

# Mineralogical and Chemical Characterization of Palygorskite from East-Algeria

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## INTRODUCTION

Palygorskite is a fibrous hydrated 2:1 aluminum - magnesium phyllosilicate with needle shaped crystals. Its structure consists of talc like ribbons parallel to the fiber axis (Bradley, 1940; Serratos, 1978; Barrios et al., 1995). Like other clay minerals, palygorskite also has tetrahedral (silica) and octahedral (Mg-Al) sheets as its basic building blocks. The tetrahedral sheet inverts its apical directions in adjacent ribbons, each ribbon alternating with channels (dimension  $6.4 \times 3.7 \text{ \AA}$ ) along the fiber axis (Serratos, 1978). Octahedral sheets are discontinuous at each inversion of the tetrahedra. It has the ideal formula  $[\text{Si}_8 (\text{Mg,Al,Fe})_5 \text{O}_{20} (\text{OH})_2 (\text{OH}_2)_4] \cdot 4\text{H}_2\text{O}$  (Frost et al., 2003; Zandonadi et al., 1986), where  $\text{H}_2\text{O}$ ,  $(\text{OH}_2)$  and  $(\text{OH})$  represent zeolitic, coordinated and structural water, respectively (Fig. 1). The octahedral sites are principally occupied by Mg(II) cations, with some replacement principally by Al(III) or Fe(III) cations.

The main objective of this study is to investigate the palygorskite clay from Algeria, in order to determine the clay type mineralogy, chemical composition and morphological characteristics.

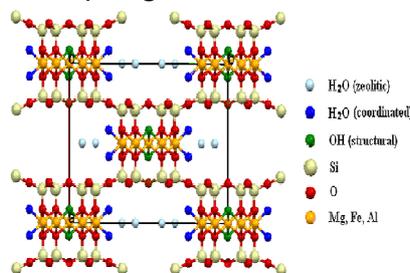


fig. 1. Projection on (0 0 1) of crystal structure of palygorskite.

## MATERIALS AND METHODS

Two palygorskite-rich sediments were

collected from East-Algeria: D-Atta@dz and C-Atta@dz. Different steps for the preparation of the sample were performed in a laboratory, including (i) crushing (ii) grinding and (iii) sieving to obtain the micrometers grain sizes for future use (Dali Youcef, 2012). In Figure 2 different forms for Algerian palygorskite are shown.

The mineralogical composition was determined by X-Ray Powder Diffraction (XRPD) using a Panalytical X-Pert Pro diffractometer with  $\text{Cu K}\alpha$  radiation (45 kV, 40 mA), Ni filter, RTMS X Celerator detector,  $4^\circ$ - $69.9928^\circ$  scan range,  $0.0084^\circ$  step size, 10.150s counting time, for a total of 7898 points and 11 min/sample.

IR spectra were obtained using a Spectrophotometer model Alpha-Bruker. The palygorskite samples were mixed with KBr (1 mg per 150 mg sample).

Chemical composition was determined using a commercial wavelength dispersive X-ray fluorescence instrument (BRUKER S4 Pioneer) equipped with an Rh anode X-ray tube (60 kV, 150 mA), three analyzer crystals (OVO-55, LiF 200 and PET) and a flow proportional counter for light element detection and a scintillation counter for heavy elements. Quantification was made by the fundamental parameters method using the software linked to the equipment (SpectraPlus).

The texture of the samples was studied using a Zeiss® DSM 950 scanning electron microscope equipped with an EDX Link Analytical Pentafet detector. TEM observations were performed in a Philips® CM20 STEM equipment by depositing a drop of diluted dispersion onto a microscope grid. These two electron microscopes belong to the Scientific Instrument Centre of the University of Granada.

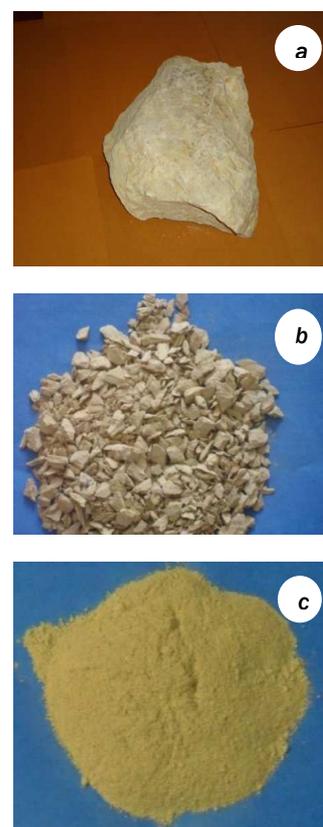


fig. 2. Different forms of Algerian palygorskite (a: rock, b: fragment, c: powder)

## RESULTS AND DISCUSSION

### X-ray Diffraction

The two palygorskite powders were characterized and the results are displayed in Figure 3. For the two samples, D-Atta@dz and C-Atta@dz, there is a strong reflection at  $10.5 \text{ \AA}$  (110) and moderate ones at  $6.49 \text{ \AA}$  (200) and  $5.42 \text{ \AA}$  (130), which indicate the existence of palygorskite (Bradley, 1940; Serratos, 1978). According to XRD data, there are also characteristic peaks corresponding to other minerals such as quartz, cristobalite, gypsum,

**palabras clave:** Palygorskita, Cristalografía, Textura, Composición, Atta@dz.

**key words:** Palygorskite, Crystalchemistry, Texture, Composition, Atta@dz.

calcite and dolomite. The results indicate that the studied fibrous clays are carbonated palygorskites: one rich in calcite (C-Atta@dz) and the other rich in dolomite (D-Atta@dz).

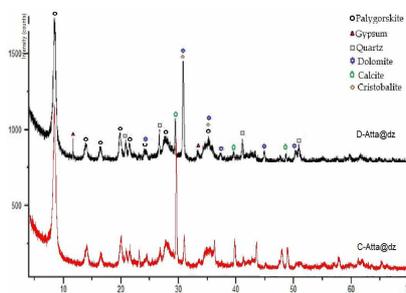


Fig. 3. XRD patterns of the D-Atta@dz and C-Atta@dz palygorskite.

### FT-IR spectroscopy

The FT-IR spectra (Fig 4) revealed the presence of the three water molecules of the fibrous clay as cited in the literature (Frost et al., 2003; Zandonadi et al., 1986): zeolitic water (two bands, one at 3383  $\text{cm}^{-1}$  and the second at 1657  $\text{cm}^{-1}$ ), coordinated water (band at 3544  $\text{cm}^{-1}$ ) and structural water (band at 3614  $\text{cm}^{-1}$ ). The absorption bands at 1437, 881, and 728  $\text{cm}^{-1}$  of C-Atta@dz are more intense than those of D-Atta@dz.

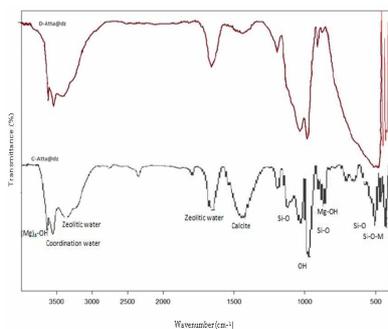


fig 4. FT-IR spectra of D-Atta@dz and C-Atta@dz palygorskites.

### X Ray Fluorescence

The chemical composition of the clays' varieties (Table 1) is dominated by  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$  and  $\text{CaO}$ . The amount of  $\text{CaO}$  in C-Atta@dz is greater than in D-Atta@dz.

### SEM

Secondary electron images of the D-Atta@dz palygorskite (Fig. 5a) show the needle-like form of particles, and confirm the fibrous aspect of palygorskite, constituting a pile of fibers.

	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$\text{MnO}$	$\text{MgO}$	$\text{CaO}$	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$	$\text{TiO}_2$	$\text{P}_2\text{O}_5$
C-Atta@dz	39,319	8,770	2,789	0,035	7,930	12,383	0,170	0,854	0,170	0,854
D-Atta@dz	40,317	8,873	3,266	0,061	8,876	9,687	0,209	0,890	0,449	0,104

Table 1. Chemical composition of Algerian palygorskite's varieties wt (%).

In addition, we notice the presence of rhomboid crystals (Fig. 5b), identified as dolomite.

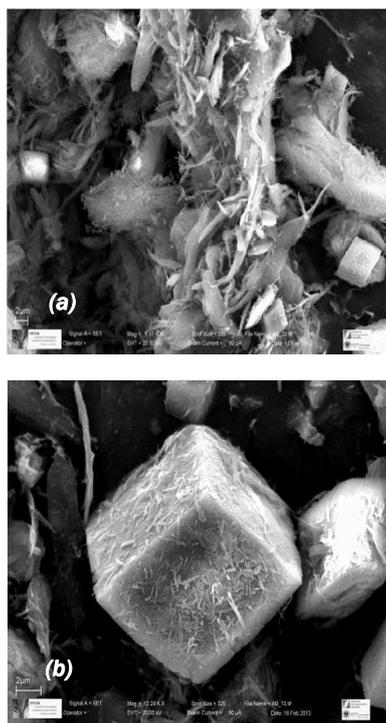


fig 5. Secondary electron image of D-Atta@dz palygorskite (a) rhomboidal dolomite (b).

### HRTEM

The observations by HRTEM done on a clay fraction of the D-Atta@dz palygorskite reveal clearly the fibrous microstructure of the clay mineral. The fibers measure between 2 and 5  $\mu\text{m}$  in length and between 10 and 50 nm of diameters (Fig. 6).



fig 6. HRTEM image of D-Atta@dz palygorskite.

### CONCLUSION

The results of this investigation show that the fibrous clays from East-Algeria are carbonate-rich palygorskites, with varying amounts of quartz, cristobalite, gypsum, dolomite and calcite, one rich in dolomite (D-Atta@dz) and the other in calcite (C-Atta@dz).

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