THE EFFECTS OF TEMPERATURE ON THE WATER WEAKENING OF CHALK BY SEAWATER

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ABSTRACT

Seawater is injected into the high temperature North Sea chalk reservoirs to improve oil recovery with great success. Increased compaction of the rock, which also is a significant contribution to the oil displacement, is observed in the water-flooded area. Very recent laboratory studies at high temperature documented that enhanced compaction took place when chalk cores under stress were flooded by seawater. The special composition of seawater regarding reactive ions towards the chalk surface appeared to be the reason. In this paper, two different water-weakening mechanisms are discussed: 1) Substitution of Ca^{2+} from the chalk surface by Mg^{2+} present in seawater (dolomitization) at inter granular contacts and 2) Chemical dissolution. The effect of temperature is opposite for the two mechanisms, and in the present paper, the impact of temperature on chalk compaction is studied both in a static and dynamic way using triaxial cells under hydrostatic conditions. Distilled water was used as a reference fluid in the static tests at 80-130 ℃. The mechanical strength of chalk containing seawater was opposite to chalk containing distilled water. With seawater, the chalk became weaker as the temperature increased, while using distilled water the chalk became stronger as the temperature increased. In the dynamic creep tests, at 70-90 ℃, the cores flooded with seawater were significantly weaker than cores flooded with seawater without sulfate, and the difference in mechanical strength decreased as the temperature decreased. The mechanism for enhanced chemical induced weakening of chalk is discussed.

1. INTRODUCTION

Chalk is an important oil reservoir rock in the southern part of the North Sea, and it is characterized as low permeability (1-3 mD) and with high porosity (40-50%). The high porosity was preserved due to an early invasion of hydrocarbons and creation of overpressure. The absence of normal diagenetic processes has resulted in a very small degree of cementation. The purely biogenic chalk surface is reactive, which has impact on the interaction between the rock surface and injected fluid.

The wetting nature of highly fractured carbonate reservoirs determines the potential of improved oil recovery by water flooding. Usually, carbonate reservoirs are less water-wet than sandstone reservoirs. Seawater appeared to be an excellent injection fluid in the Ekofisk chalk field where the oil recovery is now approaching 50%. It was documented from laboratory studies that seawater contained potential determining ions (Ca^{2+}, Mg^{2+}, and SO_4^{2-}) towards the chalk surface, which were able to improve the water-wetting nature of chalk (Strand et al. 2006; Zhang et al. 2006; Austad et al. 2005). Due to

increased affinity of SO_4^{2-} towards the chalk surface at elevated temperatures, the potential of seawater as wettability modifier increased drastically at high temperatures, i. e. T_{res}>100 ℃. The temperature of most of the chalk reservoirs in the North Sea is, however, in the range of 90-130 ℃. Cationic surfactant of the type $R-N^+(CH_3)_3$ dissolved in seawater is also an excellent wettability modifier towards various type of carbonates, even at a very low reservoir temperature (Standnes et al. 2000; Standnes et al. 2002.).

Oil production by pure pressure depletion leads to compaction of the high porosity chalk, which has been described as an important drive mechanism for oil recovery in chalk reservoirs (Ruddy et al. 1989). The contribution from compaction as a drive mechanism during the primary production phase at the Ekofisk reservoir was about 30%. After the primary production phase by pressure depletion, injection of seawater was used as a secondary IOR-method. Water injection as an IOR-method has two main purposes; (1) keep the reservoir pressure above the bubble point of the oil, (2) displace the oil. It is, however, well known that water weakens the chalk, and increased compaction of chalk is observed when oil is displaced by water. The mechanism for this water weakening of chalk is not fully understood, and several physico-chemical models have been suggested, i. e. change in capillary forces (Delage et al. 1996), increased stress solution (Hellmann et al. 1996), increased pressure on chalk grains caused by attraction of water molecules to the chalk surface (Risnes et al. 2005), chemical dissolution (Newman 1983; Heggheim et al. 2005). Injection of seawater in chalk reservoirs will lead to continued compaction, even if the reservoir is repressurised. The water weakening effect is the primary mechanism for compaction of the chalk reservoirs during the secondary production phase, which contributes to increased production and reserves, but loss of production wells. Thus, compaction due to water weakening will have positive and negative effects on the economics.

Very recently, Korsnes et al. (2006 a,b) observed that seawater at high temperature (130 ℃) lowered the mechanical strength of chalk dramatically compared to distilled water. Laboratory tests documented that Mg^{2+} was able to displace Ca^{2+} from the surface structure of the chalk, and in the presence of SO_4^{2-}, this displacement also took place in the thin films at the inter granular contacts. Based on several studies on the effect of varying the composition of the potential determining ions (Ca^{2+}, Mg^{2+}, and SO_4^{2-}), a chemical mechanism for the enhanced weakening of chalk by seawater at high temperature was proposed.

In the present paper, the effect of temperature on the mechanical weakening of chalk by seawater is studied. Knowing that the substitution of Ca^{2+} by Mg^{2+} decreases and the dissolution of chalk increases as the temperature decreases, the mechanical properties of chalk contacted by seawater must be strongly related to the actual temperature. Both static (cores aged in water) and dynamic (cores flooded with water) experiments were performed at various temperatures.

2. EXPERIMENTAL

2.1. Materials
Chalk: Outcrop chalk from the quarry of Stevns Klint near Copenhagen in Denmark was used. The main characteristics of this chalk are: Maastrichtian age; porosity between 45 to 50%; permeability 1–2 mD (1–2 × 10^{-15} m^2). The Stevns Klint chalk is mainly composed of fine graded matrix (96 wt%), whereas larger bioclasts (mostly uncemented foraminifera) constitute the remaining part of the rock (Milter, 1996). The cores were drilled from chalk blocks with an oversized bit, cooled by circulation of water. After drilling, the cores were dried overnight in an oven at approximately 110 °C. The cores were then shaped in a lathe and cut to the correct size, D = 37 and L ≈ 70 mm, and then dried a second time overnight at 110 °C before saturation.

Fluids: The chalk samples were saturated with fluids of different compositions i. e. artificial seawater (SSW), modified seawater without SO_4^{2-} (SSW−U) and distilled water (DW). The salinity was kept constant for the two brines by adjusting the NaCl concentration, Table 1.

2.2 Mechanical Tests
Static tests: Two series of static tests were performed at 130 ºC, 80 ºC, and room temperature. The cores for the different test series were made out of two different chalk blocks, and the tests series can, thus, not be compared directly since mechanical properties can be significantly different between two blocks. In *Test Series 1*, the cores were saturated with DW, and in *Test Series 2*, the cores were saturated with SSW and DW. In both cases, the cores were aged in their respective fluids for 4 weeks at 130 and 80 ºC and for 6 weeks at room temperature. For each of the temperatures, 3 cores were tested, and average values of the mechanical properties (hydrostatic yield point and bulk modulus, K) with uncertainties were reported. After aging the cores in pressure vessels containing the actual fluid, hydrostatic tests were performed at room temperature by using a Hoek cell. The cell has an external load frame where the force is measured with a load cell (±0.125 kN), and the axial displacement with a linear voltage displacement transducer (LVDT ±0.05 mm). The cell is not equipped for measuring lateral displacement. The confining pressure was established with a high pressure piston pump (±0.1 MPa). The strain rate for these tests was approximately 1%/h, and all tests were performed at drained conditions. Stress-strain plots were recorded automatically.

Dynamic tests: Two test series were performed at 70 and 90 °C. 2 cores were tested per fluid for the tests at 90 °C, and one core per fluid at 70 °C. The tests were conducted in a similar way as reported for the previous study at 130 ºC (Korsnes et al. 2006b). For each of the temperatures, the mechanical tests were performed using seawater (SSW) and seawater without SO_4^{2-} (SSW-U). The samples were saturated with the chosen injection fluid one day before the test started and stored in the fluid until the next day. During the entire test, the flooding rate was about 2 pore volumes per day (PV/D) with a pore pressure of 0.7 MPa. All tests were started by heating the cell to the chosen test

temperature (70 or 90 ºC), while increasing the effective stress to 0.5 MPa. Flooding of the core started when the set temperature was reached. Hydrostatic loading beyond yield to a stress level between 9.4 - 9.6 MPa was then performed, followed by a creep phase. During the creep phase, the stress was kept constant at this level. The strain rate for the hydrostatic tests was approximately 0.1 %/h, and the strain was logged continuously during the hydrostatic test as well as the creep phase. After flooding approximately 8-9 PV in the creep phase, the actual flooding fluid was exchanged with distilled water (DW). About 5-6 PV of DW was then injected before the fluid was changed back to the original fluid. In most cases the test ended after flooding 20 to 23 PV during the creep phase.

The cell used for the dynamic tests was a hydraulically operated triaxial cell with heating elements mounted on the outside. Three high-pressure pumps (±0.1 MPa) are used to operate the cell (confining pressure, axial pressure and fluid circulation). The cell was not equipped for measuring lateral displacement. The axial displacement was measured by an outside linear voltage displacement transducer (±0.05 mm) that followed the movement of the piston. The piston system of the cell is compensated so that the confining pressure is applied also in the axial direction. The pump in the axial circuit will thus provide only the additional axial pressure. An inconvenience of this set-up is that the axial displacement cannot be measured in a pure hydrostatic test. To keep the piston in contact with the sample, some pressure must be applied in the axial circuit. Thus, the axial stress will always be slightly higher than the confining stress as an alternative to pure hydrostatic tests in the cell. Thus, quasi-hydrostatic tests with a small additional axial stress were performed with this test cell. The heating system of the cell consisted of 6 heating elements; temperature was controlled by an external regulating system. The temperature inside the cell is measured by a resistivity Pt-100 element. During the experiments, the temperature was held constant, ±0.2 °C.

3. RESULTS AND DISCUSSION

3.1 Static Tests
In the hydrostatic tests, the hydrostatic yield point, σ_h, was determined and used as a measure for the mechanical strength. The yield point is defined by where the stress vs. strain curve starts to deviate from the linear trend. After aging the cores in the respective fluids, the hydrostatic tests were performed by recording stress-strain plots at room temperature. The average yield point was determined based on 3 separate samples. The discussion of the results from the static tests is based on the assumption that the difference in mechanical strength is established during the aging period. The cooling performed ahead of the hydrostatic tests and the test procedures were the same for all cores, and should therefore have the same effect on the mechanical behaviour.

Test series 1: The average yield point for the cores aged in distilled water is shown in Fig. 1a. The cores aged at 130 ºC have the highest yield point and are significantly stronger than the cores aged at 80 ºC and room temperature. Within the accuracy of the measurements, the cores aged at 80 ºC and room temperature appeared to be of similar strength. The difference in mechanical strength for the cores aged at 130 and 80 ºC (a

factor of 1.7) can be related to the solubility of chalk in distilled water provided that chemical equilibrium has been established between the pore filling fluid and the matrix. The observation that the cores aged at room temperature are somewhat stronger than the cores aged at 80 °C is, however, not in line with the expected solubility property of chalk, which increases as the temperature decreases. The reason may be that chemical equilibrium between the pore filling fluid and the chalk matrix has not been achieved at room temperature, even though these cores were aged 2 weeks longer. Normally, the rate of chemical reactions (in this case solubility) increases as the temperature increases.

Based on the stress vs. strain plots, obtained from the hydrostatic tests, the bulk modulus, K, is determined for each of the samples. The K–modulus measures the stiffness of the material, and is determined by the slope of the trend line in the elastic region. The data did not give any significant difference between the average K-modulus at the different temperatures, Table 2.

Test series 2: The average yield values for the two series including DW and SSW are presented in Table 2. The two series cannot be compared directly since the cores were sampled from different chalk blocks. Cores aged in DW at 80 °C have different average yield points, where the cores from test series 2 are significantly stronger than cores from test series 1. This is an indication that cores from test series 2 were stronger than cores from tests series 1. The average yield point for the cores aged in SSW at 130 and 80 °C is similar, about 6.9 MPa, Fig. 1b. Obviously, the mechanical strength of the chalk cores is no longer dictated only by the solubility of chalk, since the cores aged in DW at 80 °C from the same test series has higher yield point. The yield point at 130 °C using DW is significantly greater than the corresponding yield point using SSW.

The average K-modulus values are listed in Table 2. In this case, the K-modulus at 130 and 80 °C was similar indicating no difference in stiffness of the cores aged at 130 and 80°C, but the stiffness is significantly lower than for the cores aged in DW. The reason for the increased water weakening effect of SSW at high temperature will be discussed in more detail after presenting the results for the dynamic tests.

3.2 Dynamic Tests
The day before the start of the hydrostatic tests, the cores were flooded with approximately 1.5 pore volumes of the test brine at the chosen test temperature. An injection rate of 0.05 ml/min was used, which corresponded to about 2 PV/D. Therefore, the observed difference in chalk strength, as measured by the yield point, should be related to the fluid–rock interaction during this period.

Tests at 130°C: Previous results from mechanical studies at 130°C will be included for comparison with the new results obtained at 90 and 70 °C (Korsnes et al. 2006 b). The axial stress vs. axial strain correlation for all the tests at 130 °C is presented in Fig. 2a. Cores flooded with SSW, showed the lowest average value of the yield point, 6.3 MPa, while the cores flooded with SSW i. e. without SO_4^{2-}, SSW-U, had the highest average yield point value, 8.5 MPa. The samples flooded with SSW-U were a factor of 1.3 stronger than samples flooded with SSW. Results from the additional creep test with

constant flooding at 130 ºC is presented in Fig. 2b, and show that the cores flooded with SSW have much greater compaction compared to the cores flooded with SSW-U. After a flooding period of 9 PV, the average difference in axial strain corresponded to a factor of about 2.7

Tests at 90ºC: The axial stress-strain correlation for all the tests at 90°C is presented in Fig. 3a, and the average values of the hydrostatic yield point for the different fluids are presented in Table 3. After one day of flooding, distinct differences in yield point were observed. Cores flooded with SSW, showed the lowest average value of the yield point, 4.8 MPa, while the cores flooded with SSW-U, had the highest average yield point value, 7.3 MPa. The difference is significant. The samples flooded with SSW-U were a factor 1.5 stronger than samples flooded with SSW. These results are in line with tests performed at 130ºC using the same test procedure (Korsnes et al. 2006b). The results cannot be compared directly since the chalk cores were sampled from different chalk blocks, but the trends can be compared. Both at 130°C and 90°C there is significant difference in mechanical strength between cores flooded with brine containing SO_4^{2-} and cores flooded with SSW without SO_4^{2-}.

After reaching an axial stress level of about 9.6 MPa, additional creep tests at constant stress level and flow rate were conducted. Only one core with SSW was tested during the creep period, since axial compaction measurements could not be performed after the hydrostatic test for one of the two cores tested. All the results are summarized in Fig. 3b. Also during this creep phase, the core flooded with SSW obtained an enhanced compaction compared to the cores flooded with SSW-U. After a flooding period of 8-9 PV, the average difference in the axial strain corresponded to a factor of about 2.7. This is of course a very large effect. When disregarding the flooding period with distilled water, the difference in compaction between the SO_4^{2-} containing cores and those without SO_4^{2-} became even larger as the number of PV increased. This is evident from the slope of the respective curves.

After flooding between 8-9 PV, the flooding fluids were changed to DW, in order to check possible changes in the creep behaviour of the cores. The response from the cores flooded with SSW and SSW-U were different when switched to DW. There was an increase in compaction for the core flooded with SSW, while the compaction appeared unchanged for the cores flooded with SSW-U, Fig. 6. The reason for increased compaction with SSW was previously explained as dissolution of $MgCO_3$ formed at the inter granular sites (Korsnes et al. 2006b). Without SO_4^{2-} ions in the brine less $MgCO_3$ is formed at the inter granular sites. The solubility of $MgCO_3$ is much higher than for $CaCO_3$.

The substitution of Ca^{2+} at the chalk surface by Mg^{2+} present in the seawater was verified by detecting the concentration of the two ions in the effluent in a separate flooding test at 90 ºC. The concentration relative to the initial concentration present in SSW of the two ions is plotted in Fig. 4a. It is obvious that the concentration of Ca^{2+} increased while the concentration of Mg^{2+} decreased. Brine samples from the core flooding tests were

analyzed with a Spectroquant photometer NOVA 60, where Ca^{2+} and Mg^{2+} concentration was determined photometrically, and SO_4^{2-} concentration was determined by the turbidimetric method.

Tests at 70ºC: The axial stress-strain correlation for all tests at 70°C is presented in Fig. 5a, and the value of the hydrostatic yield point for the different fluids are presented in Table 3. As documented by the figure, there is no significant difference in the mechanical strength of the cores containing SSW (7.7 MPa) and SSW-U (7.3 MPa) during the hydrostatic loading phase. These results can be compared with the dynamic tests at 90°C since the cores are sampled from the same chalk block. The cores tested at 70 °C have the same mechanical strength as the cores tested with SSW-U at 90 °C.

After reaching an axial stress level of about 9.6 MPa, additional creep tests at constant stress level and flow rate were conducted. Only one core with SSW and another with SSW-U were tested during the creep period, and the results are summarized in Fig. 5b. Also during this creep phase, the core flooded with SSW had greater compaction compared to the core flooded with SSW-U. After a flooding period of 9 PV, the average difference in the axial strain corresponded to a factor of about 2.

After flooding 9 PV the flooding fluids were changed to DW and the cores responded differently to the change of flooding fluid. The core flooded with SSW continued almost with the same slope, while the slope for the core flooded with SSW-U flattened out. At 70ºC the substitution process of Ca^{2+} by Mg^{2+} is very low (Korsnes et al. 2006a) and small amounts of $MgCO_3$ will be formed at the inter granular sites, which will lead to less dissolution of $MgCO_3$ when flooded with DW.

The small changes in the concentration of Ca^{2+} in the effluent when flooding the chalk core with SSW supports the idea that water weakening of chalk by SSW due to chemical substitution is reduced with decreasing temperatures, Fig. 6.

3.3 Mechanism for Water Weakening by SSW.
Flow studies by Korsnes et al. (2006a) have shown that Mg^{2+} present in SSW is able to substitute for Ca^{2+} at the chalk surface at elevated temperatures. The concentration of Ca^{2+} in the effluent increased more than 50% when SSW was flooded slowly through a chalk core at 130 °C. At 100ºC, the concentration of Ca^{2+} increased by about 20%, but no significant increase in Ca^{2+} concentration was observed at 70ºC. From this study, it was verified that the concentration of Ca^{2+} increased about 10% when flooding the core with SSW at 90ºC, Fig. 4a, and only a very small increase was observed at the start of the flooding at 70ºC, Fig. 6. Previous studies by Strand et al. (2006) showed that the adsorption of SO_4^{2-} onto the chalk surface increased as the temperature increased. The relative concentration of actual potential determining ions in seawater is: $[SO_4^{2-}]\sim2\times[Ca^{2+}]$ and $[Mg^{2+}]\sim2\times[SO_4^{2-}]$. Thus, the relative concentration of positive and negative potential determining ions is determining the surface charge on chalk (Zhang et al. 2006). In the absence of SO_4^{2-} and at high temperature (130 ºC), Mg^{2+} was able to substitute Ca^{2+} at the positive chalk surface in the pore body without any extra water

weakening of chalk (Korsnes et al. 2006b). This is also the case at 90ºC. Ca^{2+} was released from the chalk surface when flooding with SSW-U, Fig. 4b, but no enhanced water weakening was observed, Fig. 3b. Enhanced water weakening of chalk can only take place if the strength of the inter granular contacts are reduced. The fact that enhanced water weakening of chalk only was observed when SO_4^{2-} was present in SSW, indicated that substitution of Ca^{2+} by Mg^{2+} at the inter granular contacts in the thin films only takes place when SO_4^{2-} is present, as schematically illustrated in Fig. 7 by Korsnes et al. (2006b).

The previously suggested mechanism for enhanced water weakening of chalk by seawater at elevated temperature is further supported by the dynamic studies performed at 90 and 70ºC. The mechanical behaviour of chalk in the presence of SSW was the same at 130 and 90ºC, i. e. the presence of SO_4^{2-} in SSW increased compaction due to substitution reactions at the granular contacts. At 70ºC, the mechanical strength of the chalk is not so sensitive to the presence of SO_4^{2-} (see Fig. 5a). In line with Fig. 6, the substitution of Ca^{2+} by Mg^{2+} is low at this temperature, but it still leads to enhanced compaction during the creep test (Fig. 5b). Thus, there exists a temperature below 70ºC that is the lower limit for enhanced weakening of chalk by seawater. Below this value, the mechanical strength is mostly governed by dissolution of chalk, which increases as the temperature decreases. The static experiments also support the suggested mechanism. The effect of temperature on the mechanical properties using seawater is, however, less pronounced because the reacting brine was not removed by flooding. Thus, besides the general water weakening of chalk, which is independent of the composition of the water, enhanced chemical induced water weakening will take place by using seawater at high temperature ($T_{res} \geq 70ºC$) due to the presence of SO_4^{2-} and Mg^{2+}.

4. CONCLUSION
The conclusion from this work can be summarized as:
- Enhanced compaction takes place when flooding chalk cores with seawater at high temperature (70, 90 and 130ºC) under hydrostatic stresses above the yield point.
- Depending on the number of PV flooded, the enhanced compaction using seawater compared to seawater without SO_4^{2-} can amount to several hundred %.
- The enhanced compaction using seawater is due to chemical substitution of Ca^{2+} at the chalk surface by Mg^{2+} present in seawater. Substitution at the inter-granular contacts of the chalk only takes place if SO_4^{2-} is present. Thus, SO_4^{2-} and Mg^{2+} are the key ions present in seawater causing the enhanced compaction.
- At 70 ºC, the chemical substitution of Ca^{2+} by Mg^{2+} in the presence of SO_4^{2-} is very low, but enhanced compaction was observed during creep tests.
- Static hydrostatic tests after aging the cores for weeks in different types of water confirmed the dynamic observations, even though the reacting brine were not displaced from the pores.

5. ACKNOWLEDGEMENT

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Table1. Brine compositions

Ions	SSW [mol/l]	SSW-U [mol/l]
HCO_3^-	0.002	0.002
Cl^-	0.525	0.583
SO_4^{2-}	0.024	0.000
Mg^{2+}	0.045	0.045
Ca^{2+}	0.013	0.013
Na^+	0.450	0.460
K^+	0.010	0.010
TDS [g/l]	33.39	33.39

Table 2. Results from mechanical tests for test series 1 and test series 2.

Aging temperature		Average yield point, $\overline{\sigma_h}$ [MPa]	Average bulk modulus, $\overline{K}$ [GPa]
130 °C, DW	Test series 1	10.3 ± 0.7	0.42 ± 0.02
80 °C, DW		6.1 ± 1.2	0.48 ± 0.06
Room temp, DW		7.4 ± 1.0	0.36 ± 0.08
130 °C, SSW	Test series 2	6.9 ± 0.2	0.35 ± 0.02
80 °C, SSW		6.9 ± 0.6	0.33 ± 0.02
80 °C, DW		7.7 ± 0.3	0.41 ± 0.02

Table 3. Yield points for cores flooded at 90°C and 70°C.

Fluid	SSW	SSW-U
$\overline{\sigma_h}$ [MPa], 90 °C	4.8 ± 0.4	7.3 ± 0.4
σ_h [MPa], 70 °C	7.7	7.3
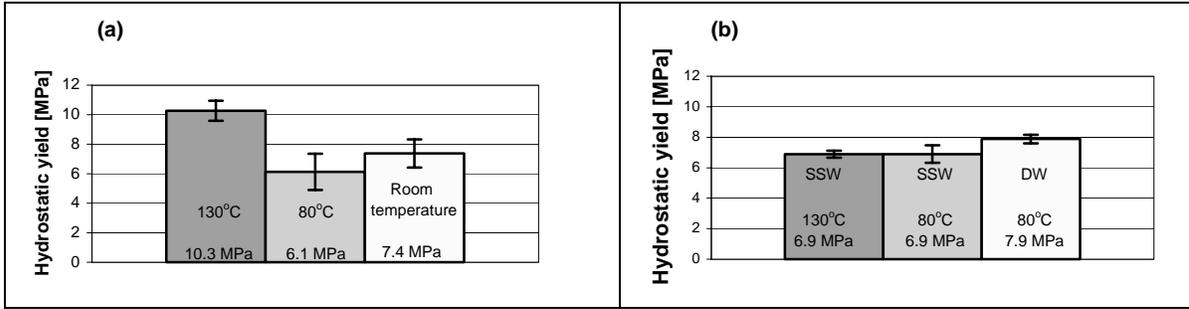
Figure 1. Average yield points for cores aged different temperatures. (a) Test series 1 (aged in DW) and (b) Test series 2 (aged in SSW and DW, as shown)
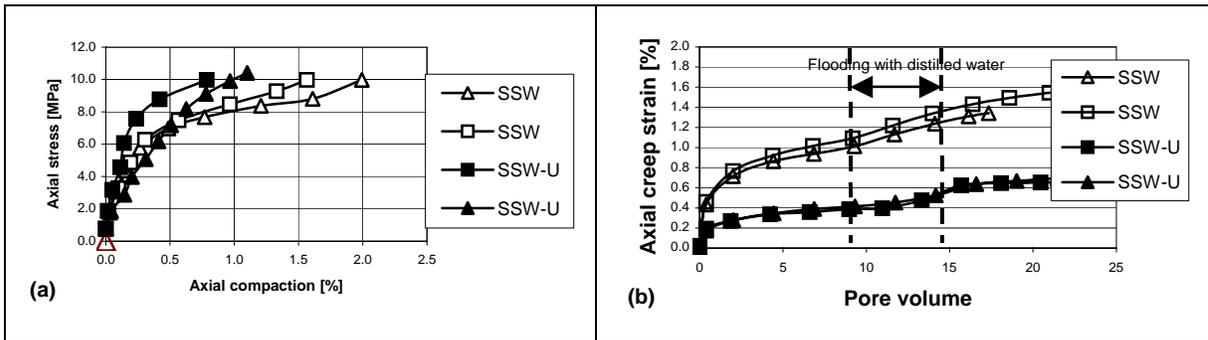
Figure 2. (a) axial stress vs. axial strain and (b) axial creep strain vs. PV, at 130 °C.
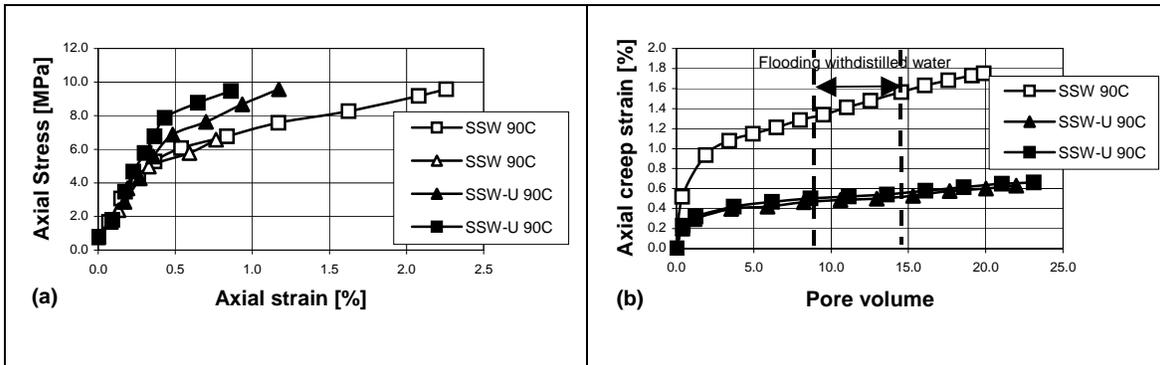
Figure 3. (a) axial stress vs. axial strain and (b) axial creep strain vs. PV, at 90 °C.
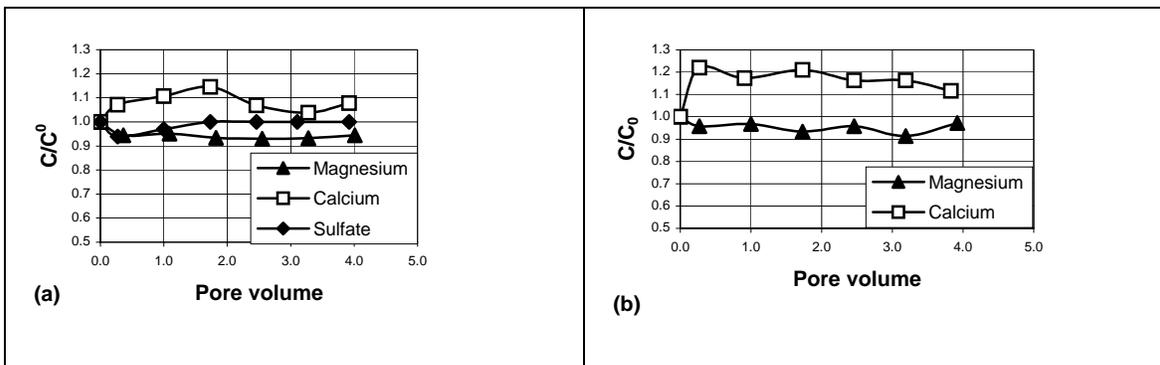
Figure 4. (a) C/C_0 vs. PV using SSW and (b) C/C_0 vs. PV using SSW-U, at 90 °C
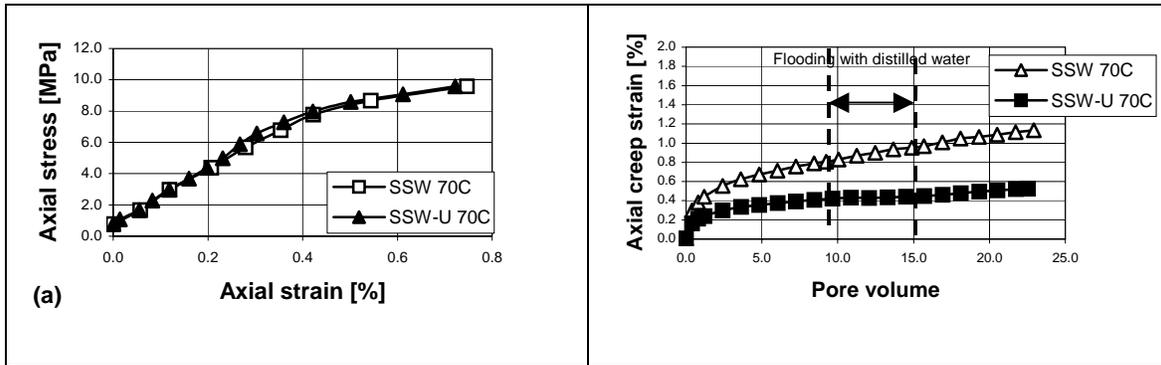
Figure 5. (a) axial stress vs. axial strain and (b) axial creep strain vs. PV, at 70 °C.
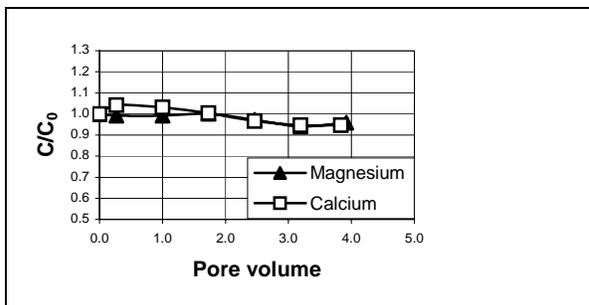
Figure 6. C/C_0 vs. PV at 70 °C using SSW
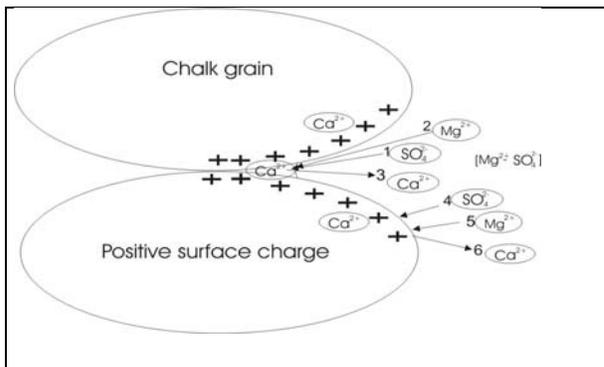
Figure 7. Suggested mechanism of enhanced water weakening in chalk