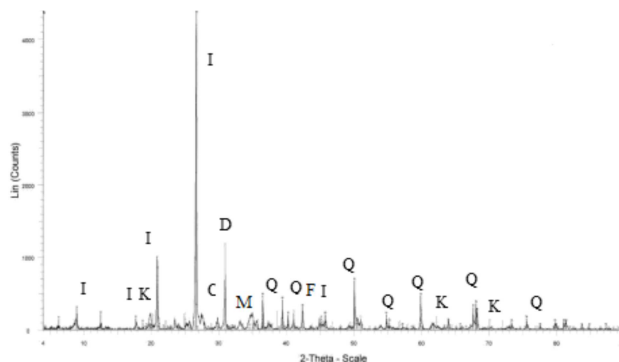
3. Results and Discussion

3.1. X-ray Analysis (XRD)

Treatise contain minerals

The X-ray analysis diffractometric "Figure 2" of the white

clay of Taza shows that there is a majority phase (illite, kaolinite) and minority phases (feldspar, calcite and dolomite) which identified by the cards ASTM (American Society for Testing materials) which are justified by the characteristic peaks for the phyllosilicates, we note the presence of kaolinite ($d = 7.14 \text{ \AA}$) and ($d = 3.79 \text{ \AA}$) and illite ($d = 9.98 \text{ \AA}$) and ($d = 4.48 \text{ \AA}$), we note the presence of: quartz ($d = 4.25 \text{ \AA}$ and 3.34 \AA) as the major impurity; dolomite ($d = 2, 90 \text{ \AA}$), and calcite ($d = 3.03 \text{ \AA}$).



(I): illite, (K): kaolinite, (C): calcite, (D): dolomite, (F): feldspar, (Q): quartz

Figure 2. RX diffractograms of clay white Taza.

3.2. Differential Thermal Analysis and Thermogravimetric Analysis

A substance subject to heat treatment may change its physicochemical properties, such as a phase change, a change in structure, decomposition, a change in volume, etc... [5] the thermal analysis allows observe these changes as a function of temperature. Among the techniques used include differential thermal analysis (DTA), thermogravimetric analysis (TGA).

3.2.1. Differential Thermal Analysis (DTA)

The method involves measuring the temperature difference ΔT between the sample to be studied and a reference sample, inert, both subject to the same warm-up act, used the device can work in a temperature range from 25°C to 1000°C. The heating rate that we have adopted is 10°C / min. The reference sample is alumina. This difference is related to the amount of heat released or absorbed by the material studied. And ΔT is recorded as a function of temperature. This allows the detection of peak endothermic and exothermic changes.

3.2.2. Thermogravimetric Analysis (TGA)

The idea is to continuously monitor the change in mass of a sample as a function of temperature. The sample, placed in an alumina boat suspended from the beam of a balance, is located in a chamber at controlled temperature.

The equilibrium of the balance is provided by an electromagnetic compensation system. The change in mass, given by rebalancing the system, is recorded as a function of the temperature rise.

There are basically three endothermic peaks: the first between 95°C -100°C corresponding to the dehydroxylation

of minerals clay and a second at 530°C and the third at 720°C corresponding to the structural reorganization of the clay minerals.

Differential thermal analysis (TGA-DTA) is very useful, especially for groups of clay, the thermal analysis of clay Taza three steps, as shown in Figure 3, the first endothermic peak at 98°C. We initially attributed to the departure of the water which is about 2 wt% clay [6-7] The endothermic reaction that occurs in the range 110°C-630°C due to the gradual exit of water molecules associated with interlayer cations, the structure of water can be removed without destroying the network of clay. Both endo reactions in sequence in the range 500°C-800°C are due to the departure of OH groups of structure (loss of 8% by weight). This suggests that in the range 630°C-830°C, as for the peak located around 710°C, can be attributed to the amount of iron in octahedral sites [8].

The portion of the curve above 900°C, reflecting the phase changes after the destruction of the structure of the clay is quite variable. it appears for the first quartz (α) or (β) and cristobalite, finally, the mullite.

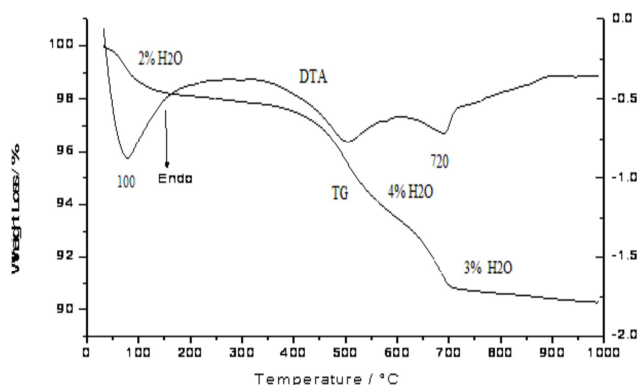


Figure 3. TG and DTA curves of white clay of Taza.

3.3. Spectroscopy Fourier Transform Infrared (FTIR)

Results:

From Figure 4, there is an absorption band at 3646 cm^{-1} which is due to stretching vibration of the OH clays, it is a chemical absorption, another band corresponding air also a stretching vibration ν (H_2O) observed around 3383 cm^{-1} but it is a physical absorption of water between the clay layers, a band corresponding to the bending vibration δ (H_2O) of the physical sorption of water observed around 1643 cm^{-1} , there is also a band corresponding to stretching vibration of Si-O band observed around 1032-1210 cm^{-1} group tetrahedron (SiO_4), the band observed around 3430-1430 cm^{-1} is due calcium carbonate, the bands observed around 520 cm^{-1} and 470 cm^{-1} are due to bending vibration of Al-O-Si and Si-O-Si, respectively (TOT) bands observed around 2526, 1817 and 712 cm^{-1} are due to dolomite and observed to 874, 726 cm^{-1} correspond to calcite, vibration bands observed at 920,880 and 841 cm^{-1} correspond to AlAlOH , and AlFeOH AlMgOH respectively [9-10-11].

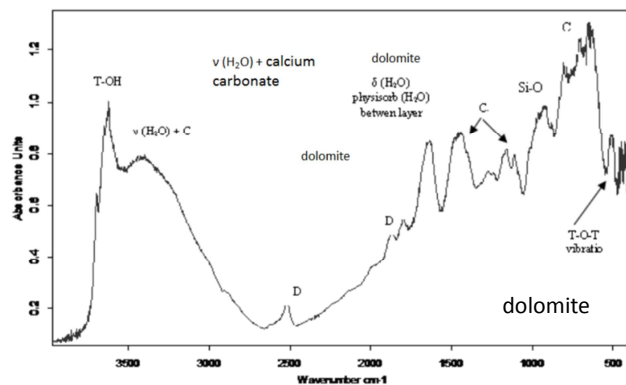


Figure 4. FTIR white clay of Taza.

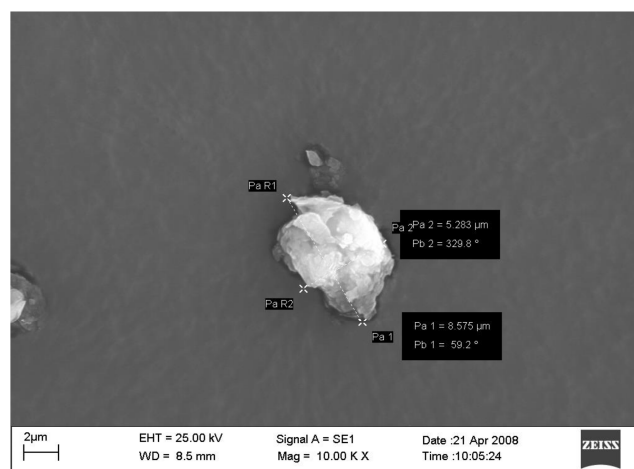
3.4. Elemental Chemical analysis Scanning Electronic Microscope (SEM)

Interpretation:

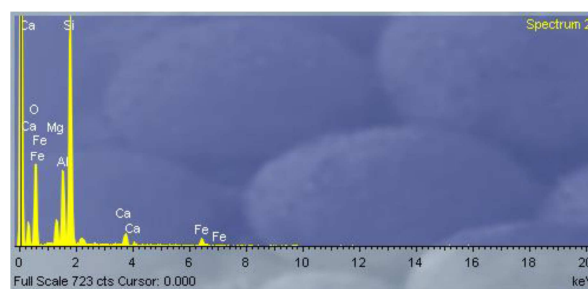
The elemental chemical analysis of the white clay of Taza (Table 1) shows that there is a significant percentage of SiO_2 (53,19%) of Al_2O_3 (22,49%), FeO (1,29%) and K_2O (3,39%) which proves that clay is rich in illite and a small percentage of TiO_2 (0,18) Na_2O (2,05%). The morphology shows an irregularity of the particles forming the aggregate of clay.

Table 1. Characterization by Fluorescence X of white clay of Taza.

compounds	SiO_2	Al_2O_3	FeO	K_2O	CaO	MgO	TiO_2	Na_2O
wt%	53,19	22,49	1,29	3,39	0,92	2,09	0,18	2,05



(a)



(b)

Figure 5. SEM ((a) EDS and (b) morphology) of white clay Taza.

The Figure 5 (a) shows the energy spectra of the element present in the sample corresponding to the chemical analysis of the scanning electron microscopy (SEM), (b) the morphology showed irregularity fine micrometer particle forming the aggregate of the sample.

3.5. Study of the White Clay of Taza

3.5.1. Humidity

The decrease in the weight of the material between the wet and drying at 110°C is determined using the following formula:

$$\text{Humidity \%} = 100 (m_w - m_{110}) / m_w$$

with m_w the mass of material in the wet state (removed) and m_{110} mass of matter at 110°C.

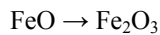
3.5.2. Weight Loss (LOI)

The decrease in the weight of the material from drying at 110°C and the firing temperature to 1000°C is determined using the following formula:

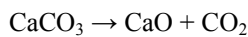
$$\text{Weight loss \%} = 100 (m_{110} - m_{1000}) / m_{110}$$

m_{110} with the mass of the material at 110°C and m_{1000} mass of the sample fired at 1000°C temperature. The weight loss can only know the amount of products that may decompose or evaporate during cooking. At 500°C, the product loses its water content. Between 700 and 900°C, the following reactions may occur [12]:

*Oxidation of FeO:



* Decomposition of carbonates:



3.5.3. Linear Shrinkage During Drying

Determining the shrinkage value is by studying the variation of the average lengths of lines recorded on the briquettes between wet and drying at 110 ° C. The following formula allows the calculation of drying shrinkage:

Linear shrinkage on drying

$$(\%) = (L_w - L_{110} / L_w) \times 100$$

with the length wet L_w , the length drying at 110°C (L_{110}).

3.5.4. Withdrawal of Cooking

Determining the shrinkage value is by studying the variation of the average lengths of lines recorded on the briquettes from the drying at 110°C.

C and firing at 1000°C. The following formula allows calculation of the withdrawal to cook:

Withdrawal of cooking

$$(\%) = (L_{110} - L_{1000} / L_{110}) \times 100,$$

L_{1000} length with cooking, the length L_{110} drying at 110°C.

3.5.5. Water Absorption Capacity (WAC) (%)

This test is the ratio of the difference between the dry weight after absorption and dry weight cooked, the formula allows the calculation of the absorption capacity of water:

$$\% \text{ absorption} = (W_{\text{dry}} - W_{\text{cooked}} / W_{\text{cooked}}) \times 100$$

with W_{dry} abs dry weight after absorption and W_{cooked} dry weight cooked.

3.5.6. The Plasticity Index (PI)

This test is the ratio of water weight and the weight of dry matter, the formula allows the calculation of the plasticity index (PI):

$$\text{Plasticity index \%} = (W_w / W_{\text{dm}}) \times 100$$

with W_w the weight of water and W_{dm} weight dry matter.

3.5.7. Mechanical Resistance to Flexion (RMF)

The determination of the resistance in $\text{kg} \times \text{f/cm}^2$ that can develop the bar against bending under the effect of a load on the bar, is given by the following formula:

$$R (\text{kg} / \text{cm}^2) = P \times 3 \times d / 2 \times w \times e^2$$

R with the mechanical resistance to bending, the force in kg P that causes the breakdown of the bar (60kg), d distance between two supports of the unit (120mm), w the width of the strip (20mm), e the thickness of the bar (10mm).

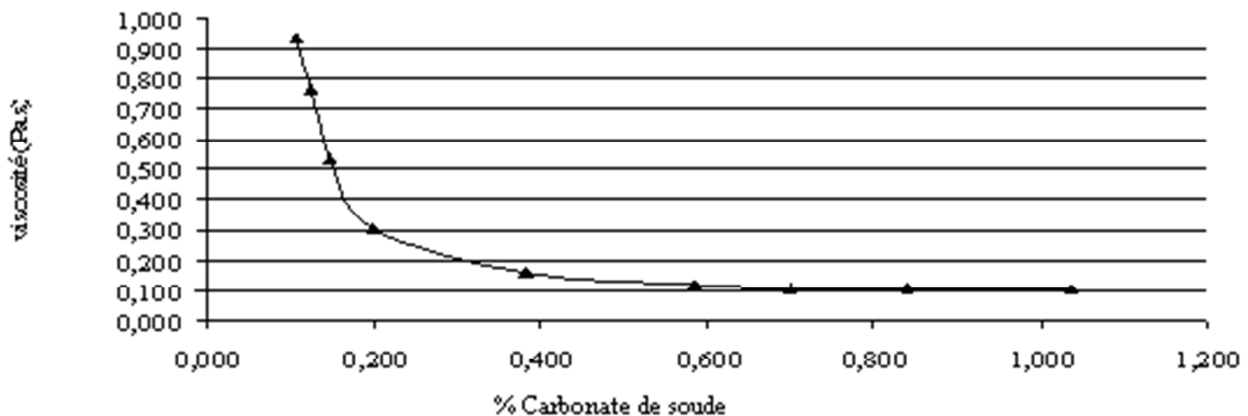


Figure 6. Deflocculation of Taza white Clay with Sodium carbonate (Na_2CO_3).

Interpretation

The white clay of Taza has a medium heat loss, low shrinkage, good flexural strength and good behavior in plasticity (Table 2), In a slurry composed of 320 g clay a white clay of Taza and 300 ml of water was added gradually increasing amounts of deflocculant (sodium carbonate) to each dosage, was allowed to stir for 20 min and then measured the viscosity of the slip. The result of this study is shown in figure 6. In this study, it was found that from 0.59% of deflocculation, the viscosity is minimum and stable. This suggests that, for the white clay of Taza has a flowability optimal deflocculant of 0.59% sodium carbonate (Na_2CO_3).

Table 2. Technological characteristics of white clay Taza.

Characteristic	Result
- Humidity (H) (%)	10,18
- Loss on ignition (PF) (%)	11,05
- Linear shrinkage during drying (RL) (%)	1,32
- Total shrinkage during cooking (RT) (%)	10,42
- Water absorption capacity (WAC) (%)	34,01
- The plasticity index (PI) (%)	20,12
- Mechanical resistance to flexion (RMF) (kg/cm^2)	239

Table 3. Preparation of slurry with different formulations (25, 35 and 45%).

Formula (%)	Materiel				Result (% loss of ignition)	prepared slurry
	¹ white clay Taza	² chamotte	³ feldspar	⁴ quartz		
Formula 1	45,00	35,00	8,00	12,00	7,41	Good
Formula 2	35,00	45,00	8,00	12,00	7,53	Good
Formula 3	25,00	55,00	8,00	12,00	*	Poor

* not determined

1: white clay Taza. 2: commercial chamotte: commercial feldspar: commercial quartz

4. Conclusion

From the results we can say that the white clay of Taza has the same characteristics of clays used in the ceramics industry (medium heat loss, low shrinkage, good flexural strength and good behavior in plasticity). For this white clay of Taza adding 0.57% sodium carbonate (Figure 6) is sufficient to have a good deflocculation and the viscosity is minimum corresponds to the stability of the slip, in his introduction to a formula of slip was successful with a rate of 35 to 45% (formula 1-2, Table 3). The white clay of Taza has an average loss on ignition is due to the elimination of the water content, the decomposition of certain minerals such as carbonates and associated with the combustion of organic matter in association with minerals such as micas, feldspars or carbonates, the temperature of appearance of a liquid phase during sintering is reduced. The levels of iron oxide and titanium influence the color of ceramic shards. As for organic matter, they affect the rheology of suspensions and behavior of matter at the formatting. The X-ray diffraction patterns allowed us to identify the different minerals that make up the white clay of Taza, compared with the available data, we identified illite and kaolinite as clay minerals, other minerals present as impurities major are quartz, calcite, dolomite and magnetite. We can conclude that these results show the important features to justify its use in the ceramic industry.

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