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## Mineralogy and plasticity in clay sediments from north-east Tunisia

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### ABSTRACT

Several cross-sections carried out in the Bir M'Cherga area (northern Tunisia) provided a complete Triassic-Miocene stratigraphic sequence, rather representative of the whole Tunisian Ridge Field located in the northern Atlas. Mineralogical analysis revealed a predominance of illite in the Early Cretaceous, while smectite is dominant in the Late Cretaceous and Tertiary. In terms of Atterberg limits, the Bir M'Cherga samples can be divided into two groups: one of moderately plastic clay samples until the Early Cretaceous, and another represented by the Late Cretaceous and Tertiary clays, which are the most plastic. As expected, the Atterberg limits increase with the amount of phyllosilicates present in the sample, which is dependent on the amount of smectite. This analysis was complemented by the use of the equivalent basal spacing (EBS) parameter, which gives a good correlation between the mineralogical characteristics of the clays and their plasticity. Using EBS, we can predict the mechanical/plastic behaviour of any clay sample according to its mineralogical composition.

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#### 1. Introduction

Interest in clays has increased in recent years due to their physical-chemical and plastic properties, which make them some of the most widely used materials in industry. Since Atterberg (1911) much work has been done in soils and sediments in an attempt to evaluate the influence of the various factors involved in the plasticity of clay samples, such as their mineralogical composition, shape and size distribution of particles, interaction among clays or with water or dissolved salts, the effect of cementing, clay genesis, etc. Casagrande published his well-known soil chart in 1948, and Dumbleton and West (1966) studied the relationships between clay contents and the plastic and liquid limits of natural soils from around the world, in an attempt to define the contribution of clay component to the engineering properties of soil as a whole. More specifically, Bain (1971) focussed on industrial clays (halloysite, kaolinite, illite, mixed layers, several kinds of smectites, sepiolite and paylygorskite), Decleer et al. (1983) correlated mineral composition, chemistry and granulometry with plastic and liquid limits in Belgian clays, and Hawkins et al. (1986) did similar analysis in the UK, while Al-Homoud et al. (1996) focussed on clay beds causing landslides and Ohtsubo et al. (2002) on marine clays.

Perhaps the most recent and conclusive contribution is by Schmitz et al., (2004) who introduced equivalent basal spacing (EBS), a parameter obtained by multiplying the relative amount of a clay with its basal spacing (Å) known from the literature. In this paper we report the Atterberg limits and the mineralogical composition of a significant number (approximately 120) of Tunisian clayeymarly samples in order to investigate their relationships with a good statistical significance. The Diffuse Double Layer (DDL) theory can explain mechanical disparity between different clay minerals (Wersin et al., 2004; Schmitz, 2006).

This study examines the relevant mineralogical parameters affecting the plasticity of samples, in order to easily distinguish and select those suited for certain technological applications. With such a large number of samples we can check the validity of the EBS method. Obtaining a direct prediction of plasticity based on this may be useful if other phases such as organic matter (Xiao Gang et al., 2002) or salts (Schmitz and van Paassen, 2003) are absent.

#### 2. Materials and methods

Our study area includes the Jebel Oust massif and neighboring areas (near the city of Bir M'Cherga) in north-eastern Tunisia



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Fig. 1. Simplified geological map of the study area and location of samples.

(Fig. 1). The sampled materials, all of which are marine sediments, belong to the Tunisian Dorsal Domain. The clayey-marly sediments analysed range from Triassic to Miocene in age.

The stratigraphic sequence begins with evaporitic and clayey facies in contact with massive Early Jurassic limestones overlaid by marly-calcareous alternations 380 m thick, which become progressively more clayey and reach thicknesses of up to 2240 m (Valanginian to Late Barremian). During the Albian new limestone and marly limestone levels appeared, coinciding with a transgressive evolution of the basin to more clayey sedimentation during the Coniacian–Santonian and ending in a massive 50 m thick white limestone level containing inoceramus fauna. The Cretaceous–Tertiary transition consists of marls and is topped by 35 m thick nummulitic limestones. The rest of the studied cross-section is represented by clayey sediments with some sandstone intercalations accumulated during the regressive Oligocene–Miocene period.

Mineralogical analyses on a total of 121 samples were carried out by X-ray diffraction (XRD, Siemens Cristalloflex 810 diffractometer, using Cu-K $\alpha$  radiation) on powdered bulk sediment and on the <2 µm fraction following the normal procedure for clay analysis, as described by Moore and Reynolds (1997). For bulk sediment analysis the relative abundance of minerals (including carbonates) was estimated from the height of the main peak. For clay fraction analysis the whole sediment was decarbonated with HCl (0.1 mol/L) and the <2  $\mu$ m fraction separated by settling in a water column. Samples were mounted as oriented aggregates on glass slides. The X-ray patterns were recorded in natural conditions (air-dried) and, after ethylene glycol and thermal treatment. Semiquantitative analysis was performed following Biscaye (1965), Zevin and Kimmel (1995), Moore and Reynolds (1997) and Kahle et al. (2002). The final percentages of the different mineral phases were calculated combining data obtained by XRD and chemical analysis, following the method used by Torres-Ruiz et al. (1994) and López-Galindo et al. (1996). The experimental error is ±5%.

Chemical analyses were carried out at the Scientific Instrument Centre of the University of Granada. Major elements were measured using Philips PW 1404 fluorescence equipment with a Cr/ Au X-ray tube and Be slit. Acceleration power applied was 10– 100 kV at intensities varying from 10 to 80 mA. Detection limit was 0.01%.

CaCO<sub>3</sub> content was determined using a Bernard calcimeter, on the basis of the volumetric analysis of the CO<sub>2</sub> liberated during hydrochloric acid leaching. The specific surface area of the 88 most clayey samples was estimated by the methylene blue method, consisting in titration of an aqueous solution (100 ml) including 10 g of clay. The point of optimum absorbed methylene blue dye ( $C_{16}H_8N_3SCl\cdot 3H_2O$ ) is given when the sample is saturated (Tran, 1977).

The Casagrande method (LCPC, 1987) was selected for the determination of Atterberg limits in some samples, since it gives more reliable results than the penetrometer cone, as suggested by different authors (Christaras, 1991; Grabowska-Olszewska, 2003). Experimental error is  $\pm 3\%$ .

#### 3. Results

#### 3.1. Mineralogy

The main mineral phases are phyllosilicates (illite, smectite, kaolinite, irregular mixed layers I–S, chlorite and palygorskite), calcite and quartz, while feldspars and dolomite are present as minor compounds. The mineral distribution throughout the stratigraphic sequence (Fig. 2) shows important variations: calcite is almost absent during the Triassic and Oligocene–Miocene, and is also minimal during the Hauterivian–Valanginian, coinciding with a maximum for quartz abundance, contemporary to strong sea-level regressions occurring at various periods (Hauterivian–Valanginian, Early Oligocene, Barremian). Some increases in calcite contents are associated with marine transgression (e.g., late Barremian), this phase reaching its maximum during the Late Cretaceous. By contrast, during the Tertiary the trend is the opposite, and the relative amount of phyllosilicates is near its maximum.

As regards clay minerals, illite, kaolinite and smectites are the main phyllosilicates, whereas interstratified I/S, chlorite and palygorskite are secondary. The dominance of illite is observed until the Upper Albian, and smectites are almost absent during this entire interval (Jamoussi, 2001). They appear in significant quantities only during and after the Cenomanian, in a net change observed in all Tunisia (Jamoussi et al., 2003), whose importance is also recorded by other clay minerals, as the I/S and chlorite distributions indicate. Kaolinite, on the other hand, is always present, but its amount increases notably during the Tertiary.

#### 3.2. Calcimetry

The percentage of  $CaCO_3$  shown by calcimetry is, as expected, very close to the amount inferred by XRD (see Fig. 2). Changes

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