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ABSTRACT

Experimental data showed that water content has a profound influence on the uniaxial compressive strength of shale. Testing has shown great decrease in the uniaxial compressive strength as the water content increases. Regression analysis was used in this work to develop a general equation for predicting uniaxial compressive strength of shale from the available information on its water content and dry uniaxial compressive strength.

The impact of ionic diffusion on the compressive strength of shale has been investigated under three saturation conditions: wet shale, dry shale and chemically balanced wet shale. A chemically balanced shale has a water activity (chemical potential) which equals that of the test solution. Results show that, except for potassium ions, ionic diffusion has reduced the compressive strength of all studied shales. It has also been confirmed that diffusion osmosis has a detrimental effect on the mechanical stability of shale by reducing its compressive strength. Furthermore, it was found that when the water activity of shale is slightly higher than that of the test solution, chemical osmosis plays a major role in strengthening the shale by extracting water out of the shale. However, when the water activity of the shale is much higher than that of the test solution, diffusion osmosis weakens the shale. In other words, the detrimental impact of diffusion osmosis overtakes the beneficial effect of chemical osmosis.

Moreover, this work shows that compressive strength measurements for completely dried shale could be misleading due to the development of capillary forces that significantly modifies the compressive strength of shale.

Finally, the impact of ionic diffusion on the compressive strength of shale was carried out in the absence of both chemical osmosis and capillary forces. Results show that the invasion of sodium and calcium ions into shale reduced its compressive strength considerably while the invasion of potassium ions enhanced its compressive strength.

Keywords
Compressive strength; ionic diffusion; shale; water content; capillary forces; chemical osmosis
I. Introduction

Shales are the most troublesome formation to drill and cost the oil and gas industry around $1 billion annually [1]. Shales are characterized as fine grained, highly compacted and partially dehydrated sedimentary rocks. While shale related problems include bit balling, stuck pipe, high torque and drag and side tracking, wellbore instability is considered to be the most challenging problem when dealing with shale. Wellbore instability is thought to be caused by the unfavorable interactions between shale formations and drilling fluids [2]. Although such interactions including mechanical, chemical, physical, hydraulic, thermal, and electrical phenomena are very complicated [3,4], the overall effect of these interactions is directly related to the movement of water and ion into or out of shale [5]. Such movement could alter the physico-chemical and mechanical properties of shale around the wellbore, such as permeability, strength, pore pressure, and elastic modulus.

It is agreed upon that the properties of some argillaceous rocks are thought to be highly influenced by the adsorption of water and ions. For example, montmorillonitic rocks tend to swell and fail when contacted by low salinity water. Although, the literature is rich of quantitative data describing the behavior of montmorillonitic sandstone and pure clay when contacted with water, little is done to investigate the impact of water and ions on fine grained shales.

Researchers working with clays and clayey sands have found that clay swelling and expansion is caused by water and ions adsorption onto the electrically charged surfaces of clays [6-11]. They postulated that on a molecular scale, an ordering of the water molecules takes place on the clay causing the water to be rigid or captured. The degree of capture is best represented by an adsorption isotherm. The adsorption of water causes the clay platelets to separate thus inducing internal expansive stresses [12]. Water adsorption and subsequent expansion reduces the shale's compressive strength which ultimately causes shale failure and collapse.

The adsorption of water into shale could lead to shale strength and elastic modulus reduction, swelling and pore pressure increase [13]. In addition, the unfavorable adsorption of ions by shale could lead to shale strength reduction, cementing bonds deterioration, shale fabric alteration, pore fluid composition changes, all of which could lead to shale failure [14-19].

The compressive strength of shale under different water content conditions have been studied by many researchers [20-25]. Their studies show that water content of shale significantly changes most aspects of its physical and mechanical properties. In fact, such studies show that shale disintegrates rapidly when subjected to change in water content. This nondurable behavior of shale is thought to be responsible for most wellbore instability problems during drilling.

Van Eeckhout investigated the impact of water content on the strength of different shales [26]. His research showed a significant reduction in the strength of shale due to an increase in water content from dry condition to saturated condition. He lists five processes of strength loss in shales due to increased moisture content: fracture surface
energy reduction, capillary tension decrease [27], pore pressure increase, frictional reduction, and chemical deterioration.

Hsu and Nelson reported a strong correlation between compressive strength and water content for Cretaceous clay shales of north America [23]. Steiger and Leung reported that, in shales, unconfined compressive strengths measured with dry samples can be 2 to 10 times higher than for wet samples [22]. Colback and Wiid found that the compressive strength of quartzitic shale under saturated condition was about 50% of that under dry condition [28]. Lashkaripour and Ajalloeian studied the impact of water content on the compressive strength and elastic modulus of fine grained sedimentary rock [29]. Their work showed that the compressive strength and elastic modulus of shale is significantly influenced by its water content.

Chenevert reported that the compressive strength of shales was highly affected by its moisture content [12]. T. AL-Bazali et al showed that the shale physicochemical and mechanical properties of shale are greatly altered by water and ions invasion into the shale [30]. Other studies show that water and ions uptake into shale reduces its strength and causes shale failure [17, 31, 32, 7, 1, 33 and 5].

One way to combat wellbore instability issues is to minimize the overall effects of water and ions on shale. This paper presents experimental data that shows the detrimental impact of water and ions on shale uniaxial compressive strength. The main focus of this work is to experimentally prove that water and ions uptake into shale could reduce its inherent strength and eventually causes shale failure.

II. Impact of Water Content on Shale Uniaxial Compressive Strength

The main goal of this test is to establish a correlation between the measured uniaxial compressive strength of shale and its water (moisture) content. To investigate the impact of moisture content on the mechanical behavior of fine-grained shales, three different shales were used (shales; A, B & C). These shales were donated by an oil company that operates in the Middle East. The mineralogical composition and petrophysical properties of these shales are shown in Tables 1 and 2 respectively.

Water Content Determination

In order to investigate the impact of water content on the compressive strength of shale, we needed to vary the water content of shale samples by heating the shale samples in an oven for different periods of time. Therefore, 10 cubical samples of shales A, B and C of dimensions 1.5" x 1.75" x 1.75" were cut, weighted and placed inside an oven and the temperature was set to 120C. This temperature should be high enough to cause free water(pore water) evaporation without affecting the bound and hydration water contained within the clay platelets [34]. At different time intervals (time = 0, 2, 4, 6, 8, 10, 12, 14, 16 and 24 hours), one sample is taken out of the oven and its water content is measured. The actual (native) water content (WCa%) of the shale was obtained from the samples that were oven dried for 24 hours as follows:
WCa % = (Ws – Wcd ) / Ws  …(1)

Where;
Ws is the weight of saturated sample.

Wcd is the weight of completely dried sample (after 24 hours).

The time dependent water content was estimated as follows. I will explain the water content determination for the sample that was dried for 2 hours as an example. The same procedure applies to the other samples that were dried for 4, 6, 8, 10, 12, 14, 16 and 24 hours. After 2 hours, the sample was taken out and weighted (Wt=2) and then placed back in the oven to dry for 24 hours. After 24 hours, the sample was taken out again and weighted (Wt=24). The water content for this sample after initially drying it for 2 hours is estimated by:

\[ WCt=2\% = (Wt=2 – Wt=24) / Wt=2 \] … (2)

Table 3 shows the time dependent water content of shales A, B and C samples as a function of drying time.

**Uniaxial Compressive Strength Determination**

In order to study the effect of water content on the shale compressive strength, shale samples were placed inside a uniaxial test chamber (compression machine) to measure its compressive strength. Prior to applying an axial load, a confining pressure of 34.5MPa was applied over a 10 min period, then was held constant for one hour to make sure that the pore pressure within the sample has completely dissipated before axial load is applied. A 3x10-5 s-1 axial strain rate was applied until failure occurred. During axial loading under this strain rate, pore pressure within shale sample might build-up over time. Therefore, the measured strength is qualitative, and they have significance only in relative terms.

After establishing shales A, B and C water contents as a function of drying time, ten cylindrical samples of shales A, B and C were cut and placed inside the oven to dry for different intervals of time (0, 2, 4, 6, 8, 10, 12, 14, 16 and 24 hours). At each time period, a shale sample is taken out of the oven and placed inside a standard uniaxial compression machine in order to measure its uniaxial compressive strength. Namely, the compressive strength of each sample is measured at a water content measured at the same drying time. Table 3 shows measured uniaxial compressive strength for ten samples of shales A, B and C under different water content conditions. This table shows a reduction of more than 94% in the compressive strength from oven-dried to saturated condition. This strength reduction due to moisture content for the shales studied is significantly higher than the values reported in the literature.

The measured compressive strength of shales A, B and C samples under different water content conditions have been presented graphically in figures 1, 2 and 3 respectively. These figures show a strong correlation to an exponential relationship between uniaxial compressive strength and water content as follows:
c = 91.598 e-0.443w \quad \cdots (3) \\

Coefficient of correlation \( R^2 = 0.994 \)

c = 86.2 e-0.496w \quad \cdots (4) \\

Coefficient of correlation \( R^2 = 0.972 \)

c = 86.895e-0.419w \quad \cdots (5) \\

Coefficient of correlation \( R^2 = 0.9762 \)

Where;

\( c \) is the uniaxial compressive strength in MPa.

\( w \) is the water content in \%. 

Figures 1, 2 and 3 show a consistent trend of decreasing compressive strength with increasing water content for all three shales. A correlation was made for all tested shales as shown in figure 4 and a general equation was derived as follows:

\[
c = 86.976e^{-0.444w} \quad \cdots (6)
\]

Coefficient of correlation \( R^2 = 0.9668 \)

Equation 6 clearly shows that the water content significantly affects the uniaxial compressive strength of shale. A more general equation that describes the relationship between the compressive strength of shale and its water content can be induced from equation (6) as follows:

\[
c = \text{dry}e^{-0.444w} \quad \cdots (7)
\]

Where; \( c \) is the uniaxial compressive strength at variable water content in MPa, \( \text{dry} \) is the uniaxial compressive strength at dry condition in MPa.

\( w \) is the water content in \%.

It is clearly shown from the above analysis that the water content of shale significantly affects its mechanical stability by reducing its compressive strength. Furthermore, equation (7) shows that shale's compressive strength decreases with an increase in its water content. This is mainly attributed to the adverse effects of water on shale mechanical properties such as compressive strength and friction angle. Namely, the increase in water content of shale leads to mechanical strength deterioration, cementing bonds weakening, shale fabric alteration, pore pressure increase and overall shale failure. Also, the shale pore fluid composition could be altered due to the increase
in water content which could destabilize the ionic chemical balance in the shale pore fluid. This could initiate ionic diffusion into or out of shale which causes further instability and strength reduction.

III. Impact of Ions on Shale Uniaxial Compressive Strength

During shale and drilling fluid interaction, both water and ions flow into and out of shale depending on the direction of their respective concentration gradients. Water will flow into shale if the water activity of the drilling fluid is greater than that of the shale and expectedly water will flow out of shale if the shale’s water activity is greater than that of the drilling fluid. The mechanism controlling the flow of water into and out of shale is called chemical osmosis. Similarly, ions will flow into or out of shale in the presence of ionic concentration gradient between the shale and the drilling fluid. The diffusion of ions is dominated by a concentration gradient that can be expressed by using Fick’s law:

\[
J = -D_{si} (C_{i,\text{shale}} - C_{i,\text{mud}}) / (X) \quad \ldots(8)
\]

where \(J\) is mass flux of \(i\)th ion; \(C_{i,\text{shale}}\) is the concentration of \(i\)th ion in pore fluid; \(C_{i,\text{mud}}\) is the concentration of \(i\)th ion in mud; \(D_{si}\) is the the diffusion coefficient of the \(i\)th ion; and \(X\) length of shale.

The flow of ions is normally coupled with the flow of its associated water (water cloud). The mechanism describing the flow of ions and their associated water is called diffusion osmosis. It is agreed upon that diffusion osmosis has a detrimental impact on shale stability during drilling. The flow of ions and their associated water could lead to various destabilizing events including and not limited to:

- Compressive strength alteration
- Elastic moduli alteration
- Cementing bonds weakening
- Matrix fabric deterioration
- Pore fluid composition alteration
- Micro fractures and fissures initiation
- Shale swelling and expansion
- Overall shale collapse
Although many researchers have conducted much work to verify the occurrence of diffusion osmosis, little has been done to investigate its impact on the shale mechanical integrity. The following presents an experimental investigation that sheds light on the detrimental impact of ionic flow on the compressive strength of shale, using standard uniaxial compression testing, for three cases: wet shale, dry shale and chemically balanced wet shale.

The water activity (chemical potential) of a chemically balanced wet shale is the same as that of the test solution.

**Case I: Wet Shale**

In this test, seven samples of shales A, B and C were immersed in different concentrated solutions for a month before subjecting them to uniaxial compressive testing. The compressive strength of each immersed shale sample is measured and compared to that of the native shale sample. The native compressive strength of shales A, B and C was measured after immersing the shales in a simulated pore fluid for a month. Table 4 shows the concentrated salt solutions and their respective relative humidity (water activity) that were used to immerse the shale samples. Table 5 shows the measured compressive strength of the altered shale samples after immersing them in the different concentrated salt solutions. Figures 5, 6 and 7 compare measured compressive strength of shales A, B and C samples after immersion in different concentrated salt solutions with their native compressive strength.

A closer look at the data presented in figures 5, 6 and 7 yields the following observations.

1. Comparing to their native compressive strengths, the compressive strength of shales A, B and C samples was reduced after immersion in concentrated K2SO4 solution. This could be attributed to the osmotic flow of water into the shale samples, shale swelling phenomenon, since the water activity of the K2SO4 solution is 0.98 which is higher than the water activities of shales A, B and C. I believe that the flow of water into the shale increased its pore pressure and dissolved the cementing bonds between the grains which has resulted in the overall strength reduction of the shale samples.

2. When immersed in KNO3 solution (aw = 0.94), the compressive strength of shales A and C samples has increased while the compressive strength of shale B has decreased. I think that the strength enhancement for shales A and C is mainly due to the osmotic flow of water out of the shale samples since the water activity of the shale samples is higher than that of the KNO3 solution. The flow of water out of shales A and C could have decreased its pore pressure and increased the friction between the grains which has led to the observed strength enhancement. In addition, potassium ions in the solution could have invaded the shale samples and contributed to its strength enhancement. Horsrud et al. found that the adsorption of potassium ions causes shale shrinkage and therefore enhances its strength [35]. As for shale B, the small loss of
strength could be attributed to the osmotic flow of water into the shale since its water activity is less than that of the KNO3 solution.

3. The compressive strength of shales A, B and C samples has increased after immersion in KCl solution (aw = 0.86). This could be due to both the osmotic flow of water out of the shales and the diffusion of potassium ions into the shales.

4. The compressive strength of shales A, B and C has decreased after immersion in NaCl, Ca(NO3)2, CaCl2 solutions despite the fact that the water activities of shales A, B and C were higher than that of the solutions. This is counter intuitive since one would expect that the shales would become stronger owing to the osmotic flow of water out of the shales as a result of the imposed water activity gradient (chemical potential). I believe that the observed strength reduction was mainly due to the flow of ions and their associated water into the shale owing to the imposed ionic concentration between the shales and the solutions. This phenomenon is referred to as "diffusion osmosis". The flow of ions and their associated water into the shales could have dissolved the cementing bonds between the grains, reduced the friction between the grains, introduced micro fractures and fissures, caused shale swelling and expansion, increased shale pore pressure and ultimately caused strength reduction. In other words, the damaging effects of diffusion osmosis has far exceeded the beneficial effects of chemical osmosis.

5. All shale samples have crumbled when immersed in ZnCl2 solution. I believe that the shale failure is mainly due to the damaging effects of the invasion of ions and their associated water into the shale as explained in point 4.

Case II: Dry Shale

In order to only investigate the impact of ionic diffusion on the compressive strength of shale, I needed to eliminate the effect of water content on the shale's compressive strength. The following procedure was adopted to establish the impact of ions on the mechanical behavior of shale.

• Seven samples of shales A, B and C were immersed in the aforementioned saturated salt solutions for a month.
• After a month, these samples were taken out of the solutions and placed inside the oven where the temperature was set to 120C.
• After 24 hours of heat drying inside the oven, the shale samples were placed inside a uniaxial compression machine and its compressive strength were measured.

Figures 8, 9 and 10 show measured compressive strength of dried shale A, B and C samples after immersion in different concentrated salt solutions respectively. It can be seen from these graphs that when drying out the shale samples, its compressive strength was higher than those obtained when the shale samples were not dried out. The same observation was made by Steiger and Leung when they reported that, in shales, unconfined compressive strengths measured with dry samples can be 2 to 10 times higher than for wet
Samples (30). Also, Colback and Wiid found that the compressive strength of quartzitic shale under saturated condition was about 50% of that under dry condition [28].

The compressive strength enhancement could mainly be due to capillary forces. Many argue that during shale heat drying, air or vapor could invade the pore throats and capillary forces could develop and modify the shale's compressive strength [36-38]. It is very difficult to quantify the strength enhancement due to capillary forces but one can argue that if such forces did not exist, the compressive strength of the shale samples would have been much lower. This argument is supported by the fact that many researchers have reported that ionic diffusion, except for potassium, weakens shale and alters its mechanical properties [12,18,30,7,5 and 35]. To prove this point, the following experiment was conducted.

**Case III: Chemically Balanced Wet Shale**

In order to investigate the impact of ions on the shale’s compressive strength without capillary forces development and chemical osmosis involvement, the following experimental procedure was adopted.

- Prepare three sets of NaCl, KCl, CaCl2, and Ca(NO3)2 solutions of 0.96, 0.92 and 0.97 water activities. This is done by controlling the amount of salt added to deionized water. Figure 11 shows water activities of NaCl solution as a function of NaCl concentration in the solution. Water activity charts for KCl, CaCl2 and Ca(NO3)2 solutions are widely available in the literature.

- Obtain 4 samples of shale A (aw = 0.96) and immerse each sample in the 0.96 water activity solutions. Namely, the first sample is immersed in 0.96 NaCl solution, the second sample is immersed in 0.96 KCl solution, the third sample is immersed in 0.96 CaCl2 solution and the forth sample is immersed in 0.96 Ca(NO3)2 solution. Making the water activity of the solution the same as that of the shale should stop the osmotic flow of water between the shale and the test solution. However, ionic flow will take place due to the presence of ionic concentration imbalance between the shale and test solution.

- Similarly, Obtain 4 samples of shale B (aw = 0.92) and immerse each sample in the 0.92 water activity solutions. Also, 4 samples of shale C (aw = 0.97) were immersed in 0.97 water activity solutions.

- After one month of immersion in the test solutions, place shale A, B and C samples inside a biaxial compression chamber and measure their compressive strengths.

Figures 12, 13 and 14 show measured compressive strengths of shales A, B and C after immersion in 0.96, 0.92 and 0.97 salt solutions respectively. These compressive strengths were measured in response to ionic diffusion only and in the absence of chemical osmosis and capillary forces. It can be clearly seen from these results that the compressive strength of shales A, B and C were considerably lower when compared to
figures 5, 6, 7, 8, 9 and 10. This is attributed to the absence of chemical osmosis and capillary force that contribute to the shale’s strength enhancement. The following key points were deduced from figures 12, 13 and 14.

- The compressive strength of shales A, B and C samples was considerably reduced when immersed in NaCl, CaCl$_2$, and Ca(NO$_3$)$_2$ solutions having the same water activity as that of the shale. The invasion of sodium and calcium ions could have weakened the shale. Diffusion osmosis played a detrimental role on the mechanical integrity of these shale as discussed previously.

- The compressive strength of shales A, B and C was enhanced when immersed in KCl solution. This could be attributed to the strengthening effect potassium ions have on shale as discussed previously.

- When immersed in the same solution, the compressive strength reduction is different for each shale. This could be due to the different ionic composition of the pore fluid for each shale. Recall that diffusion osmosis depends on the ionic concentration imbalance between the test solution and the shale pore fluid [8, 5].

IV. Conclusions

The impact of water content and ionic diffusion on the compressive strength of shale has been experimentally investigated. It has been shown that water content of shale has a great effect on its compressive strength. It was proven that as the water content of shale decreases, its compressive strength increases. Regression analysis was used in this work to develop a general equation for predicting uniaxial compressive strength of shale from the available information on its water content and dry uniaxial compressive strength.

The impact of ionic diffusion into shale has also been addressed experimentally. The following key points were deduced from these experiments.

- Chemical osmosis is highly effective when shale interacts with dilute solutions.

- When interacting with concentrated solutions, the detrimental impact of diffusion osmosis far exceeds the beneficial effect of chemical osmosis.

- The compressive strength of all shales studied has improved when immersed in KCl solution.

- The flow of water and ions into shale reduces its compressive strength while the flow of water out of shale enhances its compressive strength.

- Compressive strength measurements for completely dried shale could be misleading due to the development of capillary forces that significantly modifies the compressive strength of shale.

- In the absence of chemical osmosis and capillary forces, ionic diffusion into shale reduces its compressive strength and affects its mechanical stability.
REFERENCES


