



Research paper

Characterization and traditional ceramic application of clays from the Douiret region in South Tunisia



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ABSTRACT

This study of the Aptian clays of Douiret (south-east of Tunisia) and their use in ceramic industry is original. At first, mineralogical, chemical, physical and thermal analyses of these clays are given. In terms of mineralogy, they can be considered as illitic clays (50–67%). However, other clay minerals, such as kaolinite, I/Sm mixed-layer, quartz, feldspar, dolomite and hematite, are present in small quantities. Next, this study reveals that the average amounts of silica and potassium are 53.9% and 3.3% respectively. The amount of alumina and iron oxide, with an average of 16.8% and 6.3% respectively, is variable. The plasticity test shows a high value ($PI = 39\text{--}56\%$). The mineralogical changes during the firing process were recorded via the X-ray diffraction of the raw clays and subsequent firing at 300, 700, 800, 900 and 1150 °C for 3 h with heating rate of 10 °C/min. The main transformations were observed at 900 °C with the appearance of new crystalline phases especially mullite, anortite and spinel. Technical tests show that the properties fall within the ceramic International Standards (ISO). Finally, ceramic tiles and bricks prepared from these clays have suitable characteristics without defects and can be classified in group BIII according to the European Standard NF EN 159 (1991).

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1. Introduction

Today, there is an increase in construction activity in Tunisia. The industries cannot meet the ever-increasing market demand for the construction materials needed. Few studies have been carried out on the clays or of raw materials used for traditional ceramic in the south Tunisia. For this reason, this work constitutes an inventory of useful substances which are widely present in the Saharan platform ridge. The Said inventory showed that this part of the country is rich in useful clay substances which stimulated the interest of researchers in studying the most important sites. The studied sector is Douiret series (see the geological map of Douiret No. 107 at 1:100,000 scale), located at 22 km in the south–west of the city of Tataouine and at 6 km from Douiret town (Fig. 1). The road Tataouine/Douiret passes by these series of clays.

The studied materials are outcropped at the base of the Chenini member and Zebbag formation. The Douiret formation consists of a thick, sandy and clayey material which outcrops mainly in south–east Tunisia (Fig. 2). The studied area is characterized by a grey clay mass with a thickness of 50 m and covered of thin Quaternary deposits, and

showing important reserves. The present study was carried out in order to characterize six samples of clay, collected from sedimentary Aptian Douiret deposit.

2. Materials and methods

2.1. Sample preparation

This study focused on the characterization of six samples named from the base D1 to tops of the section D6 taken from sedimentary Aptian materials (Fig. 2). In order to ensure a representative sample, obtained by quartering, no <50 kg of clays are collected. After extraction, they were initially dried at 105 °C until a constant weight was obtained, and then they were powdered for 30 min. To prepare the oriented aggregates, the clay fraction was purified through classical methods. The raw material was completely decarbonated by adding a small amount of 1 N HCl, and treated with H₂O₂ to eliminate any organic matter. The material was washed five times with 1 N NaCl and excess salt was removed by dialysis. The fraction <2 μm was separated in a centrifuge, and the suspension was dried at 60 °C (Grim, 1962; Środoń, 1981; Bergaya et al., 2006).

In order to perform the technological tests, the clays were humidified in 6% water content, mixed and sieved (Ø 1 mm) until a

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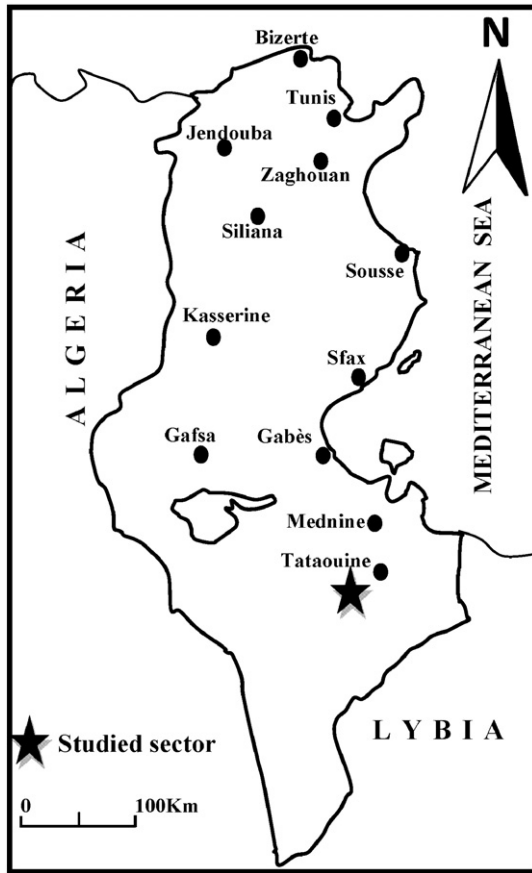


Fig. 1. The geographic location of the studied sector.

homogeneous paste was obtained. Specimen tiles were prepared using 60 g of green material per test piece by pressing them at 200–250 kg/cm² using 100 × 50 × 10 mm molds and then drying them in air at room temperature for one week and at 105 °C for 24 h.

2.2. Methods

X-ray diffraction (XRD) patterns were recorded on a Philips X’Pert diffractometer at the Energy Research and Technology Center (CRTE) using CuK α radiation (1.5418 Å), on the bulk or powder samples and on the oriented aggregates (natural (N) and treated with ethylene glycol (EG) and heated to 550 °C for 2 h (H)). The accelerating voltage and filament current were maintained at 40 kV and 40 mA, respectively. The percentages of the different mineral phases were estimated by measuring the areas of representative peaks, using X’Pert HighScore Plus software and PDF-4 (JCPDS- ICDD data base). The standard deviation was \pm 5%.

The major element compositions (SiO₂, Al₂O₃, Fe₂O₃, K₂O, Na₂O, MgO, CaO, MnO and TiO₂) were determined by atomic absorption spectroscopy.

Loss on ignition (LOI) was measured from the total weight after ignition at 1000 °C for 2 h. FT-IR spectra were recorded in the region 400–4000 cm⁻¹ in an EQUINOX model 55 infrared transform spectrometer, using the KBr pellet technique (about 2 mg of sample and 200 mg of KBr were used in the preparation of the pellets).

The cation exchange capacity was measured with the standard acetate ammonium procedure (Meir and Nuesch, 1999; Bergaya et al., 2006). The specific surface area was determined with the methylene blue index method. A suspension of 10 g of powdered clay and 100 ml of distilled water was prepared and mixed with the methylene blue solution (10 g/l). One drop of this suspension was then placed on a filter paper and the value for the specific surface was derived from the quantity of the adsorbed methylene blue (Ferrari and Gualtieri, 2006). This method is commonly used in earth sciences. It is used to classify the argillaceous fraction activity and to detect sensible clays like smectite.

For the fraction inferior to 63 μ m, the grain-size distribution was determined by a Micromeritics 5000 sedigraph.

The plasticity parameters (liquid limit (LL), plastic limit (PL) and plastic index (PI), $PI = LL - PL$) were defined by Proust et al., 2004, Ancy (2007); Yu et al. (2007); Modesto and Bernardin (2008). These parameters were determined by the Casagrande method (Casagrande, 1947; LCPC, 1987), in accordance with the French Standard NF P 94–051.

The differential thermal analysis and thermal gravimetric DTA/TG were obtained with a SETARAM type 124.

To typify the drying process, the Bigot curves were drawn with a barelattograph, which measures, within 24 h, the change in length and the shape of a slide by adding water to the raw powder until a normal paste that does not stick to fingers is acquired. Solid cast bar (length = 11 mm, trapezoidal section with area: $[15(15 + 17) / 2]$ mm²) were obtained by slip casting into suitable plaster molds. Just after demoulding, two pieces of approx. 40 mm were cut, weighed and carefully placed in the barelattograph (Taï and Ferreira, 1997, 1998). A sample was used to calibrate the apparatus weighing system. When the sample finished up shrinking, its drying continued at 110 °C. The weight (m) and length (l) of each slide were measured before recording and after complete drying. This method determines graphically the critical point which separates the two drying phases: In the first one, the sample shrinks as it gives off water. In the second phase, the external dimensions of the specimen remain slightly constant in spite of continued water extraction. The ratio ISS was calculated by: $ISS (\%) = \text{interlayer water} (\%) / \text{moisture water} (\%)$.

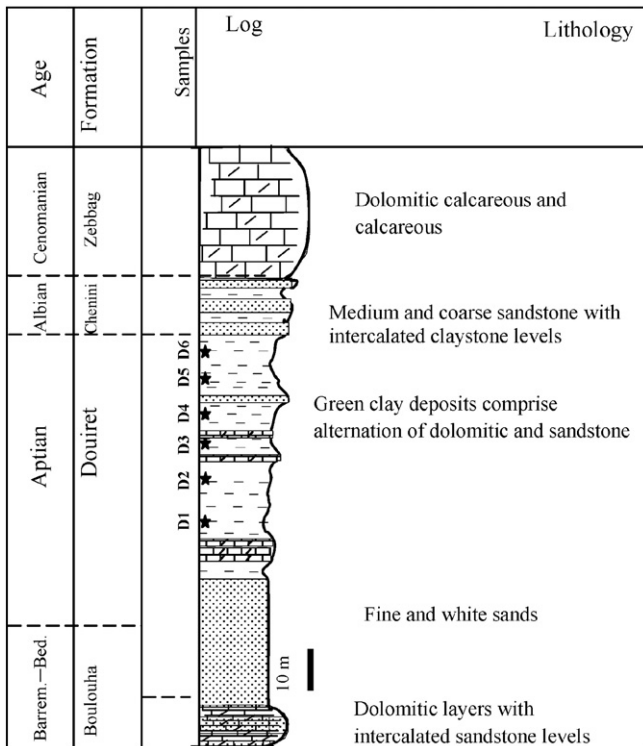


Fig. 2. Synthetic Lithostratigraphic column of the studied sector.

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