Physicochemical and mineralogical characterization of Moroccan bentonite of Trebia and its use in ceramic technology

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Abstract: This study concerns the results of Physicochemical and mineralogical characterization of a white bentonite of Trebia located in Nador 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 look for the usability of bentonite of Trebia in ceramic technology. The second objective was to develop gels of oxides of high purity from these clays. We can say that the white bentonite of Trebia 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 bentonite of Trebia adding 0,42% 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 10 to 14%. 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 Montmorillonite, feldspath and quartz as bentonite of Trebia, other minerals present as impurities major are magnetit and iron oxide. These results show the important features to justify its use in the ceramic industry.

Keywords: Bentonite, 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. The ceramic materials have a wide range of application include dental prostheses based on silica and alumina.

The bentonite of Trebia that is the subject of this work is known as "Nador white Clay" consisting essentially of Montmorillonite, feldspath and quartz, in its natural state. Most bentonite of Trebia deposits in this region of north Morocco are heterogeneous and are composed of some smectites mixed with kaolinite and other impurities [1]. In the liquid state, mud bentonite of Trebia is defined as a water-bentonite of Trebia 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 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. Bentonite of Trebia Material

It is located on the western flank of Jebel Tidiennit. Its

deposit is stratiform type affected by brittle tectonics. It appears as white to yellowish-white bentonites. reserves are estimated at 1,44 million tonnes.

Mineralogical analysis shows that bentonite Trebia is mainly composed of inorganic type beidelite - montmorillonite (smectite) representing (63 wt%), feldspar (30%) , magnetite represents (5 wt%) and oxide of iron in the form of scales (2 wt%), the cation exchange capacity is 89 meq/100 g and a specific surface area of 37 m²/g, the chemical analysis shows a high percentage of silica, alumina and low Na_2O K_2O (Table1).
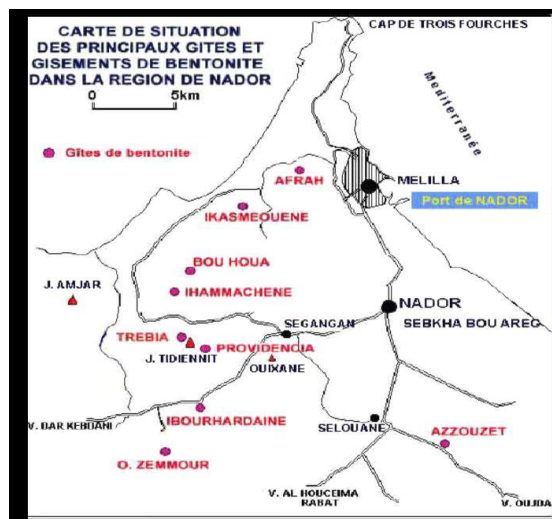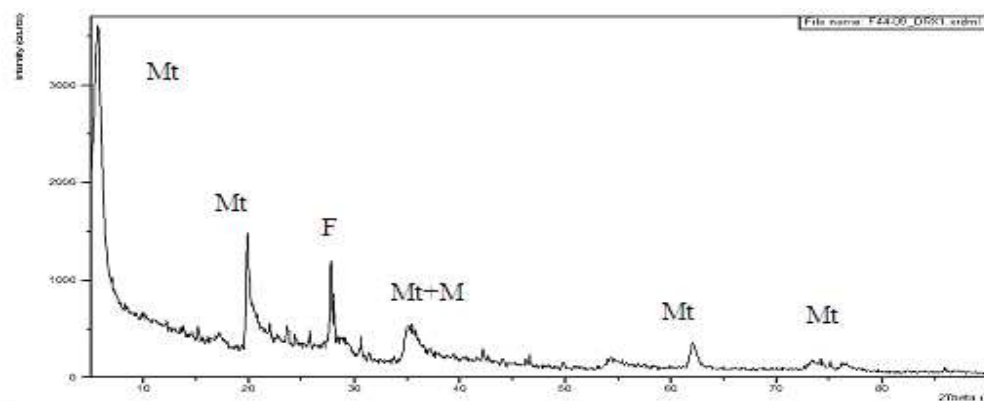

Figure 1. Location map of the city of White Bentonite of Trebia in Morocco.

2.2. Methods

The raw bentonite of Trebia 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 complex, it seemed necessary to characterize the clay by 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, XRD analysis was performed by a diffractometer (45kv, 40mA whose technical characteristics are: the thermograms were carried out by operating a XPERT-PRO inder the following conditions: heating rate = 10 ° C / min, sample weight = 40 mg, atmosphere: air.

The Fourier transform infrared spectrometry (FTIR) 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)

3.1.1. Treatise Contain Minerals

The X-ray analysis diffractometric "Figure 2" of the white bentonite of Trebia shows that there is a majority phase (Montmorillonite, feldspar) and minority phases (magnetit, iron oxide) which identified by the database and the cards ASTM (American Society for Testing materials) which are justified by the characteristic peaks for the phyllosilicates.

(Mt): montmorillonite, (F): feldspath, (M): magnetit.

Figure 2. RX diffractograms of bentonite of Trebia.

3.2. Differential Thermal Analysis and Thermo Gravimetric Analysis

A substance subject to heat treatment may change its physicochemical properties, such as a phase change, structural reorganization, decomposition, etc.[5]. 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. Thermo Gravimetric 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 ° - 100 ° C corresponding to the dehydroxylation of minerals bentonite of Trebia and a second at 530 ° C and the third at 720 ° C corresponding to the structural reorganization of the bentonite of Trebiaminerals.

differential thermal analysis (TGA-DTA) is very useful, especially for groups of clay, the thermal analysis of bentonite of Trebia 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% bentonite of Trebia[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 956 ° C, reflecting the phase changes after the destruction of the structure of the bentonite of Trebia is quite variable. it appears for the first quartz (α) or (β) and cristobalite, finally, the mullite.
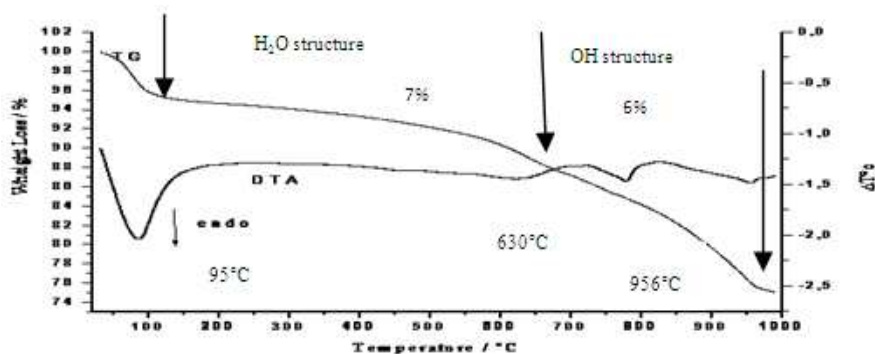

Figure 3. TG and DTA curves of white bentonite of Trebia.

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

From Figure 4, there is an absorption band at 3646 cm[-1] corresponds to stretching vibration of the OH group chemically adsorbed on clays. Another band around 3383 cm[-1] corresponds to stretching vibration ν (H₂O). This is mostly from physical absorption of water between the bentonite of Trebia layers. a band corresponding to the bending vibration δ (H₂O) 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 (SiO₄). 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. Vibration bands observed at 920,880 and 841 cm[-1] correspond to AlAlOH, and AlFeOH AlMgOH respectively [9-10-11].
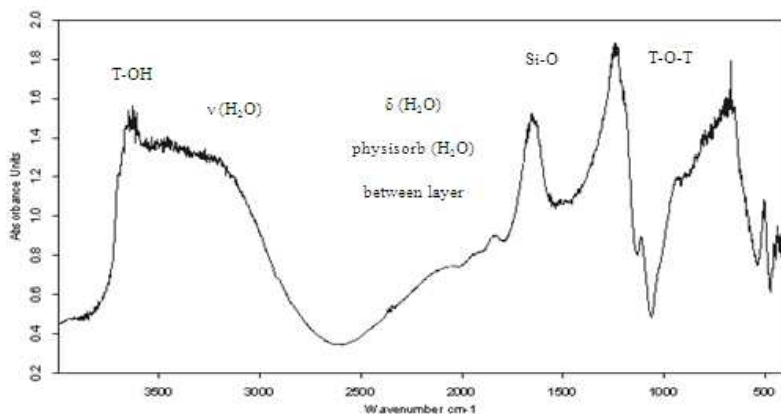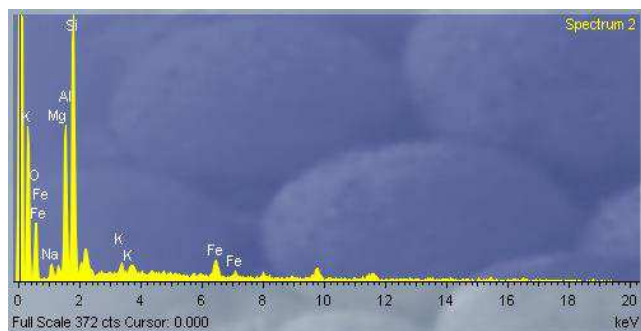

Figure 4. FTIR white bentonite of Trebia.

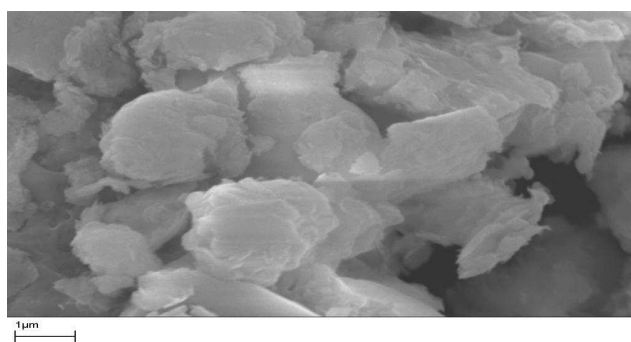### 3.4. Elemental chemical analysis Scanning Electronic Microscope (SEM) Interpretation

The elemental chemical analysis of the white bentonite of Trebia (Table 1) shows that there is a significant percentage of SiO_2 (58,55%) of Al_2O_3 (26,89%), Na_2O (2,05%) and K_2O (0,42%) and a small percentage of TiO_2 (0,15). The morphology shows an irregularity of the particles forming the aggregate of clay.

Table 1. *Characterization by Fluorescence X of white bentonite of Trebia.*

compounds	SiO_2	Al_2O_3	K_2O	CaO	MgO	TiO_2	Na_2O
wt%	58,55	26,89	0,42	0,82	2,01	0,15	2,01

(a)

(b)

Figure 5. *SEM ((a) EDS and (b) morphology) of white bentonite of Trebia*

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.

3.5.2. 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:
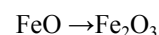
$$\text{Weight loss}\% = 100 \ (m_{110} - m_{1000}) \ / \ m_{110}.$$

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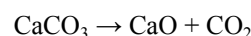
The weight loss can only know the amount of products that may decompose or evaporate during cooking. At 500°C, the product loses its water content. Between 700 and 900° C, the following reactions may occur [12]:

*Oxidation of FeO:

$$FeO \rightarrow Fe_2O_3$$

* Decomposition of carbonates:

$$CaCO_3 \rightarrow CaO + CO_2$$

3.5. Study of the White Bentonite of Trebia of Nador

3.5.1. Humidity

The evolution in the weight of the material between the initial state and drying at 110 °C is determined using the following formula:

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

with m_i the mass of material in the initial state and m_{110} mass of matter at 110 ° C.

3.5.2. 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

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

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

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

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

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

3.5.4. 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:

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

with W_{dry} abs dry weight after absorption and W_{cocked} dry

weight cooked.

3.5.5. 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):

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

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

3.5.6. Mechanical Resistance to Flexion (RMF)

The determination of the resistance in kg × f/cm² that can develop the bar against bending under the effect of a load on the bar, is given by the following formula:

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

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).

Interpretation

The white bentonite of Trebia has a medium heat loss, low shrinkage, good flexural strength and good behavior in plasticity (Table 2). In a slurry composed of 320 g bentonite of Trebia 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 0,42% of defloculation, the viscosity is minimum and stable. This suggests that, for the white bentonite of Trebia has a flowability optimal deflocculant of 0,42% sodium carbonate (Na_2CO_3).

Table 2. Technological characteristics of white bentonite of Trebia.

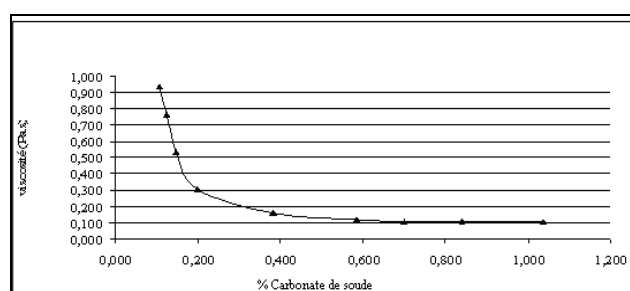| Characteristic	Result
- Humidity (H) (%)	10,19
- Loss on ignition (PF) (%)	11,08
- Linear shrinkage during drying (RL) (%)	2,15
- Total shrinkage during cooking (RT) (%)	11,07
- Water absorption capacity (WAC) (%)	34,01
- The plasticity index (PI) (%)	23
- Mechanical resistance to flexion (RMF) (kg/cm²)	99

Figure 6. Deflocculation of white Bentonite of Trebia with Sodium carbonate (Na_2CO_3).

Table 3. Preparation of slurry with different formulations (08, 10 and 14%).

Formula (%)	Materiel				Result (% loss of ignition)	prepared slurry
	[1] white bentonite	[2] chamotte	[3] feldspar	[4] quartz		
Formula 1	14,00	66,00	8,00	12,00	7,75	Good
Formula 2	10,00	70,00	8,00	12,00	7,69	Good
Formula 3	08,00	72,00	8,00	12,00	*	Poor

* not determined

1 : white bentonite Trebia. 2 : commercial chamotte 3 : commercial feldspar 4 : commercial quartz

4. Conclusion

From the results we can say that the white bentonite of Trebia 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 bentonite of Trebia adding 0,42% 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 10 to 14% (formula 1-2, Table 3). The white bentonite of Trebia 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 bentonite of Trebia, compared with the available data, we identified montmorillonite and feldspar as bentonite of Trebia minerals, other minerals present as impurities major are magnetit and iron oxide.

We can conclude that these results show the important features to justify its use in the ceramic industry.

Acknowledgements

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