Ion Movement and Laboratory Technique to Control Wellbore Stability

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Abstract

The movement of water and ions into or out of shale causes physico-chemical and mechanical property alterations, and can lead to wellbore instability related problems.

This paper presents experiments used to analyze the effects of chemical osmosis, diffusive osmosis, and capillary suction on water and ion movement when shale interacts with drilling fluids. Results show that water movement is not only controlled by chemical osmosis (water activity), but is also influenced by diffusive osmosis and capillary suction. The immersion of shale into fluids changes the chemical composition of the shale due to ion movement, and thus its physico-chemical and mechanical properties are altered. The gravimetric-swelling test (GST) is used in the laboratory to acquire a better understanding of the relationship between water flow, ion flow, and shale swelling. This insight provides information used to optimize drilling fluids to effectively control or mitigate wellbore instability when drilling through shale.

Introduction

Wellbore instability in shale formations has been a significant problem in oil and gas industry since its inception. The primary cause of this problem is the unfavorable interactions between shale formations and drilling fluids [1-3]. Although such interactions including mechanical, chemical, physical, hydraulic, thermal, and electrical phenomena are very complicated [3-6], the overall effect of these interactions is directly related to the movement of water and ions into or out of shale [7]. The physico-chemical and mechanical properties of shale around the wellbore, such as permeability, strength, pore pressure, and elastic modulus are altered due to such movement.

It is well-accepted that the adsorption of water results in shale strength and elastic modulus decrease; swelling and pore pressure increase [1, 8-11]. These changes around the wellbore may cause formation instability problems during drilling and completion operations. The magnitude of water movement and the effect of absorbed water on shale properties are influenced by the presence of ions in the solution [7]. Ionic diffusion may also result in the movement of ions into the shale formation, resulting in chemical alteration [12]. Horsrud et al. (1998) found the adsorption of potassium ions caused shale shrinkage due to cation exchange [13]. Simpson and Dearing (2000) demonstrated in their lab that ions diffusion altered the fabric of the shale and caused shale failure [14].

From the above review, it was found that the movement of water and ions into or out of shale is critical to wellbore instability. Zhang et al. developed a Gravimetric-Swelling Test (GST), which has been used to quantitatively measure water and ions movement during shale/mud interaction. Results of a study were published in 2004 which indicated water and ions movement by performing various gravimetric and swelling experiments using several different shale cores and ionic solutions [7]. This approach has also been used to better understand the design of emulsion based fluids as described by Rojas [15].

Mechanisms Controlling the Movement of Water and Ions

The mechanisms driving water and ions movement are convection and chemical activity. The hydrostatic pressure difference between a drilling fluid and the formation pore fluid causes bulk flow, including both water and ions movement. The basic chemical property involved in water and ions movement is water activity, although the literature often refers chemical effects to various mechanisms as chemical osmosis, ionic diffusion, and capillary effects [5, 11, 14, 16].

Chemical Osmosis

By definition, the chemical potential of an aqueous system is the partial molar free energy of the water with respect to a given component at constant pressure and temperature. It is this coefficient that describes the way the Gibbs free energy of a system changes per mole of component if the temperature, pressure, and number of moles of all components are

held constant. For a system in equilibrium, the chemical potential of each component must be the same in all phases.

It is difficult to directly measure the chemical potential of a system. However, the chemical potential of the water phase can be closely estimated through its water activity. It is most difficult to independently measure the chemical potential of the ions, however, their effect on water activity is well known.

By definition, the water activity (a_i) of a giving component is related to the chemical potential by the equation:

$$\mu_i = \mu_i{}^0 + RT \ln(a_i) \qquad (1)$$

In the above equation, μ_i is the chemical potential of the particular component at temperature T and pressure P; $\mu_i{}^0$ is the chemical potential of the particular component at standard condition; R is the gas constant; and T is temperature. Chenevert (1970) introduced the concept of water activity into wellbore instability study in shales [17]. A result of this work indicated that the water activity of the shale is an excellent indicator of the shale's state of hydration and its potential to absorb water. Unfortunately, this parameter cannot be measured directly. In the laboratory, it is determined by measuring the relative aqueous vapor pressure of the atmosphere above the shale using the following relationship:

$$a_{w,shale} \cong \frac{P}{P_0} \qquad (2)$$

By considering shale as a semi-permeable membrane that allows the movement of water and restricts the movement of ions, Low and Anderson (1958) presented an osmotic pressure equation for determining the swelling of soils [18]. Their theory suggests osmosis as a mechanism explaining the movement of water and ions during interactions between a shale and a drilling fluid. Using thermodynamic principles and the classical concept of an osmotic cell, Low and Anderson (1958) derived the following equation to determine the osmotic pressure that could develop between shale and the drilling fluid.

$$P_\pi = -\frac{RT}{\overline{V}_w} \ln\left(\frac{a_{w,shale}}{a_{w,mud}}\right) \qquad (3)$$

For an osmotic pressure to develop that is equal to the theoretical osmotic potential defined by the above equation, a perfect membrane restricting ion movement must exist. However, studies have shown that a shale does not act as a perfect semi-permeable membrane when contacted by the drilling fluid, so a membrane

efficiency term (α_m) is introduced to correct for the "non-ideality" [19-21]. The non-ideal osmotic pressure equation becomes;

$$P_\pi = -\alpha_m \frac{RT}{\overline{V}_w} \ln\left(\frac{a_{w,shale}}{a_{w,mud}}\right) \qquad (4)$$

In the above equation, P_π is osmotic pressure; α_m is membrane efficiency; $\overline{V}_w$ is molar volume of water; $a_{w,shale}$ is water activity of shale; $a_{w,mud}$ is water activity of mud.

As shown in Figure 1, the following cases can be highlighted from Equation (4):

1) $a_{w,shale} < a_{w,mud}$, chemical osmosis flow of water into the shale increases the water content and the pore pressure near the wellbore wall;

2) $a_{w,shale} = a_{w,mud}$, no aqueous chemical osmosis flow; and

3) $a_{w,shale} > a_{w,mud}$, chemical osmosis flow out of the shale reduces the near wellbore pore pressure and water content.

Diffusive Flow

Although the movement of ions through shale is restricted due to the narrow pore throats, cation exchange capacity and negatively charged clay surfaces, shale does not act as a perfect semi-permeable membrane. The diffusion of ions and the associated water is dominated by a concentration gradient that can be expressed by using Fick's law [22]:

$$J = -D_{si}\left(\frac{C_{i,shale} - C_{i,mud}}{\Delta x}\right) \qquad (5)$$

where J is mass flux of ith ion; $C_{i,shale}$ concentration of ith ion in pore fluid; $C_{i,mud}$ concentration of ith ion in mud; D_{si} the diffusion coefficient of the ith ion; and Δx length of shale. Similarly, the following cases can be highlighted from Equation (5):

1) $C_{i,shale} < C_{i,mud}$, diffusive flow of hydrated ions into shale.

2) $C_{i,shale} = C_{i,mud}$, no diffusive flow; and

3) $C_{i,shale} > C_{i,mud}$, diffusive flow of hydrated ions out of shale.

Capillary Suction

When a wetting phase, such as pore water comes into contact with a non-wetting phase, such as air, surface tension forces, σ_{w-a} develop at their interfaces [23]. These surface tension forces give rise to capillary pressures, which can be expressed as:

$$\Delta P = P_c = P_a - P_w = \frac{2\sigma_{w-a}\cos\theta}{r} \quad (6)$$

In classical capillary pressure diagrams, such as the one shown in Figure 2, the rise of liquid level from the dish into the capillary tube is governed by Equation (6). These surface tension forces result in the reduction of the vapor pressure of the water just above the meniscus (P_m) relative to the vapor pressure of the water in the solution (P_s). This indicates there is an aqueous activity imbalance that governs the capillary rise, i.e.,

$$a_{wm} < a_{ws} \text{ where } a_{wm} \cong \frac{P_m}{P_0} \text{ and } a_{ws} \cong \frac{P_s}{P_0}.$$

There has been much discussion regarding the mechanisms whereby water is driven into shale. One camp has postulated that it is driven by the present of clays and ions that results in a reduced water activity environment [17, 24]. Another camp insists that such surface forces could not exist, and the water flow is driven by capillary suction [25]. It is very difficult, if not currently impossible, to settle this conflict from direct experimental observations because of the complexity of shale composition, and the submicroscopic site of such clay surfaces and capillarity variance. The key to both theories is the water activity of the shale and the drilling fluid. The intensity of water moved by either mechanism is reflected the water activity difference. Thus, the measurement of the net water activity of shale reflects the intensity of the driving forces regardless of being caused by clay surfaces, ions, or capillaries.

Convective Flow

Convective flow is governed by the Darcy equation [22]:

$$v_0 = -\frac{k}{\mu}(\nabla p - \rho g) \quad (7)$$

In the above equation, ∇p is the pressure gradient between the hydraulic fluid pressure and the pore pressure. This flow is quite slow as the result of a low-pressure gradient (∇p) that typically exists for most drilling operations and the extremely low permeability (k) of shale. This well-known mechanism for water and ions movement will not be discussed in this paper.

Results and Discussions

Pierre I and Arco shales were used in this study. Pierre I was an out-crop shale, and Arco was cored in a well located in Northern USA at about 15,600ft. The mineralogical composition, water content and water activity are shown in Table 1.

The first series of experiments was performed in order to study the mechanisms controlling the movement of water and ions when the shale samples interact with various ionic solutions.

Desiccator-Immersion-Desiccator Test

Experimental steps were designed to study the effects of chemical osmosis, capillary suction, and diffusive flow on the movement of water and ions into or out of shale. The steps for the study were:

Step 1: Three Pierre I and three Arco shale samples with dimension of 0.5" x 0.75" x 1.0" were placed in a controlled relative humidity environment of 85% (a_w = 0.85) desiccator, weighed and recorded as $W_{0.85-1}$ until equilibrium was achieved.

Step 2: The above "conditioned" samples were then immersed into 0.85 a_w KCl, 0.85 a_w NaCl, and 0.85 a_w CaCl$_2$ solutions separately for 24 hours, and then removed, weighed, and their altered weights, W_a, was recorded.

Step 3: These samples were then placed back into the 85% controlled relative humidity desiccator until equilibrium was reached. Their weight, $W_{0.85-2}$, was recorded.

Step 4: The final step consisted of drying them out, by placing them in a 200ºF oven for 24 hours. Their "dried" weight, W_{ad}, was recorded.

a) Result for Pierre I shale/NaCl solution test

In order to determine the water and ions weight change during each step of the process, it was necessary to determine the initial water content of the Pierre I and Arco shale samples. This was done by drying three separate native pieces of Pierre I shale in a 200ºF oven overnight. The average water content of these three samples was considered as the native water content. The native weight is referred as N_a and dried weight as N_d.

Using these measurements, it was possible to obtain the change of weight, relative to dry weight, of the Pierre I when it interacted with 0.85 a_w NaCl solution during the above four steps. The results are shown in Figure 3.

In Figure 3, "N_a" represents the native state of a Pierre I sample. After this native sample was dried, it lost 10.2 wt% of water and changed into the dried state. This dried weight was selected as the base weight, so its weight change is zero, shown as "N_d". Following the procedure in "Step 1", the native Pierre I shale

equilibrated in the 0.85 a_w atmosphere, lost 4.1 wt% water. Its weight change was 6.1 wt% compared to the dried weight. In this state, equilibrium was reached by means of "gaseous osmosis". Under such conditions, the Pierre I sample was not brought into contact with liquid water, so water molecules moved by means of the gaseous third phase. Equilibrium was established between the shale and the solution through their vapor pressure. Since there was no ion movement, the atmosphere can be taken as a perfect semi-permeable membrane, and water movement is caused by chemical osmosis. This 4.1 wt% lost water was the result of the water activity difference between the 0.98 a_w Pierre shale and the conditioned 0.85 a_w atmosphere.

After "Step 1", the Pierre I sample was immersed into a 0.85 a_w NaCl solution as part of "Step 2". The weight increased by 11.8 wt% as shown in Figure 3. This weight increase was a result of water and ions movement, primarily a consequence of ion and water diffusion.

After "Step 2", water was removed from the sample in two steps; first it was placed in the 0.85 a_w atmosphere as a part of "Step 3", and then it was dried out in an oven to complete "Step 4". In "Step 3" there was a larger weight change compared to "Step 1", even though the sample was exposed to a 0.85 a_w atmosphere in Step 1 and to a 0.85 a_w NaCl solution in "Step 2". *From this comparison, it can be concluded that hydrated ions diffused into the shale.*

By drying the shale in Step 4, it was observed that ions have definitely entered the shale, its weight has increased by 0.64 wt% as compared to the dried native weight, "N_d".

b)   Results for Pierre I shale/ KCl and CaCl$_2$ tests

The test results for Pierre I shale with 0.85 a_w solutions of KCl and CaCl$_2$ are shown in Figures 4 and 5 respectively.

Comparing the amount of lost water in "Step 1" among the three Pierre I shale samples indicates they lost approximately the same amount of water (4 wt%) after they were equilibrated in the 0.85 a_w atmosphere. This demonstrates that the native water contents of these three samples are virtually identical.

Review of the amount of lost water in "Step 3" among the three Pierre samples, shows the amount of lost water for the CaCl$_2$ solution is more than that for the NaCl and KCl solutions. This can be explained by the diameter difference between the dehydrated and hydrated ions, as shown in Table 2. The diameter of the hydrated calcium ion is ten times larger than the dehydrated ion. Therefore, the associated water carried by these ions is easily lost.

Comparing "N_d" with "Step 4" in Figures 3 - 5, the Pierre I shale gained ions when immersed in ionic solutions. This ionic gain raises a question: "is such ionic transfer sufficient to significantly alter the chemical properties of the shale?" To answer this question, adsorption isotherms were developed for the immersed shale sample in Step 2, and compared to the adsorption isotherm for native Pierre I shale. The results are shown in Figure 6.

As indicated in Figure 6, the amount of water adsorbed was highest for the samples immersed in 0.85 a_w CaCl$_2$ solution and least for the samples immersed in 0.85 a_w KCl solution. This result can be used to demonstrate that the adsorption of ions has changed the chemical properties of the shale. The difference in behavior between the samples immersed in CaCl$_2$ and KCl solutions was probably due to the fact that the adsorption of K^+ ions led to the collapse of the spacing between the clay platelets, thus reducing the amount of water adsorbed [26].

c)   Results for Arco shale/ NaCl, KCl and CaCl$_2$ tests

Tests similar to the above Pierre I tests were carried out for the Arco shale samples. The results are shown in Figures 7 - 9. The three Arco shale samples gained water after they were equilibrated in the 0.85 a_w atmosphere. As discussed previously, this amount of water gain is caused by the chemical potential difference between the 0.78 a_w Arco shale and the 0.85 a_w atmosphere.

Comparing the amount of lost water in "Step 3" among the three Arco shale samples, it was again observed that the lost water for the CaCl$_2$ solution test is greater than that for NaCl and KCl solutions. Once again, this can be explained by the difference in diameter between the dehydrated and hydrated ions.

Comparing "N_d" with "Step 4" in Figures 7 - 9, we see that the Arco shale samples also gained ions when they were immersed into ionic solutions. This amount of ion adsorption has altered the chemical properties of the shale. Due to a lack of samples, it was not possible to develop adsorption isotherms for the Arco shale, as was done for the Pierre I shale.

Test of Direct Exposure to 0.85 a_w Solutions

A second series of experiments were performed in order to study the movement of water and ions for native Pierre I shale interacting with different ionic solutions of identical water activity.

The native 0.98 a_w Pierre I shale samples were directly immersed into 0.85 a_w solutions of CaCl$_2$, NaCl and KCl *without* removing water in a 85% relative humidity desiccator, as was done in the first series of experiments. The movement of water and ions during the exposure of native Pierre I shale to such salt solutions were determined by using the gravimetric method as discussed in the paper by Zhang *et al.* (2004) [7]. The results of such movement are shown in Figure 10.

In these tests, capillary, osmotic, and diffusive processes are acting simultaneously. If the movement of water and ions was the result of capillary effects alone, as suggested by Santarelli *et al.* (1995) [25], the shale should absorb the whole solution fluid (both water and ions) simultaneously. However, it is seen from Figure 10 that these native Pierre I shale samples lost water after immersion into 0.85 a_w solutions of $CaCl_2$, $NaCl$ and KCl. This demonstrates that osmosis was involved in the water and ion movement.

For the native Arco shale directly exposed to 0.85 a_w $NaCl$ and $CaCl_2$ solutions, the results of water and ion movement are shown in Figure 11. It was observed that the Arco shale samples absorbed 1.16 wt % water and 0.7 wt% ions for the $CaCl_2$ solution, and 0.9 wt% water and 0.3 wt% ions for the $NaCl$ solution. Such movements are reasonable because of the large imbalance of water activity and ion concentration between the 0.78 a_w Arco shale and the 0.85 a_w salt solutions. As discussed previously, this amount of water and ions movement is probably driven by capillarity, diffusion, and chemical osmosis.

The above tests were repeated using $CaCl_2$, $NaCl$, and KCl solutions of other water activities, specifically 0.75, 0.95, and 1.0. The results are presented in Figure 12. Results were as generally expected and the amount of lost water increased at the lower water activities. It is also shown in Figure 12 that there exist differences among these three types of ionic solutions. These differences in water movement demonstrated that in addition to osmosis which is driven by water activity, the movement of water and ions is influenced by ionic diffusion.

Effects of Water and Ion Movement on Swelling Property of Shales

From the above discussion, it is seen that water and ions moved into or out of the shale when interacted with ionic solutions. This movement alters the physico-chemical and mechanical properties of the formation. In order to investigate the effects of water and ions movement on swelling properties of Pierre I shale, a third series of experiments were performed. The results for these experiments are discussed below.

a) Effects of Water Movement on Swelling Properties of Pierre I Shale

This test was developed to investigate the effects of water movement on swelling property of Pierre I shale. A preserved Pierre I shale sample with dimensions $0.5'' \times 0.75'' \times 1.0''$ was taken out of the storage can and quickly washed using "Skelly B" to remove all surface oil. It was then positioned between the movable and stationary anvils in a swelling transducer and then placed in a controlled 85% humidity environment and its swelling behavior measured. As shown in Figure 13, the shale shrank continuously due to the loss of water only.

b) Effects of Water and Ions Movement on Swelling Properties of Pierre I Shale

Additional swelling tests were performed differently from the above test in that the sample was immersed directly into 0.85 a_w KCl solution. The procedure for this experiment consisted of: 1) washing the sample with "Skelly B"; 2) placing it in a small plastic bag and positioning it in the swelling transducer; and 3) pouring 50 ml 0.85 a_w KCl solution into the bag and measuring the swelling properties. The result is shown in Figure 14.

It is shown in Figure 14 that the sample swelled initially and later began to shrink. As mentioned previously, the change of swelling behavior with time was most likely caused by the water and ions movement. This swelling behavior of Pierre I shale in Figure 14 can be classified into four stages, A, B, C, and D. In stage A, about 30 minutes, the shale swelled quickly. This early swelling is mainly caused by a surface capillary effect as described by Zhang *et al* [7]. After this early capillary swelling, the Pierre I shale began to shrink in stage B. This shale shrinkage was mainly caused by the water desorption due to the chemical potential difference between 0.98 a_w Pierre I shale and 0.85 a_w KCl solution. Although the ionic diffusion was also acting in this stage, its velocity is much less than that for the water movement. Therefore, the chemical osmosis is in dominant in stage B. After stage B, the shale still continues to reduce in size, but the shrinkage rate is lower than that in stage B. This is because in stage C, the ionic diffusion is dominant. Finally, equilibrium between ionic diffusion and chemical osmosis was obtained in stage D.

After correcting Figure 14 for capillary effects, the comparison of the swelling properties of Pierre I shale placed in a 0. 85 a_w atmosphere and immersed into a 0.85 a_w KCl solution is shown in Figure 15.

It is seen that the sample shrank after it was exposed to the 0.85 a_w KCl solution. The sample experienced more shrinkage when it was placed in a 0.85 a_w atmosphere than when immersed into the 0.85 a_w KCl solution. It is postulated that, for the immersion test the movement of ions into the shale lowering the water activity of the shale, thus reducing the osmotic driving force, and swelling percentage.

Conclusions

1. The new simple method, called the Gravimetric–Swelling Test, that measures water and ion entering or leaving a shale sample provides a good way to study water and ions movement and the effects of such

movement on the swelling properties of shales.

2. After the native Pierre I shale samples were immersed in 0.85 a_w solutions, they lost water and gained ions. However, they gained both water and ions if the Pierre I shale samples were equilibrated in a controlled relative humidity environment of 85% before immersed in 0.85 a_w solutions.

3. Both the native and the conditioned Arco shale gained water and ion after they were exposed to 0.85 a_w solutions.

4. The transport of water is not only controlled by chemical osmosis, but also affected by ionic diffusion and capillary effects under zero hydraulic pressure difference.

5. The swelling of the shale is influenced by both water and ion movement.

6. Combined with other tests, the Gravimetric–Swelling Test can be used to evaluate mud systems for wellbore instability mitigation.

Nomenclatures*

a_i = Water activity of ith component, dimensionless

$a_{w,mud}$ = Water activity of mud, dimensionless

$a_{w,shale}$ = Water activity of shale, dimensionless

$C_{i,mud}$ = Concentration of ith ion in mud [=] amount/volume

$C_{i,shale}$ = Concentration of ith ion in pore fluid [=] amount/volume

D_{si} = Diffusion coefficient of the ith ion in the shale [=] L^2/t

g = Gravitation acceleration [=] L/t^2

J = Mass flux [=] mol/L^2-t

k = Permeability [=] L^2

P = Vapor pressure of shale at temperature T [=] m/L-t^2

P_a = Pressure of the air phase [=] m/L-t^2

P_c = Capillary pressure between water and gas phases [=] m/L-t^2

P_m = Vapor pressure of water above the meniscus [=] m/L-t^2

P_0 = Vapor pressure of pure water at temperature T [=] m/L-t^2

P_s = Vapor pressure of water above the solution [=] m/L-t^2

P_π = Osmotic pressure [=] m/L-t^2

∇p = Pressure gradient between the mud hydraulic pressure and pore pressure [=] m/L^2-t^2

ΔP = Pressure difference between air and water phases [=] m/L-t^2

r = Tube radius [=] L

R = Gas law constant, $8.314 \times 10^3 \, kg \cdot m^2 / S^2 \cdot Kg-mole \, K$

T = Absolute temperature [=] T

u_i = Chemical potential of ith component

u_i^0 = Chemical potential of ith component under standard condition

v_0 = Bulk flow velocity [=] L/t

$\overline{V}_w$ = Molar volume of water, 0.018 m^3/mol

W_n = Native weight [=] m

W_{nd} = Native dried weight [=] m

ρ = Density of drilling fluid [=] m/L^3

σ_{w-a} = Interfacial tension [=] m/t^2

θ = Contact angle between the fluids interface and the solid surface [=] degree

α_m = Membrane efficiency, dimensionless

μ = Viscosity of drilling fluid [=] m/L-t

* [=] means has unit of, L, m, t and T are length, mass, time and temperature respectively.

Acknowledgements

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Table 1 - Mineralogical composition, water content, and water activity of Pierre I and Arco shales.

Constituent		Pierre I (% by weight)	Arco (% by weight)
Quartz		19	23.6
Feldspar		4.0	4.0
Calcite		3	-----
Dolomite		7	1.2
Pyrite		2	2.4
Siderite		1	4.1
Clay	Chlorite	4	3.6
	Kaolinite	11	5.7
	Illite	19.0	15.0
	Smectite	17	11.0
	Mixed layer	49	29.4
	Total	64	64.7
Water Content		10.2	3.2
Water activity		0.98	0.78

Table 2 - Comparison of the radii of dehydrated and hydrated ions.

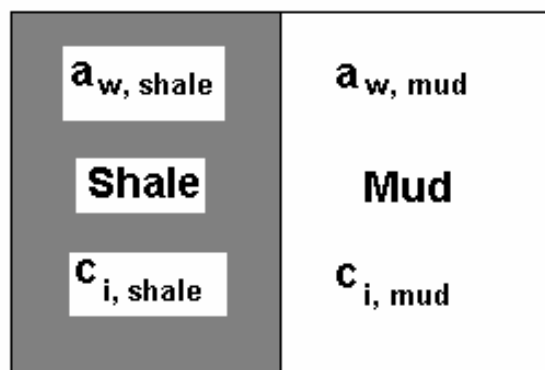|	Dehydrated Diameter (Angstrom)	Hydrated Diameter (Angstrom)
Sodium	1.9	5.5-11.2
Potassium	2.66	4.64-7.6
Cesium	3.34	4.6-7.2
Magnesium	1.3	21.6
Calcium	1.9	19

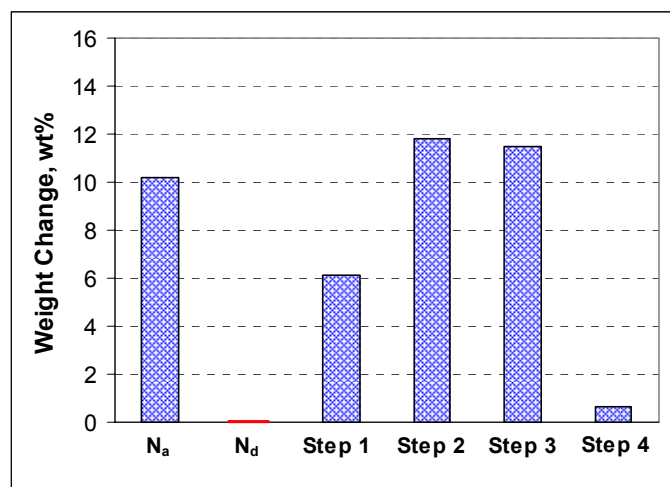**Figure 1 – Chemical osmosis and ion diffusion.**

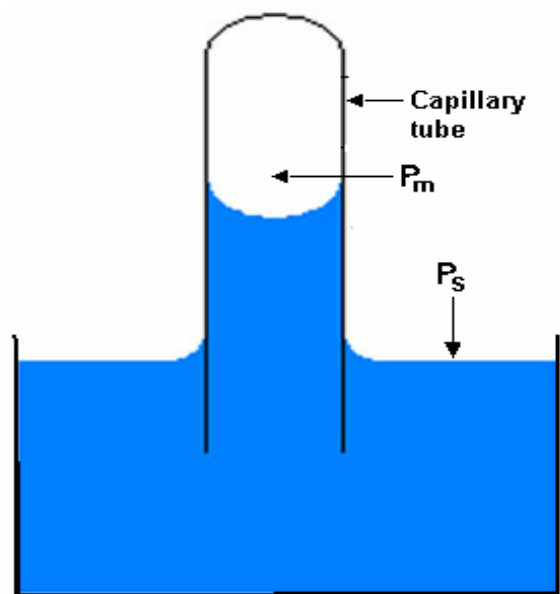**Figure 3 – Weight changes of Pierre I shale during desiccator-immersion-desiccator test (NaCl).**

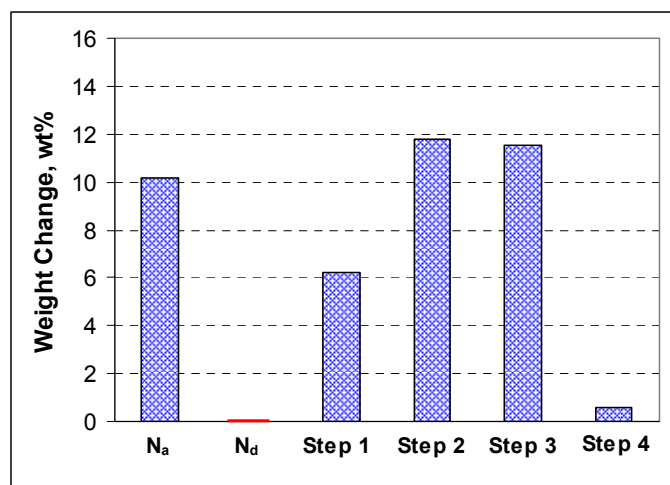

Figure 2 – Brine water capillary imbibitions.

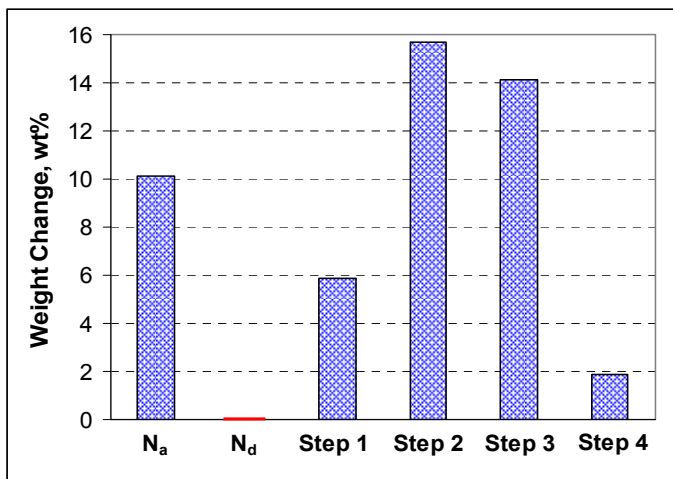**Figure 4 – Weight changes of Pierre I shale during desiccator-immersion-desiccator test (KCl).**

Figure 5 – Weight changes of Pierre I shale during desiccator-immersion-desiccator test (CaCl$_2$).
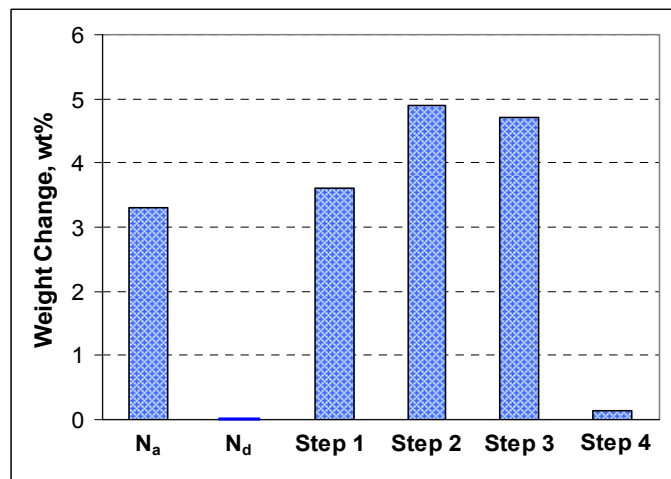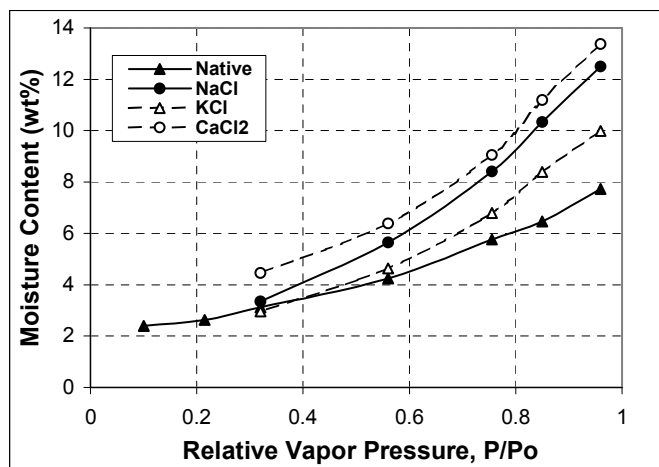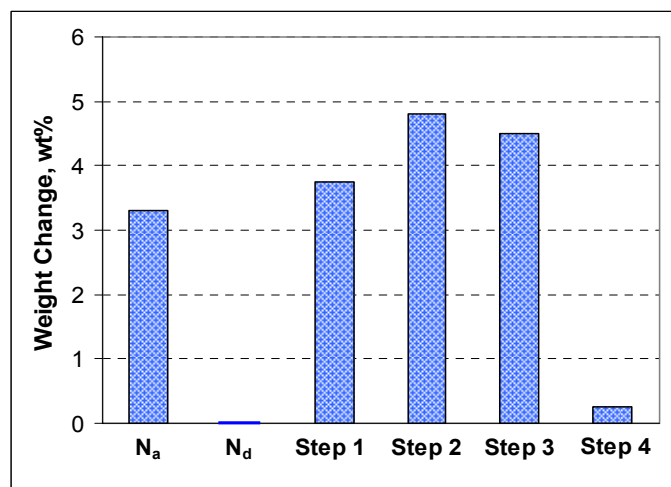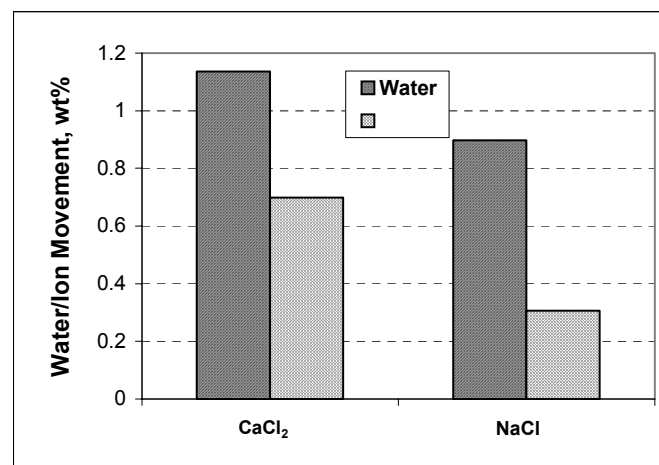

Figure 7 – Weight changes of Arco shale during desiccator-immersion-desiccator test (NaCl).

Figure 6 – Combined adsorption isotherms of Pierre I shale.

Figure 8 – Weight changes of Arco shale during desiccator-immersion-desiccator test (KCl).
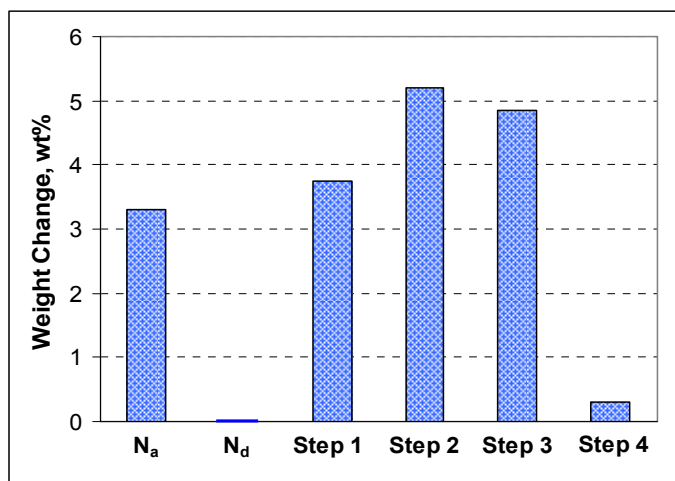
Figure 9 – Weight changes of Arco shale during desiccator-immersion-desiccator test (CaCl$_2$).

Figure 11 – Water and ions movement after Arco shale was directly immersed in 0.85 a$_w$ NaCl and 0.85 a$_w$ CaCl$_2$ solutions.
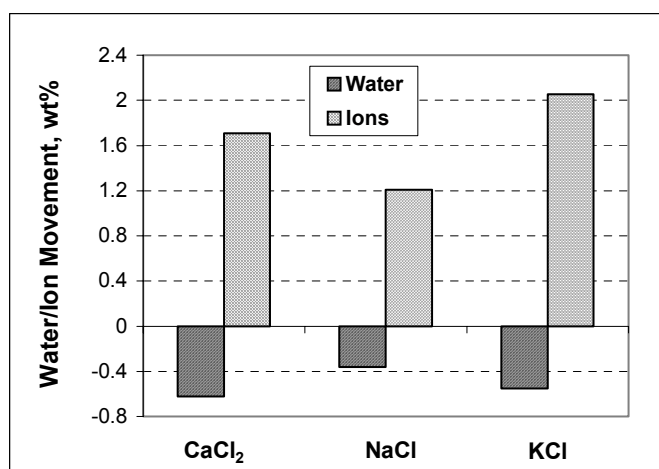
Figure 10 – Water and ions movement after Pierre I shale was directly immersed in 0.85 a$_w$ CaCl$_2$, 0.85 a$_w$ NaCl, and 0.85 a$_w$ KCl solutions.
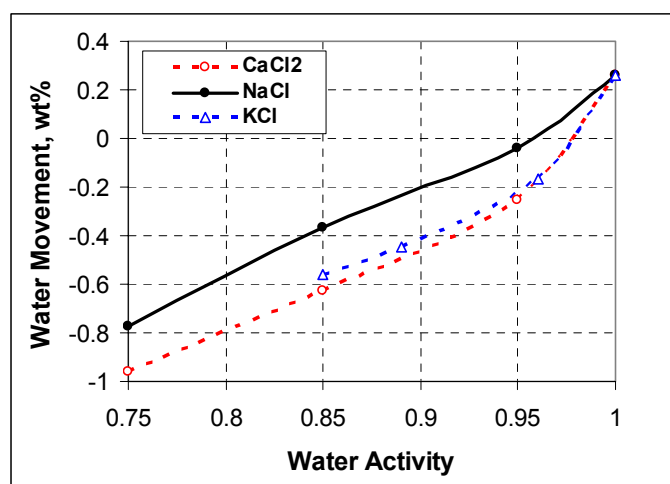

Figure 12 – Effects of water activity on water movement of Pierre I shale.
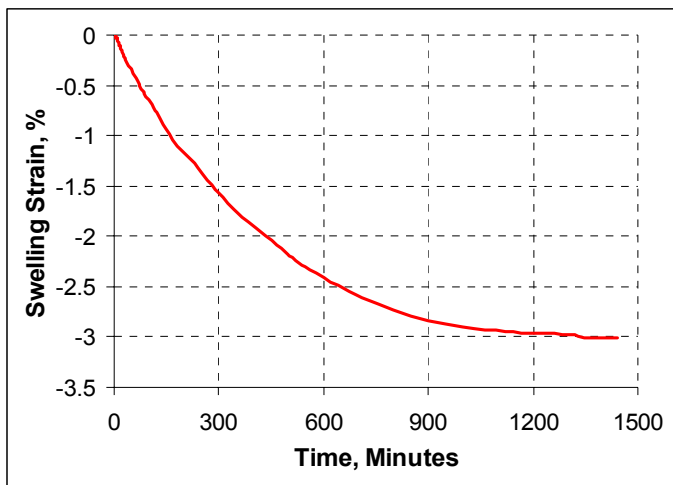
Figure 13 – Swelling of Pierre I shale placed in a controlled 85% humidity desiccator.
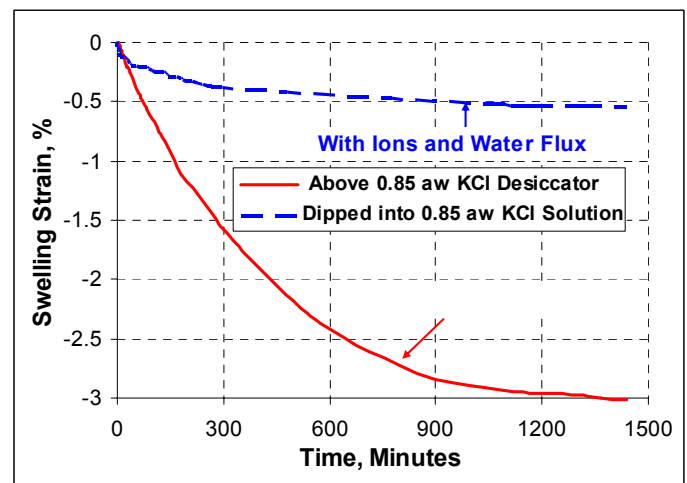

Figure 15 – Comparison of shale swelling when sample is immersed in a 0.85 a_w KCl solution versus placed in a 0.85 a_w atmosphere (Pierre I shale).
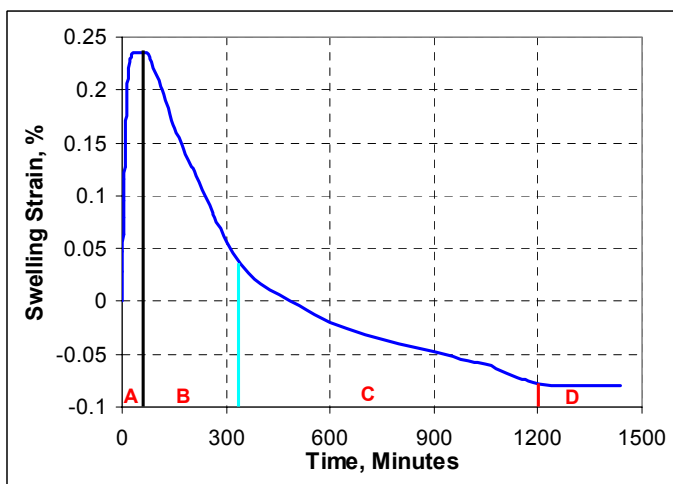

Figure 14 – Swelling properties of Pierre I shale immersed in a 0.85 a_w KCl solution.