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# Unraveling chalk microtextural properties from indentation tests

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ABSTRACT

The petrographical, petrophysical and geomechanical properties of the less commonly studied low permeability or tight chalks are presented in this study. The latter are relevant as potential unconventional reservoirs or intra-reservoir seals. Tight chalks encompass different lithotypes, in which the main factors controlling the petrophysical properties as shown in this study are the non-carbonate content and the degree of cementation. Those parameters strongly modify chalk microtexture and thus its porous network, reducing pore-sizes hence altering poroperm properties. In order to better understand the characteristics of tight chalks, an integrated petrographical, petrophysical and geomechanical study was carried out on a set of 65 chalk samples from Northwestern European outcrops, covering a wide range of lithotypes. The dataset gathered covers a broad spectrum of values for the determined petrophysical (e.g. porosities from 9 to 45%) and geomechanical properties (e.g. strengths from 3 to 60 MPa). In the framework of this study, indentation tests were performed on the chalk samples. This technique proved to be a successful method to quickly estimate rock strength. Indeed, a good linear correlation ($R^2 = 0.90$) has been established between indentation strength and UCS. Furthermore, indentation tests yielded valuable information about the chalk properties, both in terms of petrographical (cementation/clay-content), petrophysical (exponential relationship with porosity) and geomechanical (in relation to the elastic parameter and plasticity index) properties. This cheap and easy-to-operate method is a promising tool to indirectly estimate the mechanical parameters of chalks, when core samples are unavailable for laboratory testing in oil-field wells.

## 1. Introduction

Characterization of rock microtextures has been a focus of interest over the past decades, because microtexture is the key-link between a rock depositional/diagenetic history and its physical properties. Indeed, within many studies, textural characteristics have been correlated to petrophysical (e.g. Barone et al., 2015; Brigaud et al., 2014; Deville de Periere et al., 2011; Vincent et al., 2011), as well as geomechanical properties (Azzoni et al., 1996; Howarth and Rowlands, 1987; Kekec et al., 2006; Zorlu et al., 2008).

Pure porous reservoir chalks have been extensively described and characterized in terms of petrography as well as petrophysical and geomechanical characteristics, and the role of microtexture has been addressed (e.g. Duperret et al., 2005; Fabricius et al., 2007; Gommesen et al., 2007; Olsen et al., 2008; Richard et al., 2005; Schroeder, 2002). Mallon and Swarbrick (2008) were one of the first to address the large diversity of non-reservoir chalk lithologies which are present throughout much of the North-Sea Chalk Group. Low-reservoir quality or tight chalks recently became a focus of interest for petroleum industry since they might be underexplored reservoirs, or play a critical role in hydrocarbon migration, acting as seals or fluid conduits in the latter case depending on their fracture patterns. Characterization of tight chalks showed an unequivocal control of the microtexture on the petrophysical properties, including porosity, permeability, pore throat and body sizes (Fabricius and Borre, 2007; Gennaro et al., 2013; Faÿ-Gomord et al., 2016).

Knowledge of the rock mechanical properties is an essential factor in rock engineering projects (Fjaer et al., 1992; Garcia et al., 2008; Mateus et al., 2007) for example the unconfined compressive strength (UCS) is a critical parameter widely used to understand wellbore stability or reservoir compaction behavior (Chang et al., 2006). In highly porous rocks, porosity has been shown to control the mechanical behavior (e.g. Dearman et al., 1978; Dunn et al., 2002; Palchik and Hatzor, 2004). Typically, UCS values are either determined through laboratory tests on cylindrical rock samples, or inferred from acoustic well log interpretation combined with lithological analysis (Fjaer and Holt, 1999; Mateus et al., 2007). Consequently, core samples and well logs need to be available, which is not always the case. In addition, the cost involved and time required restrain UCS data acquisition in hydrocarbon-field wells (Chang et al., 2006; Santarelli et al., 1997; Zausa and Santarelli, 1995). Such

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limitations led to the development of techniques to indirectly estimate the mechanical parameters, when core samples are unavailable for laboratory testing. Therefore, a number of empirical relationships have been proposed to relate rock strength to parameters acquired from geophysical logging. Proposed correlations usually involve P-wave velocity (e.g. Fjaer et al., 1992; McNally, 1987; Moos et al., 2003; Santarelli et al., 1989), Young's modulus derived from velocity and density measurements (e.g. Bradford et al., 1998), or porosity derived from density measurements and assuming rock matrix and fluid densities (e.g. Horsud, 2001; Plumb, 1994; Vernik et al., 1993). Other nonconventional techniques involve laboratory tests on rock cuttings. The indentation or punching test is one of these techniques. It has recently been widely used to assess mechanical properties (i.e. UCS) using empirical correlations previously established (García et al., 2008; Haftani et al., 2013; Kahraman et al., 2012; Magnenet et al., 2009, 2011; Mishra and Basu, 2012; Ringstad et al., 1998; Tshibangu et al., 2014; Yagiz, 2009; Zausa and Santarelli, 1995).

This study offers a unique and detailed analysis of indentation measurements on a broad spectrum of chalk samples, especially tight chalks. Samples were selected from Northwestern European outcrops and include chalks from various depositional settings, with different diagenetic histories. As a result, a wide range of sedimentary textures, non-carbonate content, degrees of cementation and compaction is covered. This integrated multidisciplinary approach first aims to understand how the sedimentology-diagenesis-shaped microtexture controls the petrophysical and geomechanical properties of chalk. Indentation test results have then been carefully analyzed and compared to other parameters in order to (1) establish an empirical correlation between measured UCS and indentation strength in chalks, and (2) understand the influence of microtexture, especially cementation and non-carbonate content, on some mechanical properties of tight chalks.

## 2. Methods

### 2.1. Sampling

In the field, 65 chalk samples were collected, from carefully selected outcrops which reflect various sedimentological, tectonic and diagenetic histories, resulting in chalks displaying a wide range of lithology characteristics, reservoir properties and petrographical textures. The samples were collected from 17 different locations across France, England and Belgium. These Upper Cretaceous chalk samples stratigraphically range from late Albian to Campanian and deduced maximum burial depths range from 200 m (Harmignies Quarry, Mons) to 1800 m (Flamborough Head, Yorkshire, UK). Details regarding location, stratigraphy, and burial depth of the samples are provided in Table 1. Based on such a wide sampling, these samples cover a broad spectrum of chalk deposits.

### 2.2. Petrography

Chalk is a carbonate mudstone formed essentially of coccolith fragments (Hancock, 1975). Macroscopic petrographical features of the samples were observed, and then computerized tomography scans were performed for 3D petrography on 20 selected whole samples using a Siemens somatom scanner with a resolution of 0.2 mm. The heterogeneity of the chalk samples was characterized. The micritic fraction of the chalk displays different type of microtextures, depending on depositional history and diagenetic imprint (Fabricius, 2003; Mallon and Swarbrick, 2008). Hence, petrography analysis of the microtexture includes scanning electron microscopy (SEM) observation on fresh surfaces. Investigations were carried out on an EM XL30 FEG field emission microscope equipped with an energy dispersive spectrometer (EDS). SEM was used on all samples both to identify microporosity and to study microtextural features. Mortimore and Fielding (1990) used SEM observation to investigate the textural properties of pure chalks and quantify visually several parameters. In order to quantify the degree of diagenetic alteration, a "diagenesis index", developed by Faÿ-Gomord et al. (2016), was applied on all samples, from SEM observations based on seven criteria: micritic matrix texture, grain contacts, coccolith disintegration, cemented zones, authigenic calcite crystals, cement overgrowths and intraparticle cementation. The "diagenesis index" ranks the samples on a scale from 0 to 10, with zero (0) being the original chalk with no cementation and ten (10) being the completely cemented/compacted chalk. SEM investigations were also carried out on selected samples after indentation tests to observe how the microtexture had been modified by the penetration of the indenter.

Acid-insoluble residue measurements of the samples were obtained by dissolving roughly crushed samples of chalk in 2 M hydrochloric acid until no reaction was observed, followed by washing of the residue with deionized water. SEM observation and EDS analysis allowed the determination of the non-carbonate fraction.

### 2.3. Petrophysics

Petrophysical measurements were performed following the API (American Petroleum Institut) standards (API rp40, 1960). The porosity was determined by direct measurement of grain volume and bulk volume from 25.4 mm-diameter 4 cm-long plugs. The grain volume was determined by helium expansion in a Boyle's Law EPS Porosimeter. The grain density was calculated from the measured weight of the sample and the grain volume. The gas permeability of the samples was determined with a Vinci nitrogen permeameter. The plugs were mounted in a "Hassler" type core holder at a confining pressure of 400 psi and a steady state gas flow was established through the samples. The permeabilities were corrected for gas-slippage using Klinkenberg's empirical correlation.

### 2.4. Uniaxial compressive strength tests

Unconfined compression testing is a widespread measurement performed to mechanically characterize rock material. Tests were performed on cylindrical-shaped plugs samples of 1 in. diameter and a length/diameter ratio of 1.2–1.6. In order to work under standardized conditions, before analysis, all samples were dried in an oven at 60 °C until a stable weight was reached (Fleury et al., 2013), after 48 h. Tests were performed on a stiff frame with servo-controlled loading rate of 0.12 MPa/s. Pressure transducers were used to determine the axial stress (ranging from 0 to 35 MPa or 0–493 MPa depending on the used transducer). Axial stress records allowed the determination of the unconfined compressive strength (UCS). For 25 samples, inductive displacement transducers were added and allowed computation of axial strains, leading to a full record of the stress-strain curve. These curves were used in order to quantify the Young's modulus using the average modulus of the linear portion of axial stress-strain curve as defined by the ISRM proposed method (Bieniawski and Bernede, 1979).

### 2.5. Indentation

Because samples available from oil fields are not always large enough to allow UCS test, other techniques performed on smaller samples, such as the indentation test were developed. Indentation or punching test measurements were performed on 1 cm-thick dry block samples on a device based on Schreiner's theory (Brych et al., 1989; Descamps et al., 2014; Tshibangu et al., 1999). The system used in this study is based on a stiff frame equipped with an Enerpac MRT 22 hydraulic cylinder (Fig. 1). A flat tungsten carbide cylindrical indenter is forced into the samples under an increasing load until failure occurs. Instrumentation includes a 350 bar pressure transducer that allows computing the applied load and two LVDT (linear variable differential transformer) sensors (within a range of 1.3 mm) for measuring the displacement of the indenter.