Mechanical and physical behavior of high-porosity chalks exposed to chemical perturbation

M. Megawati [a,*], M.V. Madland [a,c], A. Hiorth [a,b,c]

[a] Department of Petroleum Engineering, University of Stavanger, N-4036 Stavanger, Norway
[b] International Research Institute Stavanger (IRIS), Stavanger, Norway
[c] The National IOR Centre of Norway, Norway

ABSTRACT

Extensive study on the effect of dissolution–precipitation on mechanical behavior of various high-porosity outcrop chalks (Liége, Aalborg, Kansas, Stevns Klint, and Mons) flooded with simplified aqueous chemistry at 130 °C under isotropic stress beyond the yield is performed. Chemical effects induced by injection of 0.219 M $MgCl_2$ solutions into impure chalks (Liége, Aalborg, Kansas) lead to an immediate enhancement on the macroscopic creep with more than a factor of 2 larger than that of exposed to 0.657 M NaCl solutions. In pure chalks (Stevns Klint and Mons) however, the creep response is characterized by a time lag, where creep initially diminishes before a tertiary-like creep develops. Systematic correlation between calcite dissolution and the resulting creep strain is consistently demonstrated by all the different chalk types.

The chemical effects are described as precipitation of Mg-bearing minerals and dissolution processes, which involve both the carbonate and non-carbonate phases. SEM-EDS, XRD, and BET (N_2) analyses indicate newly formed Mg-bearing minerals primarily present as Magnesite, which precipitated in the pore space. Enhanced dissolution is shown by continuous production of Ca^{2+} measured in the core effluent. The time for the dissolution to overcome intergranular friction accounts for the delay in the creep acceleration in pure chalks (Stevns Klint and Mons). For impure chalks (Liége, Aalborg, Kansas) chemical alterations on the non-carbonate phases outweigh the intergranular friction. This additional effect accounts for the immediate impact in the creep deformation.

The chemical effects are also demonstrated by marked reduction in the permeability. The porosity–permeability relationship measured at the end of creep test is shifted down from the initial correlation, indicating a dramatic increase in the chalk specific surface area.

1. Introduction

Dissolution and precipitation in porous media have significant implications for petroleum reservoirs not only related to the diagenetic history but also when the field is brought onto production. It is a major mechanism in the pressure solution processes, which have been well-documented in describing sedimentary and fault rock deformation (Durney, 1972; Rutter, 1983; Tada and Siever, 1989; Yasuhara et al., 2005), porosity–permeability evolution (Bjørkum and Nadeau, 1998; Ehrenberg et al., 2006), and development of stylolites in carbonate reservoir (Renard et al., 2004; Laronne Ben-Itzhak et al., 2012). One of the important implications of these processes is the formation of different pressure regimes in

North Sea central graben, which impacts fluid pressure release and build-up (Swarbrick and Osborne, 1998).

In the North sea, chalk reservoir compaction and seabed subsidence at the Ekofisk field has been a subject of extensive research. Chemical effects due to reactions between the chalk framework and the injected water are one of the major mechanisms in the so-called water weakening processes (Newman, 1983; Rhett and Teufel, 1991; Teufel and Rhett, 1992; Piau and Maury, 1994; Risnes and Flaageng, 1999; Risnes et al., 2005; Madland et al., 2011). Chemical dissolution precipitation have been investigated through various approaches such as numerical simulations (Pietruszczak et al., 2006; Lydzba et al., 2007; Hu and Hueckel, 2007; Hueckel and Hu, 2007), experimental studies (Hellmann et al., 2002b), as well as microscopic studies (Hellmann et al., 2002a). Despite numerous research, integrated study on direct correlation of dissolution precipitation effects and the mechanical properties are, however, limited.

Pore water chemistry and the presence of clays have been

* Corresponding author. Present address: Statoil ASA, N-4035 Stavanger, Norway.
Fax: +47 51 99 0050.
E-mail addresses: mmeg@statoil.com (M. Megawati),
merete.v.madland@uis.no (M.V. Madland), Aksel.Hiorth@iris.no (A. Hiorth).

shown to impact the kinetics of dissolution precipitation. It has been generally agreed that the presence of clays promotes pressure solution in chalk. In Baker et al. (1980) the presence of clay is shown to inhibit precipitation of calcite, but does not retard calcite dissolution. Weyl (1959) showed that the presence of thin clay seams facilitates diffusional transport in pressure solution process. Garrison and Kennedy (1977) pointed out that dissolution is most intense in layers that have high primary clay content. Fabricius and Borre (2007) also concluded that the contact between calcite and clay causes a disequilibrium for calcite dissolution.

In terms of chalk mechanical behavior, earlier works (Heggheim et al., 2005; Korsnes et al., 2006a,b; Madland et al., 2011) have shown that not only the aqueous chemistry of the pore fluid, but also the presence of non-carbonate minerals such as silica and clay minerals control the chalk mechanical behavior. In Madland et al. (2011) injecting $MgCl_2$ brine leads to supersaturation of new minerals which further shift the chemical equilibrium towards dissolution of calcite. When injecting NaCl brine with the same ionic strength, however, only a minor change in the aqueous chemistry was demonstrated.

The primary focus of the present study is to link dissolution–precipitation to the mechanical and physical properties of chalk, and to investigate how the variation in chalk mineralogy impacts the chemical and mechanical interactions. Long-term mechanical tests on various outcrop chalks were carried out. Chalk cores were loaded isotropically beyond their yield points and then a prolonged creep phase with continuous injection of different aqueous chemistry followed. Simplified brines with seawater ionic strength (0.219 M $MgCl_2$ and 0.657 M NaCl) were used. Since mechanical properties measured under isotropic compression are less scattered and highly repeatable as shown from previous studies (Risnes et al., 2003; Heggheim et al., 2005; Korsnes et al., 2006b, 2008; Zangiabadi et al., 2009; Madland et al., 2011), this type of test is chosen. Microscopic studies by Scanning Electron Microscopy (SEM) are included. High-porosity outcrop chalks of different mineralogical compositions and of different origins from Niobrara quarry Kansas US, Aalborg quarry and Stevns Klint Denmark, Liége quarry and Mons basin Belgium were used. These will also serve as a basis for geomechanical characterization of high-porosity outcrop chalks.

2. Materials and Method

2.1. Material properties

We used five different outcrop chalks representing high-porosity chalk with variable properties such as calcite purity, mineralogy, permeability, and specific surface area. The properties of the five chalk types are presented in Table 1. Porosity was calculated from the difference between the dry and wet weight after saturating the core plug with distilled water in a vacuum chamber.

Permeability was calculated from the measured pressure drop across the core plug where a constant rate was applied. Carbonate content was measured by 0.5 M NaOH titration after reacting approximately 0.3 g chalk powder with an excess of 0.5 M HCl. The accuracy of this method for chalk is 0.7% or better (Nguyen, 2008). Specific surface area (SSA) on bulk chalk sample was measured by BET (N_2) method in two different Labs using Coulter SA3100 and Micromeritics Gemini III 2375.

2.2. Rock mechanical test procedure

Rock mechanical testing was performed in a hydraulically operated triaxial cell similar with that of previous studies (Risnes et al., 2005; Korsnes et al., 2008; Madland et al., 2011). The aim has thus not been to capture strength variations from complete yield curves, but to select a repeatable test method in order to study the various chemical effects. The design of the Triaxial cell is such that the application of confining pressure is also compensated in the axial direction. Confining pressure and axial pressure were applied using hydraulic high pressure pumps. The cell is equipped with a heating jacket element with an external regulator system. The test temperature was set to 130 °C.

Vertical displacement was measured continuously using an LVDT (Low Voltage Displacement Transducer) with a resolution of 0.05 mm or better. For total strain calculation, displacement at the start of the isotropic loading was taken as a reference point.

Prior to mechanical testing, a core plug with a size 38.1 mm diameter and 70 mm long was firstly cleaned by flushing through 2 pore volumes (PV's) of Distilled Water (DW) with 0.5 MPa isotropic pressures. Differential pressure across the core was measured, and during the cleaning initial permeability was calculated (Table 1). After cleaning, pore pressure in the back side and confining pressure were simultaneously increased to 0.7 MPa and 1.2 MPa respectively. Thereafter, heating elements jacketing the Triaxial cell were switched on and the temperature was increased to 130 °C. The temperature is kept constant (± 0.2 °C) throughout the test by an external regulating system. When the temperature is stable at 130 °C, the respective brine was introduced and the system was then left to equilibrate overnight.

All the tests were performed isotropically beyond yield point, and then followed by a prolonged creep phase. The loading rates are about 0.2 and 0.3 bar/min for SK/MO/RL/ÅR chalks and KA chalk, respectively. Throughout the tests, brine was continuously injected with a fixed rate of 1 PV/day. The brine composition and initial pH are presented in Table 2. Effluent was collected periodically at the core exit and analyzed off-line by an Ion-Exchange Chromatograph (ICS-3000 Dionex) in attempt to study any chemical changes in and out of the core.

2.3. Time-dependent creep

Extensive creep tests were performed during a period ranging from 10 days to more than 300 days. Creep stress level is kept

Table 1
Physical properties of different chalk types used in the study. The number of samples measured for SSA is shown in the paranthesis. Grain density for all the chalk types is 2.7 g/cm³.

Chalk type	Age	ϕ (%)	κ (mD)	Carbonate content (%)	SSA (m²/g)
Stevns Klint (SK)	Maastrichtian	43–47	3–5	99.66	1.89₍₂₎
Aalborg (ÅR)	Maastrichtian	45–48	3–5	95.61	2.45₍₃₎
Liége (RL)	Campanian	40–42	1–2	94.84	3.90₍₄₎
Mons (MO)	Campanian	42–44	3–5	99.7	1.81₍₃₎
Kansas (KA)	Late Cretaceous	37–40	1–2	97.20	2.95₍₂₎

Table 2
Brine composition injected during mechanical tests.

Ions	0.657 M NaCl (mol/l)	0.219 M MgCl₂ (mol/l)
Cl^-	0.657	0.438
Mg^{2+}	0.000	0.219
Ca^{2+}	0.000	0.000
Na^+	0.657	0.000
Ionic strength	0.657	0.657
TDS (g/l)	38.40	20.84
pH	6.12	5.62