

# Mâadid Clay In The M'sila Region, Algeria: Evaluation And Characterization

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## Abstract

This work aims to valorize local materials, such as the clay of southern Algeria, widely used to prepare bricks as rural construction materials. We characterized and identified three types of natural clays from the region of Mâadid province of M'sila. The results show that the yellow clay is a mixture of quartz illite in a very important proportion since the percentage of illite was 47%. While in the green clay, we noted that the predominant constituents are: calcite, quartz, illite, and kaolinite, and the highest percentage is illite was 27.93%. The analytical results show that the red clay is a calcite, quartz, and illite mixture. Several techniques were used: XRD, XRF, FTIR.

**Keywords:** red clay, rural construction, swelling coefficient, colloidal, plasticity

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## I. Introduction

The world faces great challenges due to an increasing quantity of emitted gases, causing global warming. There are many sources of greenhouse gas emissions. The most important is the construction industry, which consumes a large amount of energy and emits significant greenhouse gases [1-4]. With population growth and the need for building materials, interest in suitable materials such as clay has piqued the research community's interest. Clay, for example, has been used in construction as an alternative to cement and rubble, to reduce these emissions that contribute to environmental pollution [5-8], clay-based building materials can be used, especially in rural construction, because of its advantages, including low environmental impact, thermal insulation, limited shrinkage, high strength and low-cost [9-14]. Some studies have used the characterization of different types of clay used as compressed earth blocks and reinforced with plant fibers to prepare composite materials for rural construction [15]. Koadri et al. [15] studied the effect of alkali treatment on the mechanical

behavior of a red clay-composite material reinforced with palm fibers. The authors found from the spectral analysis of the diffractogram that the red clay is heterogeneous, with high proportions of quartz compound ( $\text{SiO}_2$ ) calcite  $\text{CaCO}_3$ . Mouissa et al. [16] examined the effect of chemical treatment of sawdust on the mechanical and thermal properties of a clay-based composite from the Bou-Saâda region, intended for use as a rural construction material. The chemical analysis of clay revealed that silica and alumina are the main oxides of the sample studied. Their percentages are respectively 35.21% and 13.62%. Thus, the studied clay is an aluminosilicate rich in calcite ( $\text{CaCO}_3$ ).

Phung et al. [17] investigated the mechanical strength and drying capacity of mud made from three different soils currently used for conventional earth construction. Two earth-fiber formulations were developed using straw as fiber. The results show that the compressive strength increases as the moisture content decreases. This research aims to develop environmentally friendly, thermally insulating, and low-cost materials used in rural construction. Clay is a very abundant material in Algeria (Figure1).



**Fig.1** Mountain with Mâadid clay.

Source: Authors (January 2022)

## II. Materials and methods

For this work, we collected a sample of red clay from the region of Al-Maâdid, located 35 km north of M'sila, and two samples of yellow clay and green clay from the region of Ouled Tebbene, wilaya of Setif, northeast Algeria. The samples collected underwent the following unit operations:

- the rocks of the sample were crushed in pieces using a mortar;
- the clay sample pieces were ground in a mill;
- the sample powder was sieved (50 $\mu\text{m}$  sieve);
- the samples were dried in the oven for 24 h ( $T=105\text{ }^\circ\text{C}$ ).

### Determination of pH

A 10% solution of each clay m/v was prepared with distilled water. The mixture was left to rest for 4h at 25° C to allow the ions to pass into the solution. The clay solution obtained was homogenized by a magnetic stirrer. The reading was done directly on a HANNA pH meter (pH 211 made in Italy) [18-21].

### Moisture content

Moisture content measurement determines the moisture mass removed by drying a wet material to a constant mass at a temperature of  $105 \pm 5$  °C for 24 h. The mass of the material after steaming is considered to be the mass of the solid particles ( $m_s$ ). The determination of the moisture content is calculated from the ratio of the mass of moisture ( $m_{\text{moisture}}$ ) to the mass of solid particles ( $m_s$ ). This gives the moisture content of the sample analyzed as proposed by Chossat(2005)[22]:

$$H(\%) = \frac{m_{\text{moisture}}}{m_s} \times 100 = \frac{m_t - m_s}{m_s} \times 100 \quad (1)$$

where,  $m_{\text{moisture}}$  is the moisture mass (g),  $m_s$  is the dry sample mass (g), and  $m_t$  is the wet sample mass (g).

### Density

The bulk density measurement of the studied sample was performed by determining the volume of a mass  $m$  of the sample using a pycnometer as follows:

$$\text{Density} = \frac{\text{Mass of the sample}}{\text{Volume of the sample}} \quad (2)$$

### Swelling index

By being suspended, the clay can fix a certain amount of water, which has the effect of moving away from each other, thus reflecting a swelling. A 100 ml graduated cylinder is filled with 50 ml of distilled water, and 0.5 g of clay is added. After 45 min, another 0.5 g of clay is added. After 2H, the swelling volume is noted. The swelling index is measured by the following formula (3) [23] :

$$\text{Swelling index (\%)} = \frac{\text{Swelling volume} \times 50}{50 - \text{humidity}} \times 100 \quad (3)$$

### Colloidalilty

This property is linked to the presence of negative charges on the surface of each clay grain. The covering of each clay grain translates the colloidal character by a double layer of water-soluble ions of opposite charges. Colloidalilty is measured by suspending a given quantity of clay adding 0.2g of MgO to allow deflocculation. After a 5 min agitation, the mixture is placed in a graduated test tube. After 24h, the volume  $V$  (ml) occupied by the supernatant is measured. The colloidalilty is measured with the following formula [24]:

$$C (\%) = 100 - V \quad (4)$$

X-ray diffraction analysis was used with an X'Pert High Score X-ray diffractometer (X'Pert Pro PW3209, Panalytical, made in France). Equatorial diffraction patterns ( $2\theta$ ) were recorded from  $10^\circ$  to  $40^\circ$  with Cu-K $\alpha$  radiation at 40 kV and 20 mA. The infrared spectra were studied using an FTIR apparatus (Shimadzu, FTIR-8300, made in Japan). The adsorption range was  $500 \text{ cm}^{-1}$  to  $4000 \text{ cm}^{-1}$ . The pellets were made by mixing 0.08 mg of clay with 0.01 mg of KBr. Furthermore, the mixture was subjected to a pressure of 80 Pa. The mineralogical test used X-ray fluorescence spectrometry. XRF Bruker AXS GmbH, Karlsruhe, Germany was used to analyze the mineralogical composition of clay. The elemental chemical analysis of the clay sample was performed using an XRF Spectrometer. This chemical analysis was carried out in the Lafarge Cimenterie M'sila plant laboratory.

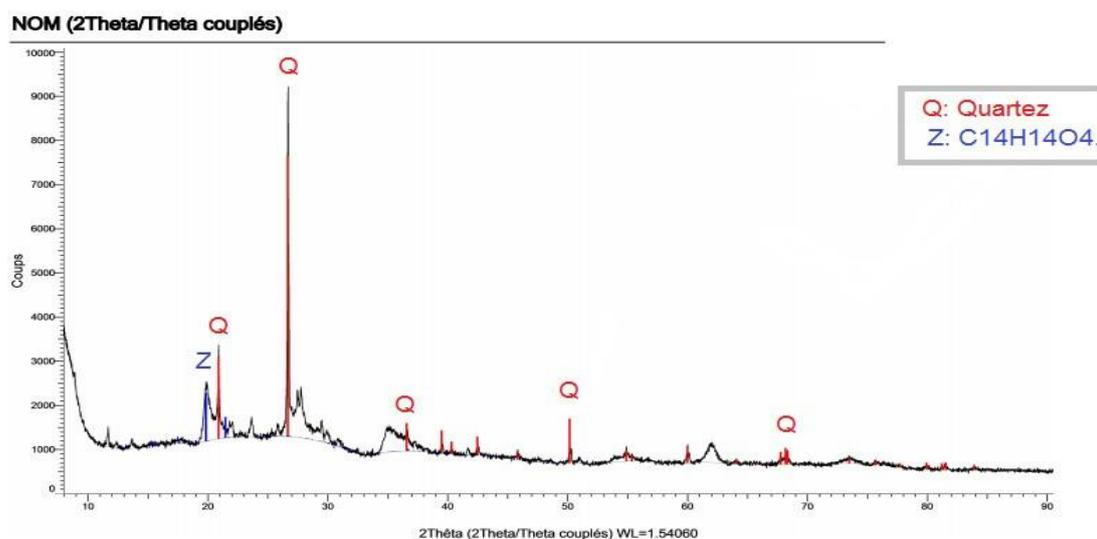
## III. Results and discussion

### Mineralogical X-ray analysis

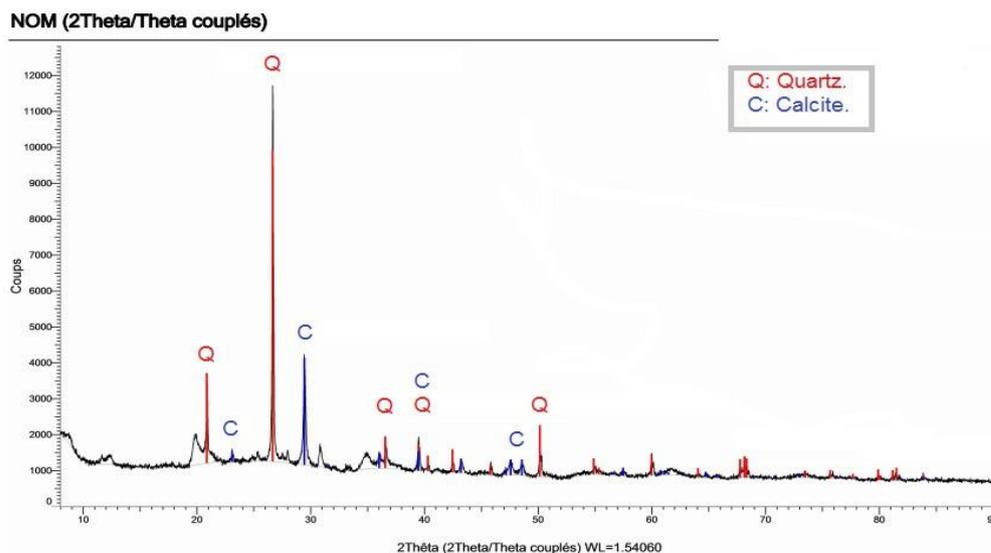
Table 1 summarizes the results of elemental chemical analysis obtained for the three clay samples. The diffractograms were obtained from the disoriented samples (placed directly in powder form in a conventional sample holder). Figures 2, 3, and 4 show the XRD pattern of three types of clays, respectively. Quartz at  $2\theta = 22^\circ$  and  $2\theta = 27^\circ$  was observed as a major impurity in the yellow and green clay samples (Figures 2 and 3). Calcite at  $2\theta = 29^\circ$  was present in the green and red clay samples. It was noted in a very small amount for the yellow clay sample. Reflections at  $2\theta = 31^\circ$  and  $2\theta = 42^\circ$  on the red clay prove the presence of dolomite (Figure 4). It can be observed that a peak at  $20^\circ$  appears on all three samples with different intensities. It can be attributed, presumably, to illite. Peaks at  $2\theta = 12^\circ$  and  $2\theta = 26^\circ$  on all three clay types indicate the presence of kaolinite. The clay fraction of the three types of clays include quartz, calcite, and dolomite. This result confirms that our clay samples are heterogeneous mixtures.

**Table. 1** Chemical analysis of clays.

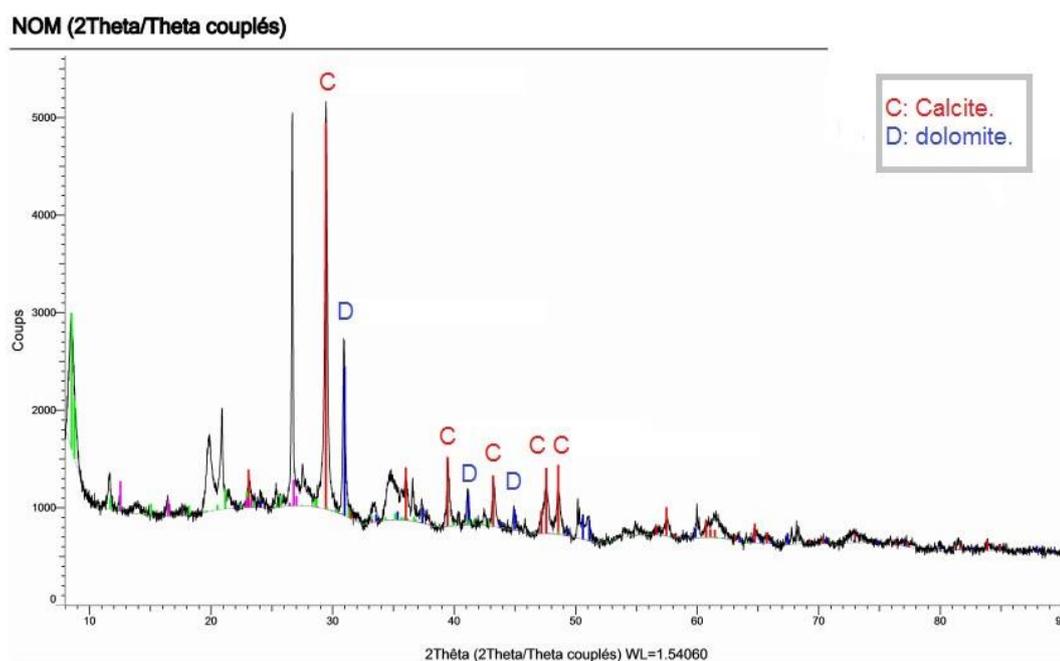
Clay	Calcite (CaCO <sub>3</sub> )	Dolomite	Quartz (SiO <sub>2</sub> )	Pyrite- (FeS <sub>2</sub> )	Illite	Chlorite	Kaolinite	Albite	CO <sub>2</sub> XRD
Yellow	9.82	0	18.62	0	43.45	0	9.04	19.07	4.32
Green	18.93	3.86	27.04	0.37	27.93	3.1	14.28	4.47	10.17
Red	31.06	10.3	11.94	0.43	31.44	3.17	6.19	5.46	18.57



**Figure 2.** Histogram of the mineralogical DRX analyses of the yellow clay.



**Figure 3:** Histogram of the mineralogical DRX analyses of the green clay.



**Figure 4:** Histogram of the mineralogical DRX analyses of the red clay.

#### Chemical composition analysis by X-ray fluorescence

Clay's mineralogical and physico-chemical properties are particularly interesting in many applications such as water treatment, paint, barrier for pollutants, adsorbent, catalyst, etc. [25-48]. Table 2 lists the results of elemental chemical analysis obtained for the red and green claysamples. We can note that silica and aluminumconstitute the clay structure. The percentage of these compositions is very important. This resultindicates the presence of Kaolinite ( $Al_2Si_2O_5(OH)_4$ ).

Clay materials contain a relatively abundant amount of  $SiO_2$ . The ratio alumina/silica gives an idea of the permeability to water and moisture. The higher the ratio, the greater the permeability [49]. The results indicate

that the SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio equals 2.65 for the yellow clay, 3.28 for the green clay, and 3.72 for the red clay, indicating significant free silica content. The overall composition of other oxides (Fe<sub>2</sub>O<sub>3</sub>, MgO, K<sub>2</sub>O, and Na<sub>2</sub>O) reaches a percentage of 13.13%, 11.73%, and 20.85 % in the three types of clay (yellow, green, and red), which shows that our clay is not pure [50].

The yellow and red clay samples present a rather high K<sub>2</sub>O content (3.78% and 2.86%) compared to the green clay sample indicating that the yellow and the red clays are richer in illite, which confirms the results obtained by DRX. The high CaO content in the red clay indicates a high amount of calcium carbonate in this type of clay. The red clay sample contains a notable amount of calcium oxide CaO (31.71%). This result is quite consistent with the presence of a calcite phase detected by XRD.

**Table 2.** Chemical composition of the clay samples (yellow, green, and red).

Clay	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O
Yellow	36.66	13.82	5.44	15.63	3.71	0.85	3.78	0.20
Green	48.86	14.89	6.34	8.43	2.66	1.02	1.98	0.75
Red	38.41	10.31	10.53	31.71	7.41	3.21	2.86	0.05

### Infrared(IR) analysis

Infrared spectroscopy was used to complete the analysis of the clay sample. Figures 5, 6, and 7 illustrate the spectra obtained. Figure 5 presents the spectrum of the yellow clay. It can be observed that the peaks which spread between 3200-3800 cm<sup>-1</sup>, located at 3620.1 cm<sup>-1</sup> and 3417.6 cm<sup>-1</sup> correspond to the elongation vibrations of the internal OH groups. The peak ranges between 1600-1700 cm<sup>-1</sup> can be attributed to the valence vibrations of the OH group of the water of constitution and the bending vibrations of the adsorbed water's bending vibrations at 1649.0 cm<sup>-1</sup>. The peaks located between 1400 and 1500 cm<sup>-1</sup> are attributed to the deformation vibrations of the CH<sub>3</sub> groups located at 1442.6 cm<sup>-1</sup> [51,52]. The 877.6 cm<sup>-1</sup> peak corresponds to the presence of calcium carbonate CaCO<sub>3</sub> [53].

Figure 6 presents the spectrum of the green clay: The peaks that range between 3397.3 cm<sup>-1</sup>, 3421.5 cm<sup>-1</sup> and 3620.1 cm<sup>-1</sup> correspond to the elongation vibrations of the internal OH groups. The peak between 1770-1800 cm<sup>-1</sup> located at 1797.5 cm<sup>-1</sup> can be attributed to the elongation vibrations of the C=O [54]. The 871.8 cm<sup>-1</sup> peak corresponds to the presence of calcium carbonate CaCO<sub>3</sub>. The absorption peak at 779.2 cm<sup>-1</sup> is in agreement with the XRD, indicating the presence of quartz in the clay. Figure 7 shows the spectrum of the red clay: The peaks that range between 3633.6 cm<sup>-1</sup> and 3442.7 cm<sup>-1</sup> correspond to the elongation vibrations of the internal OH groups. The peak that ranges between 1600-1700 cm<sup>-1</sup> can be attributed to the valence vibrations of the OH group of the constituent water, in addition to the bending vibrations of the adsorbed water located at 1645.2 cm<sup>-1</sup>. The peaks located between 900-1200 cm<sup>-1</sup> correspond to the valence vibrations of the Si-O bond [55]. The 844.8 cm<sup>-1</sup> peak corresponds to the presence of calcium carbonate CaCO<sub>3</sub> [54].

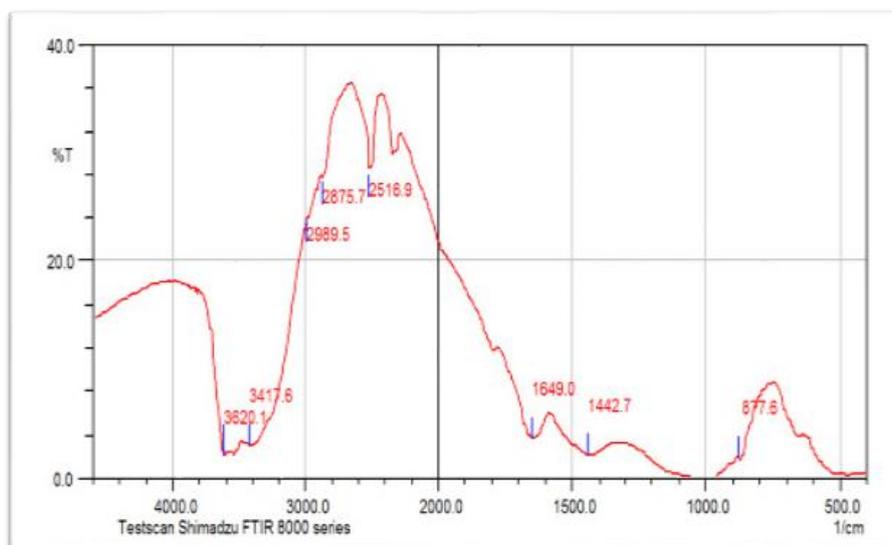


Figure 5. Infrared spectrum of yellow clay sample.

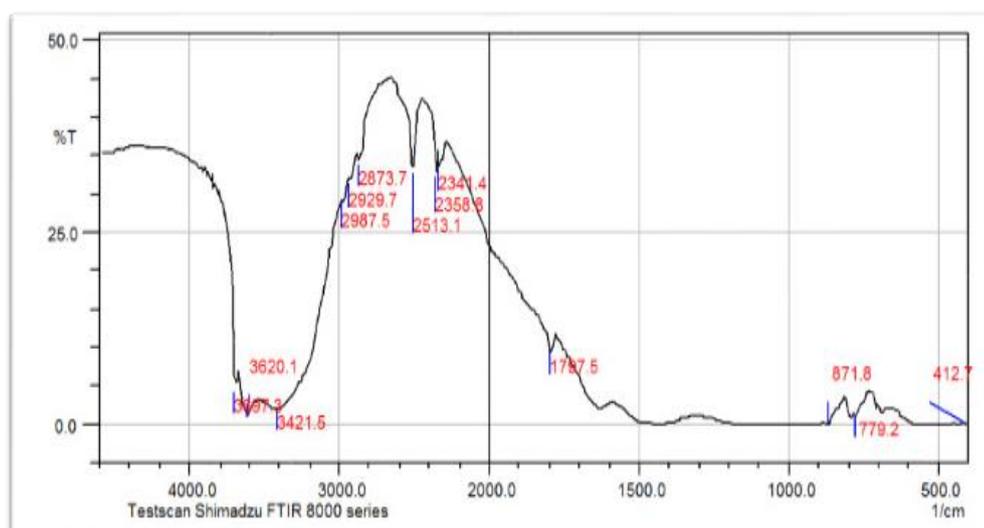
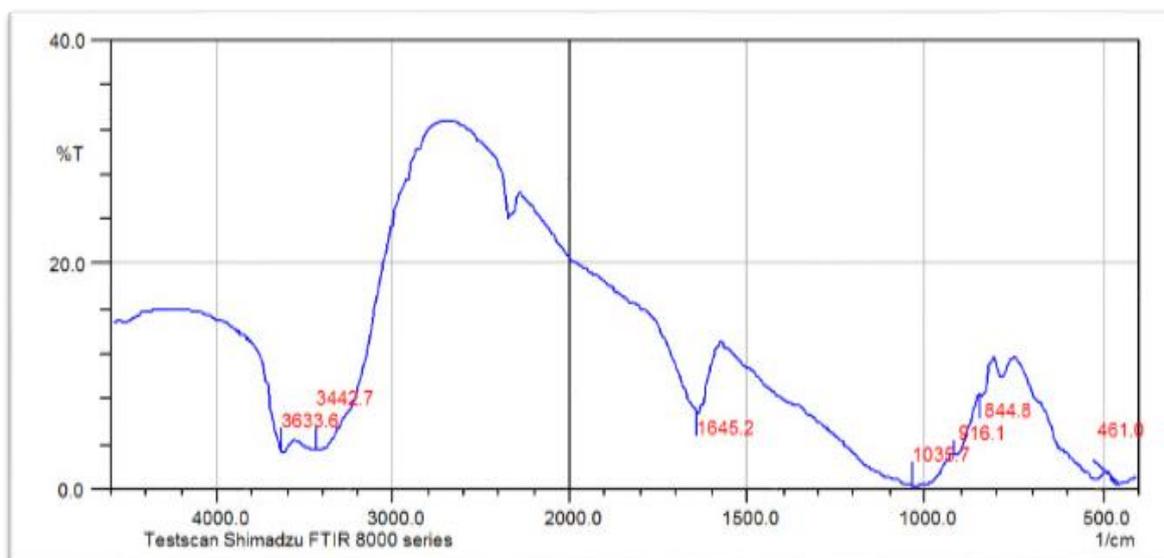


Figure 6. Infrared spectrum of green clay sample.



**Figure 7.** Infrared spectrum of red clay sample.

**Physico-chemical characterization of clay samples**

Table 3 gathers the values of the various parameters relating to our clay samples. We notice that the pH values obtained from the three clays (yellow, green, and red) are 9.18, 8.19, and 7.94, respectively. These results indicate the basicity of the studied samples, which would be due to the soluble salts and basic character as the alkaline carbonates and bicarbonates or the silicates, which generally return in the composition of the clay [56]. We note that the moisture content value is low for both yellow and green clay samples compared to the red clay sample. This explains the non-hygroscopic character and confirms the low value of porosity, which indicates that both yellow and green clay samples can be considered very low porous.

The value of colloidalilty for the three samples (yellow, green, and red) is low (1.9, 8.65, 9.44), which explains the low ionization of particles entering the constitution of the samples studied [57]. The loss on ignition corresponds to the loss of mass of a powder brought to the oven at 1000°C. In our case, we find a high value of about 17.52 % for the yellow clay, 19.96 % for the green clay, and 22.13 % for the red clay sample. The latter may be related to the presence of carbonate and silicate minerals [59]. The swelling coefficient of the studied samples of yellow clay, green clay, and red clay is 58.57 %, 54.08 %, and 55.39 %. According to the classification proposed by Holtz et al. [59], the studied samples can be considered as having a medium swelling coefficient.

**Table 3.** Physicochemical parameters of the clay sample.

Parameter	Yellow	Green	Red
pH	9.18	8.19	7.94
Moisture content H (%)	7.23	3.77	3.96
D	2.25	2.38	2.63
Swelling index I <sub>g</sub> (%)	58.57	54.08	55.39
Colloidalilty C (%)	1.9	8.65	9.44
Loss on fire PAF (%)	17.52	16.96	22.13

**The Atterberg limits**

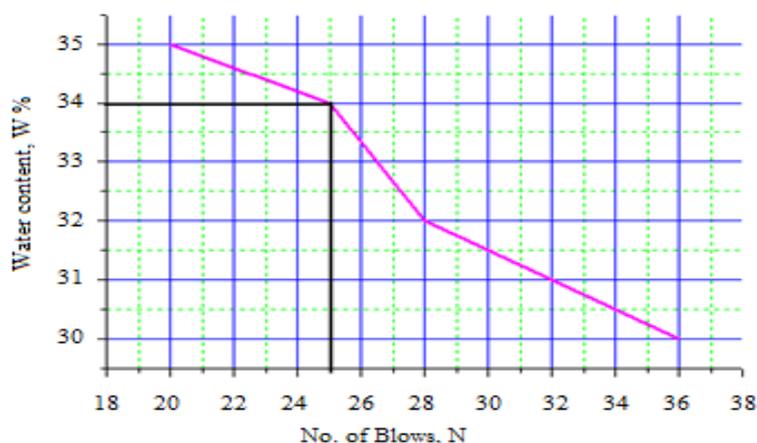
Using Atterberg limits on our clay samples, it can be noted that the plasticity  $W_p$  values of the clay samples (yellow, green and red, respectively) reached 29.33 %, 38 %, and 34.33 % (Table 4). Figures 8, 9, and 10 indicate the values of clay liquidity  $W$  in the three samples: 34%, 50%, and 38.3 %. The plasticity index values  $I_p$  of the clay samples is 4.67 %, 12 %, and 3.97 %, respectively. According to the Atterberg limits classification presented in Table 5, it can be concluded that the two clays (yellow and red) have low plasticity and that the green clay has medium plasticity [59]. Bell [60] reported that the plasticity increases in the presence of quartz, kaolinite, and decreases for montmorillonite. Dash and Hussain [61] reported the same result. The authors explained that plasticity would be increased in silica-rich soils ( $Si^{2+}$  is the main ion in quartz and kaolinite). In the case of green clay, a significant percentage of quartz (27.04 %) is obtained with the help of the DRX analysis. Thus, the plasticity index has a maximum value (12 %).

**Table 4.** Values of the Atterberg limits.

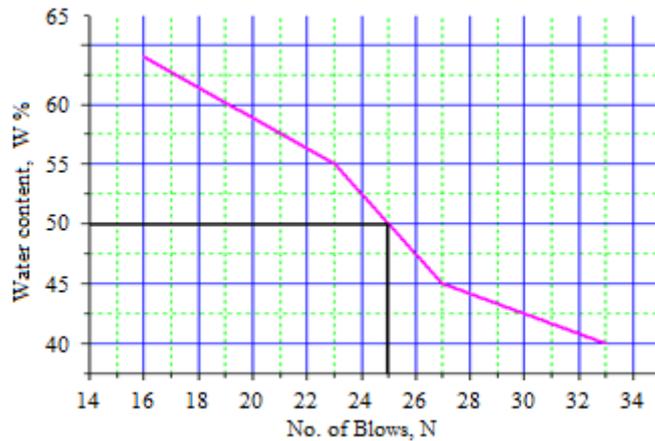
Clay	Liquidity limit $W_L$ (%)	Plasticity limit $W_p$ (%)	Plasticity Index $I_p$ (%)
Yellow	34	29.33	4.67
Green	50	38	12
Red	38.3	34.33	3.97

**Table 5.** Classification of clay based on the Atterberg limits [24].

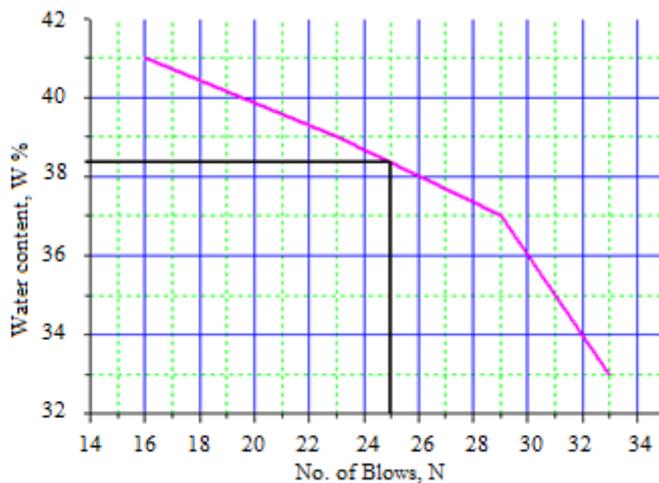
Plasticity index $I_p$ (%)	< 7	from 7 to 17	> 7
Plasticity	Low	Average	High



**Figure 8.** Diagram for the determination of the liquidity limit of yellow clay.



**Figure 9.** Diagram for the determination of the liquidity limit of green clay



**Figure 10.** Diagram for the determination of the liquidity limit of red clay.

#### IV. Conclusion

This paper highlighted the importance of clay, a very abundant material in Algeria, for rural construction when used properly. The experimental techniques allowed us to highlight the phyllite and mineral phases and the chemical composition of the clays analyzed. The results indicate that the three types of clay of Mâadid of the region of M'sila constitute kaolinite, illite, and calcite as associated clay minerals. These findings also highlighted the richness of these three types of clay (yellow clay, green clay, and red clay) in quartz, translating into a high proportion of silica and low porosity. According to the results obtained, it can be said that the red clay sample is the most profitable and close to being used in the manufacture of cement with processing recommendations such as using limestone from a quarry with low  $SO_3$  as a mixture to ensure the conformity of cement quality. Therefore, it is important to consider the results obtained before applying this type of clay to prepare composite materials for rural construction.

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