

Full Length Research Paper

Variation of petrophysical properties due to carbon dioxide (CO₂) storage in carbonate reservoirs

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Accepted 2 April, 2013

Depleted hydrocarbon reservoirs, deep saline reservoirs, and un-mineable coal seams are considered the best geological sequestration candidates for carbon dioxide (CO₂) geologic storage formations. CO₂ sequestration in carbonate reservoirs provides a good way to reduce CO₂ release to the atmosphere. This work investigates the effect of the temperature, pressure, and brine salinity on the petrophysical changes in the carbonate cores due to CO₂ storing. Two groups of experiments were undertaken; (1) investigating the CO₂ solubility under different salinities, pressures and temperatures, and (2) studying the effect of CO₂ storage duration on porosity and permeability of carbonate rocks. Actual cores saturated with 25 000 ppm NaCl brine were used. The potential of the CO₂ storage capacity and variations in porosity and permeability are evaluated and quantified. The results showed that solubility of CO₂ decreases with increase in brine salinity and/or temperature. The increase of pressure causes an increase in CO₂ solubility. The results also indicated that storing CO₂ more than 150 days increases the porosity and permeability of carbonate rocks. The application of the achieved results is expected to have good impact on design storage process of CO₂ in deep saline water incarbonate reservoirs, and on validation of developed mathematical models.

Key words: Carbon dioxide (CO₂) storage, sequestration, carbonate formation, petrophysical properties, deep saline reservoirs.

INTRODUCTION

Carbon dioxide is used in many industries such as metal industry to enhance their hardness; manufacturing and construction such as in metal inert gas / metal active gas (MIG/MAG) welding for protection and increase of the welding rate; rubber and plastic industry to remove flash with crushed dry ice in a rotating drum; Food and beverages as quick freezing, surface freezing, chilling and refrigeration in the transport of foods; Health as an additive to oxygen for medical uses as a respiration stimulant; Environment as a propellant in aerosol cans; Chemicals, Pharmaceuticals and Petroleum Industry such as CO₂ injection as tertiary recovery to increase the recovery from the reservoirs.

Geologic sequestration of carbon dioxide in aquifers or in hydrocarbon reservoirs offer a promising alternative to reduce the amount of CO₂ released to the atmosphere (McCabe et al., 2007). Among the emerging technologies for large-scale reduction of CO₂ emissions, one of the most promising is the carbon dioxide capture and storage (CCS) in deep geological formations. Geological storage of CO₂ can be undertaken in a variety of forms, including its injection in depleted oil and gas fields and its use to enhance recovery from producing wells (D'Alesio et al., 2011).

Nguyen (2003) summarized four options for the CO₂ geological sequestration as follows; (1) the CO₂ can be

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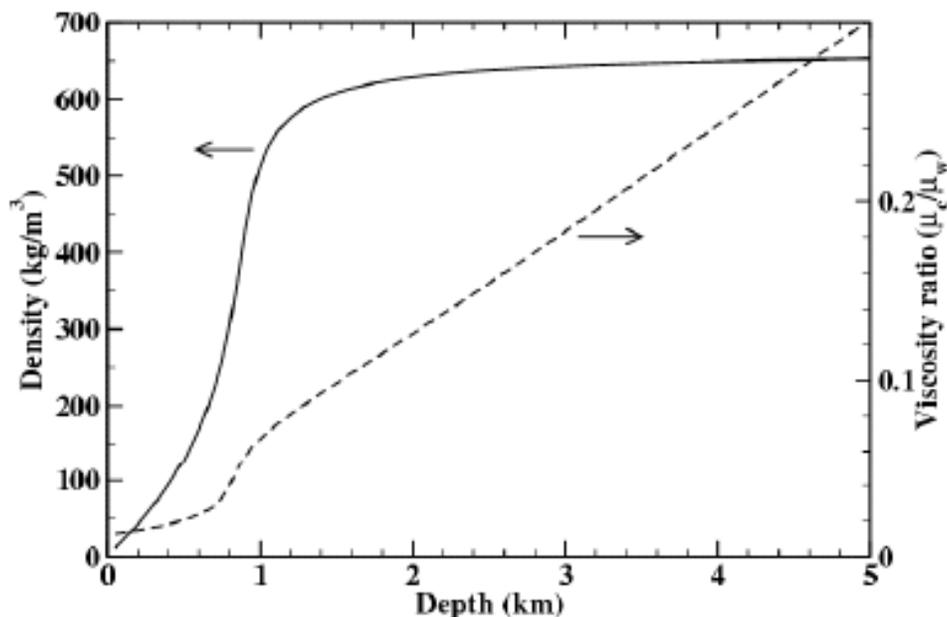


Figure 1. CO₂ density and viscosity at subsurface conditions, surface temperature 15°C/km and 10 MPa/km (Ennis-King and Paterson, 2002).

used in enhanced oil recovery (EOR) processes, (2) the CO₂ can be used to enhance coal bed methane recovery, (3) the CO₂ can be stored in depleted oil and gas reservoirs, and (4) the CO₂ can be stored in deep saline aquifers. Aquifers are considered to be the most widely available.

Physical properties of carbon dioxide (CO₂)

Based on pressure and temperature, CO₂ may exist in three phases under its critical point: solid, liquid and/or gas with a triple point at -56.6°C and 75.1 psi. CO₂ is in supercritical phase at temperature values greater than 31.04°C and pressure values above 1071 psi. Supercritical CO₂ performs like a condensed gas having low viscosity and high density. In order to avoid separation of CO₂ into gas and liquid phases during injection process, disposal of CO₂ is made at supercritical conditions. A minimum depth of 2625 ft (800 m) is required to reach these conditions (Dirik et al., 2004).

Using Peng-Robinson equation of state, the compressibility factors of CO₂ have been calculated by a computer program for different temperature and pressure values. Also, another equation of state for CO₂ has been proposed as a recent research by Span and Wagner (1996) which can be used as an alternative for the calculations of compressibility factor of CO₂.

Ennis-King and Paterson (2002) presented the density and the viscosity ratio of pure CO₂ to typical formation brine (32000 ppm) as a function of depth in Figure 1.

This ratio increases with depth, but in the depth range of 1000 to 3000 m, it varies from 0.05 to 0.20. This figure is developed based on a geothermal gradient of 30°C/km, and at mean surface temperature of 15°C and a hydrostatic pressure gradient of 10 MPa/km. Therefore the CO₂ is more mobile than in the formation than the brine. The important conclusion of this figure is that the density of the gas increases rapidly down with the depth of about 1000 m, then levels out around 600 to 700 kg/m³. This provision of 30 to 40% less dense than typical formation brine and the injected CO₂ will rise and accumulates below the top seal of the formation. In homogeneous formation, this may lead to viscous fingering in the displacement front. The effect of reservoir heterogeneity on the flow of CO₂ was investigated by Chang et al. (1994). The study used a compositional simulator to study the CO₂ flooding under unstable first-contact miscible flow with stochastic permeability fields. The study indicated that the permeability heterogeneity and gravity override - dominate the displacement pattern of the CO₂ flow in underground formation.

Carbon dioxide (CO₂) geological sequestration

The major criteria for the CO₂ storage in the depleted oil and gas reservoirs are the capacity, injectivity, lithology, and cap rock integrity (Taber et al., 1997; Shaw and Bachu, 2002; Cinar et al., 2007). The main problems of the CO₂ sequestration are the overriding of the gas to the top of the formation, the reduction of the CO₂ injectivity

over time, and the early breakthrough of the CO₂ during injection due to the heterogeneity of the formation.

Le Gallo et al. (2002) summarized the advantages of the CO₂ storage as (1) available large pressure range for injection, (2) allowing the storage of significant gas volume for a low compression power, and (3) available data of dynamic and geologic characterization of depleted reservoirs. The appropriate selection of a suitable reservoir is crucial to the success of the CO₂ sequestration. Le Gallo et al. (2002) and Izgec et al. (2005) identified three major steps (mechanisms) to achieve the CO₂ storage as follows; (1) Hydrodynamic Trapping; in which the CO₂ is injected in the host formation and flows according to the pressure gradient, (2) Solution Trapping: It is the process in which the CO₂ reaches the thermodynamic equilibrium in all available phases; and (3) Mineral Trapping: it is the kinetic (or geochemical) reaction of the CO₂ with the rock and fluids of the aquifer. These mechanisms lead to storage of the CO₂ as a free-phase gas in pore space, dissolved phase CO₂ in formation water, and CO₂-converted to rock matrix. Kaldi and Bachu (2009) summarized the basic requirements for any carbon dioxide sequestration site to be as follows; (A) sufficient economic capacity to store targeted CO₂ quantity, (B) suitable injectivity to accept CO₂ at the rate at which it is being generated by the target source, and (C) containment assurance for no leakage from the site in long-term.

Phase properties of CO₂-brine systems

There are two major steps for the CO₂ underground sequestration including (1) the dissolution and diffusion of the CO₂ in oil and brine and following the pressure gradient (hydrodynamic trapping), and (2) the injected CO₂ reacts with the formation and induces dissolution/precipitation reactions (mineral trapping), that may impair the well injectivity and/or rock properties.

Natural gas storage in saline formation has been practiced in North America, Europe, and Australia (Coats and Richardson, 1967; Hower et al., 1993; Chang et al., 1994, 1998; Ennis-King and Paterson, 2002; Nghiem et al., 2009). However, a little attention has been paid to the mineral trapping of the CO₂ storage in carbonate formation saturated with brine. Storage of the CO₂ into carbonate formation was investigated by Izgec et al. (2005). The study evaluated the potential of a carbonate aquifer in the south-east of Turkey. The results indicated that permeability was initially increased and decreased later under slower injection rate because of precipitation of calcium carbonate (CaCO₃). Porosity of the aquifer was also decreased. The study also showed that orientation of core plugs was influential in rock-fluid-CO₂ interactions. Gupta (2010) presented the results of experimental and simulation work to understand opportunities and challenges in sequestering CO₂ in

carbonate rocks. The results indicated that petrophysical properties of core samples had dissolution in some pores but precipitation in other pores of carbonate matrix.

Ülker et al. (2007) proved that the solubility of CO₂ in brine is much higher than that of hydrocarbon components. The CO₂ solubility depends essentially on pressure, temperature, total salinity and brine composition. In general, CO₂ solubility increases with increasing pressure and decreases with increasing temperature. An increase in the salinity of the formation brine decreases the CO₂ solubility significantly.

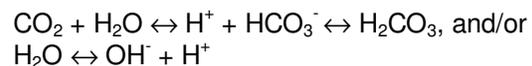
Reid et al. (2003) investigated the viability of, and optimal locations for sequestering CO₂ in the subsurface require detailed knowledge of the complex interactions among CO₂, rock matrix, and pore fluids under appropriate *in-situ* pressure and temperature conditions.

Many physical and chemical processes are known to occur both during and after geologic CO₂ injection, including diagenetic chemical reactions and associated permeability changes. Although it is commonly assumed that CO₂ sequestered in this way will ultimately become mineralized, the rates of these changes, including CO₂ hydration in brines, are known to be relatively slow. Together with hydrated CO₂, cations from brines may form solid-state carbonate minerals, ostensibly providing permanent sequestration (Reid et al., 2003).

Geochemical reaction of CO₂ and carbonate rocks

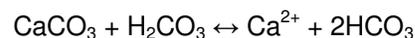
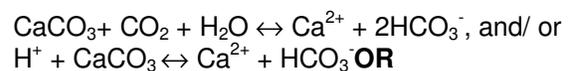
In generally, the predominant minerals of carbonate rocks are calcite (CaCO₃) and dolomite [CaMg(CO₃)₂], which are highly susceptible to dissolution, especially by carbonic acid (H₂CO₃). Dissolution processes are important to understand, as they may significantly alter the porosity and permeability of the rock. The following are the possible chemical reaction that might happen if CO₂ contact the carbonate rocks:

First the CO₂ dissolution into the formation water produces a slightly acidic solution as follow:

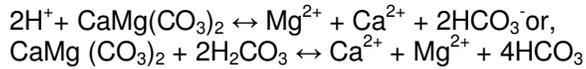


As a result of the decreased pH of the reservoir brine, dissolution of calcite and dolomite along with increased alkalinity (HCO₃⁻) occur as represented by the following equations:

Reaction with calcite formation:



Reaction with limestone formation:



The reaction rate constant of calcite (1×10^{-5} mol./cm²/s) is larger than the reaction rate constant of dolomite (1×10^{-7} mol/cm²/s) (Wellman et al., 2003). Therefore, when a formation has a combination of calcite and dolomite, carbonic acid will tend to react with calcite (Mohamed et al., 2011).

Although dissolution can enhance effective porosity and permeability, precipitation of carbonate minerals can significantly reduce those characteristics. Carbon dioxide precipitation in solid form is also important because it represents a relatively secure sequestration mechanism. As such, understanding the conditions in which dissolution and precipitation will occur is important for predicting the evolution of porosity and permeability (and resultant increases or decreases in injectivity), and long-term storage mechanisms.

Moh et al. (2010) mentioned that CO₂ dissolves in the formation brine, generating carbonic acid, which dissolves carbonate rock. Dissolution impacts brine composition, which affects solubility. Calcium carbonate may tend to precipitate with changing concentration of bicarbonates. Precipitation may occur in either EOR operations or during primary CO₂ sequestration. Injectivity changes are a concern during EOR operations, while storage capacity and seal integrity are primary concerns during CO₂ sequestration. From their study, they concluded first that the changes in porosity and permeability were independent of each other, secondly, pressure lower than critical CO₂ pressure resulted in an increase in the core porosity and permeability, thirdly, permeability increased as CO₂: water volumetric ratio was increased.

Vanorio et al. (2010) found that, the injection of CO₂-rich brine into carbonates induces dissolution of the microcrystalline matrix (that is, micrite) leading to porosity enhancement.

Alam et al. (2011) studied the effect of CO₂ sequestration in Ekofisk Formation and Tor Formation chalk (*a form of limestone composed of the mineral calcite*) of the South Arne field formation, North Sea. They observed that, an increase in porosity and decrease in specific surface is due to injection of supercritical CO₂. This indicates that a reaction between CO₂ enriched water and particles takes place which smoothens the particle surface. Accordingly, partial increase in permeability was also noticed. A porosity increase of 1 to 2% was observed in Ekofisk Formation samples. In Tor Formation samples, the increase was typically 2 to 3%. Specific surface measured by nitrogen adsorption technique shows a decrease after CO₂ injection in Tor Formation samples. In Ekofisk Formation any change in specific surface is not evident. A decrease in specific surface of pores can be due to an increase in porosity. The trend of change in specific surface is in

accordance with the change in porosity. It indicates that supercritical CO₂ injection smoothens the grain surface, which leads to increase in porosity and permeability.

Taberner et al. (2009) presented a simulated study of supercritical CO₂ injection into a deep saline aquifer from a carbonate formation (calcite and dolomite, with minor anhydrite). They found that the near well-bore porosity reduces by 5 to 17% due to halite precipitation (dryout zone); calcite and dolomite dissolve as the CO₂ plume advances during injection (Zhang et al., 2009).

Ricardo (2012) investigated the effect of CO₂ injection and storage in carbonate rocks, and they investigated experimentally the interactions between CO₂, water and carbonate rock. Their experiments involved saturating dolomite and limestone samples by carbonated water under pressures up to 10 000 psi in a high-pressure vessel for 24 and 48 h. Then they measured the porosity, permeability and mass of the rock. They concluded that the porosity and permeability changes produced improvements or damages on the flow properties, which is related to the fine grains produced during the dissolution process leading to the partial blockage of porous media. Mass reduction was up to 0.95 and 0.35%, respectively for limestone and dolomite. Permeability varied +/-60% (dolomite) and up to 86% (limestone). Porosity varied +/-3% for dolomite and +/-20% for limestone (Ricardo, 2012).

Gupta (2010) studied the capacity and constraints for CO₂ sequestration in aquifers and depleted carbonate reservoirs and found that after aging two of the samples with CO₂ for one and two weeks, the final measured porosity-permeability clearly indicate reduction in both porosity and permeability.

Izgec et al. (2006) performed an experimental and numerical modeling of direct injection of CO₂ into carbonate formations and they showed using CT monitored core-flood experiments and core scale and field scale simulations that 1) chemical reactions occurred preferentially at the center of the core (at the inlet) where CO₂ injection is performed, 2) The common feature in all experiments was that dissolution occurred at the inlet face. 3) the duration of CO₂ – rock contact and the amount of area contacted by CO₂ seems to have a more pronounced effect compared to rate effect, 4) for the temperature range studied (18 to 50°C) permeability and porosity alteration trends were similar, 5) either a permeability improvement or a permeability reduction can be obtained through the injection of CO₂ into carbonate aquifers. The trend of change in rock properties is very case dependent because it is related to distribution of pores, brine composition and as well the thermodynamic conditions, 6) Precipitation process can impact the permeability drastically while small change in porosity is observed.

It is clear that there is still a debate in the literature about variation in petrophysical properties. Therefore, this study is designed to experimentally investigate (1) CO₂

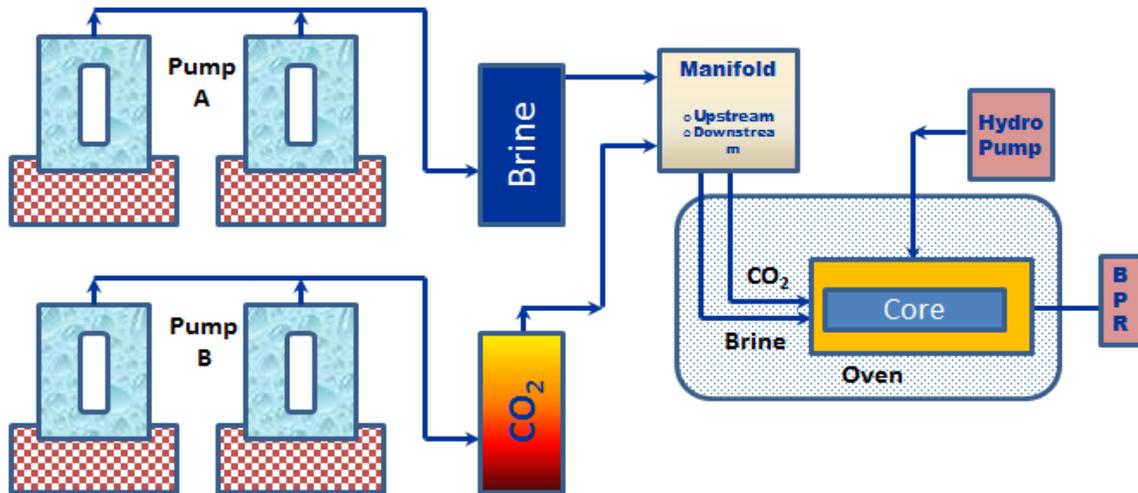


Figure 2. Schematic diagram of the experimental apparatus.

solubility under different brine salinity and also under different conditions of pressure and temperature, and (2) variation in porosity and permeability due to CO₂ storage in carbonate rocks.

EXPERIMENTAL SET UP AND PROCEDURES

Two groups of experiments were carried out. These two groups included; (A) solubility tests for carbon dioxide in different salinities and under different pressure and temperature conditions, and (B) core storage experiments to study variation in porosity and permeability under different time intervals from 7 to 170 days. The procedure for each experimental group is described below;

Solubility tests

The experimental procedure for investigating the solubility in distilled water and in different salinity brines can be summarized as follows;

1. The laboratory equipment, as shown in Figure 2, was calibrated to secure accurate measurements.
2. A brine of a specific salinity concentration or distilled water is pumped into the system according to the required volume.
3. A specific volume of CO₂ was then injected into the distilled water and also into brine of different salinities.
4. For each experiment, the volume of CO₂ was released from solution for each pressure depletion step, removed and measured at atmospheric conditions using a gas flow meter.
5. The procedure was repeated for different conditions of brine salinity, pressure, and temperature.

Variation in rock properties experiments

The experimental apparatus consists of core holder, injection system, and data recording system, as shown in Figure 2. The injection system includes constant rate displacement pumps (A and B), brine tank, CO₂ cylinder, gas flow meter, back pressure regulator (BPR), and pressure transducers.

The experimental procedure for studying the variation in porosity and permeability in carbonate reservoir rocks can be described as follows;

1. Core samples are evacuated for 24 h, and then saturated with brine of 25000 ppm NaCl solution. From weight difference, initial porosity is calculated. The permeability is measured under steady-state Darcy's flow condition.
2. The CO₂ is injected at constant pressure of 1000 psi continuously until irreducible brine saturated is well established.
3. The outlet is closed and the CO₂ is injected at 1000 psi and the pressure along the core sample is monitored till pressure equilibrium is achieved along the core sample.
4. The change in porosity and permeability is calculated by measuring these properties after each experiment.
5. The above procedure is typically repeated for different storage time intervals ranged from 7 to 170 days under 2 400 psi pressure and 200°F temperature.

RESULTS AND DISCUSSION

Tables 1, 2, and 3 presented the experimental results attained for carbon dioxide solubility for different brine salinity and under different pressure and temperature conditions. The graphical presentations of this data are shown in Figures 3, 4, and 5. Comparison of the results listed in Tables 1, 2, and 3, and Figures 3, 4, and 5, indicated that the increase of reservoir pressure decreases the carbon dioxide solubility for different brine salinity and under different temperature conditions.

Effect of pressure on carbon dioxide (CO₂) solubility

Figure 3 for distilled water, indicates that for the same brine salinity and the same reservoir temperature, the increase of pressure increases the carbon dioxide solubility. The same trend is also shown in Figures 4 and

Table 1. Carbon dioxide solubility in distilled water at different temperatures.

Pressure (psia)	Carbon dioxide solubility (SCF/STB) at different temperature			
	P / T	70°F	120°F	170°F
0		0	0	0
300		75.8	48.6	29.5
600		126.4	83.4	61.8
900		158.6	123.8	92.6
1200		168.5	137.8	110.3
1500		179.8	147.9	123.7
2000		195.2	163.2	144.2
4000		210.3	186.1	179.3
6000		227.4	196.4	193.1

Table 2. Carbon dioxide solubility in 15 000 ppm NaCl water at different temperatures.

Pressure (psia)	Carbon dioxide solubility (SCF/STB) at different temperature			
	P / T	70°F	120°F	170°F
0		0	0	0
300		48.6	34.7	23.5
600		91.4	64.7	47.8
900		128.6	73.8	74.6
1200		132.5	101.8	77.3
1500		138.5	113.6	102.3
2000		153.6	122.5	106.4
4000		162.9	138.4	137.7
6000		177.6	141.6	140.8

Table 3. Carbon dioxide solubility in 25 000 ppm NaCl water at different temperatures.

Pressure (psia)	Carbon dioxide solubility (SCF/STB) at different temperature			
	P / T	70°F	120°F	170°F
0		0	0	0
300		25.8	21.8	20.1
600		86.4	56.2	53.3
900		102.6	75.6	70.3
1200		121.5	86.5	73.4
1500		136.2	108.9	84.6
2000		141.6	120.2	100.2
4000		156.3	128.1	125.8
6000		160.2	139.4	138.8

5 for different salinities of 15 000 ppm and 25 000 ppm NaCl brines, respectively. Therefore, a conclusion can be drawn that the increase of pressure increases the CO₂ solubility for different brine salinities and different

reservoir temperatures. It is also clear that for temperature equal to or more than 120°F, the temperature effect diminishes and starts to have no effect, especially under pressures equal to or greater than 4000 psia. The reported data has real importance on validation simulation models describing the process of CO₂ storage in fresh and brine aquifers. It is also concluded that the increase of temperature and/or salinity increases the CO₂ solubility.

The results of four storage experiments undertaken studying the variation of porosity and permeability of carbonate reservoir are listed in Table 4 for different storage time intervals. Table 4 shows the petrophysical properties for the core samples used and the CO₂ storage time in carbonate reservoir rock under 2400 psia pressure and 200°F reservoir temperature.

Effect of temperature and salinity on carbon dioxide (CO₂) solubility

Based on the experimental results obtained, it is found that, as the solubility decreases the temperature increases for all prepared solutions at a given pressure, that is, the same results of distilled water, and for any brine salinities. On other hand, at a constant temperature, the CO₂ solubility increases with increase of the applied pressure. These results are depicted in Figures 6, 7, and 8 for distilled water, 15000 ppm, and 25000 ppm salinity respectively. These results are valid for any pressure applied on the system, since the pressure range studied in this work are varied from 300 to 6000 psi.

By comparing these Figures (6, 7, and 8), it is very clear that as salinity increases as the solubility decreases. This can be proved, if the CO₂ solubility at a given constant temperature and pressure have been picked and compared for different salinities. For example, the CO₂ solubility for distilled water at pressure 2000 psi and 120°F is 163.2 SCF/STB, for 15 000 ppm brine-salinity is 122.5 SCF/STB, and for 25 000 ppm brine-salinity is 120.2 SCF/STB.

Variation of petrophysical properties

Figure 9 presents the variation in porosity for the four core samples used under different storage time intervals from 7 to 170 days. Figure 9 shows that the porosity is decreased by almost 20% when the carbon dioxide is stored for 7 days. On the other side, the porosity was increased when the storage time was increased to 150 days and more. This analysis can be proven if the initial and final porosity plotted versus the time duration as shown in Figure 10. From the previous figure; one can conclude that, the porosity of carbonate rocks is decreased with the aging time from zero day to about 85 days, then the relationship reversed, which means the final rock porosity is increased if the time is greater than

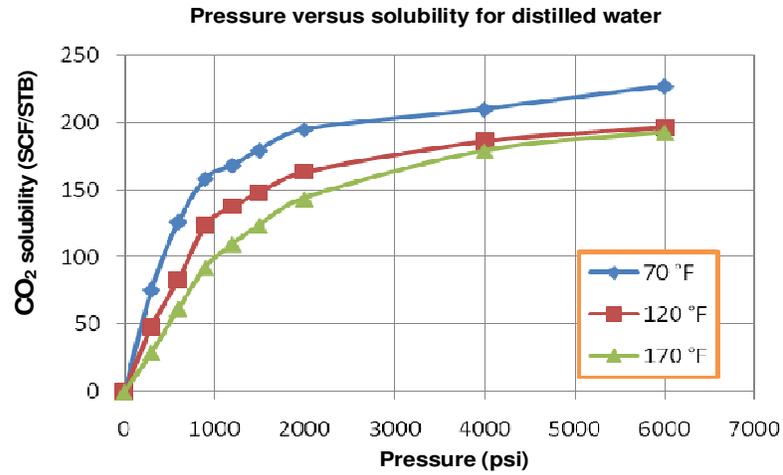


Figure 3. Carbon dioxide solubility in distilled water.

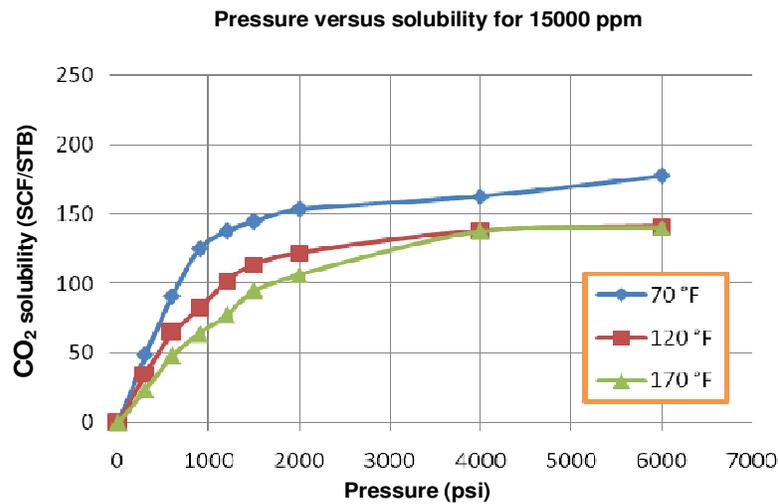


Figure 4. Carbon dioxide solubility in 15000 ppm NaCl brine.

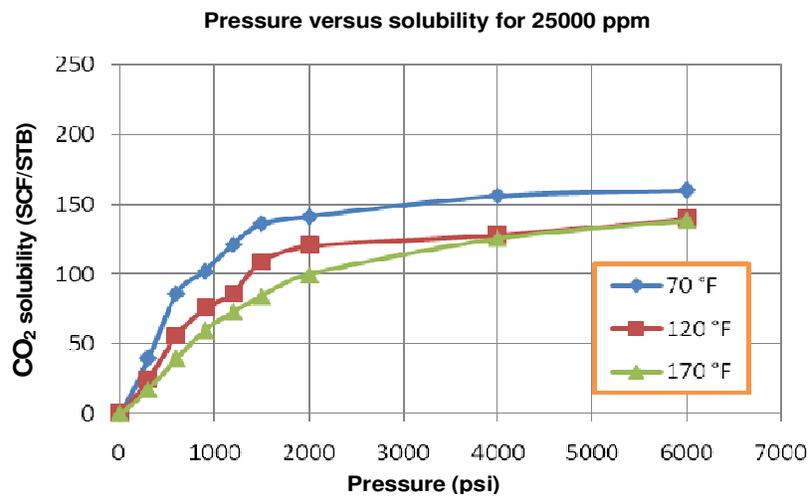


Figure 5. Carbon dioxide solubility in 25 000 ppm NaCl brine.

Table 4. Petrophysical properties of used core samples.

Sample code	PV (cc)	Initial porosity (%)	Final porosity (%)	Initial K (md)	Final K (md)	CO ₂ storage time (days)
SF-1	7.35	18.57	14.72	10.8	7.71	7
AC-4	8.21	20.12	23.35	69.12	88.05	150
BK-6	8.86	21.23	25.42	57.31	65.87	160
SD-2	9.34	26.45	29.83	75.42	90.23	170

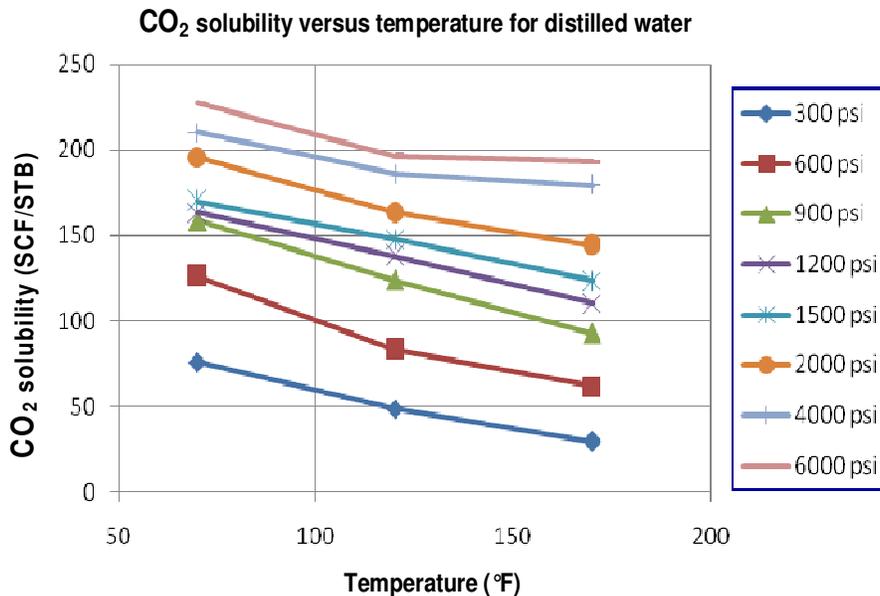


Figure 6. Carbon dioxide solubility versus temperature for a distilled water at pressure range 300 to 6000 psi.

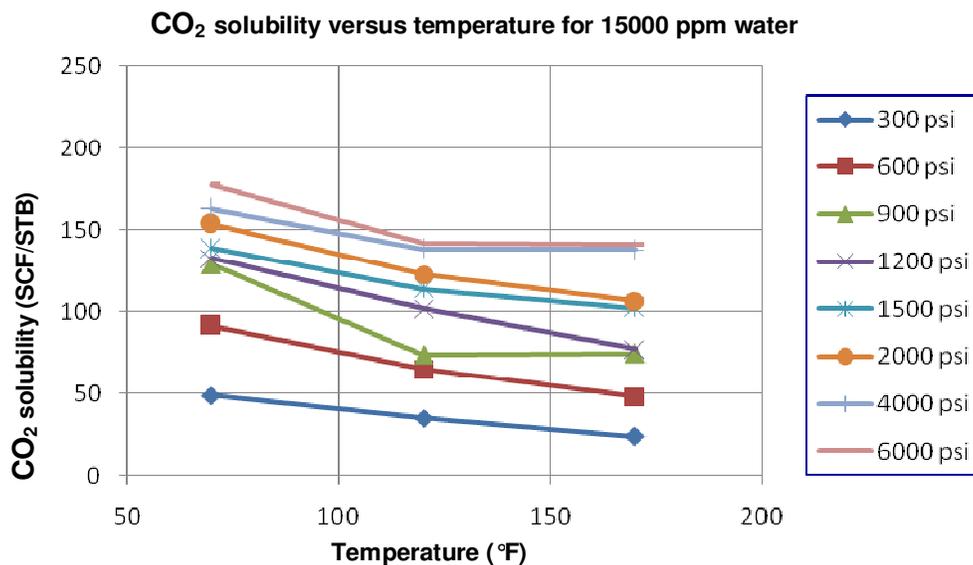


Figure 7. Carbon dioxide solubility versus temperature for a 15000 ppm water salinity at pressure range 300 to 6000 psi.

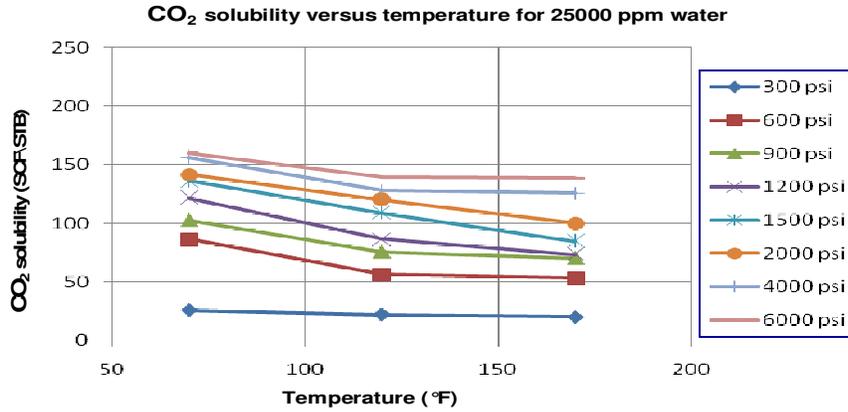


Figure 8. Carbon dioxide solubility versus temperature for a 25000 ppm water salinity at pressure range 300 to 6000 psi.

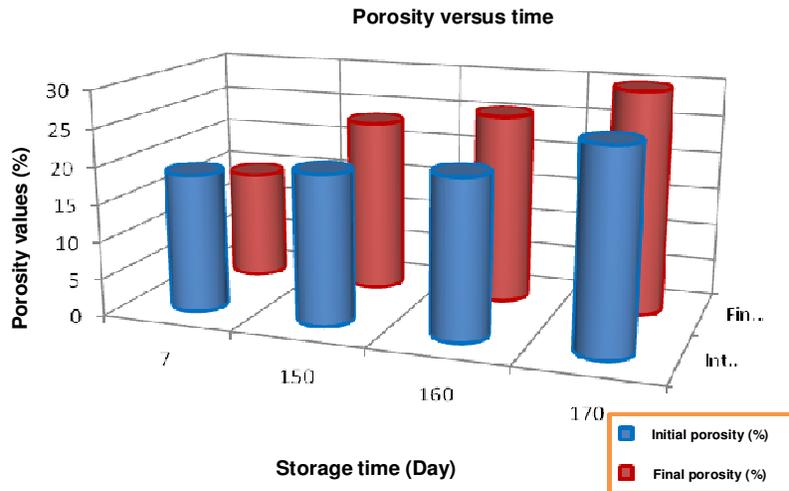


Figure 9. Initial and final porosity for different storage time intervals.

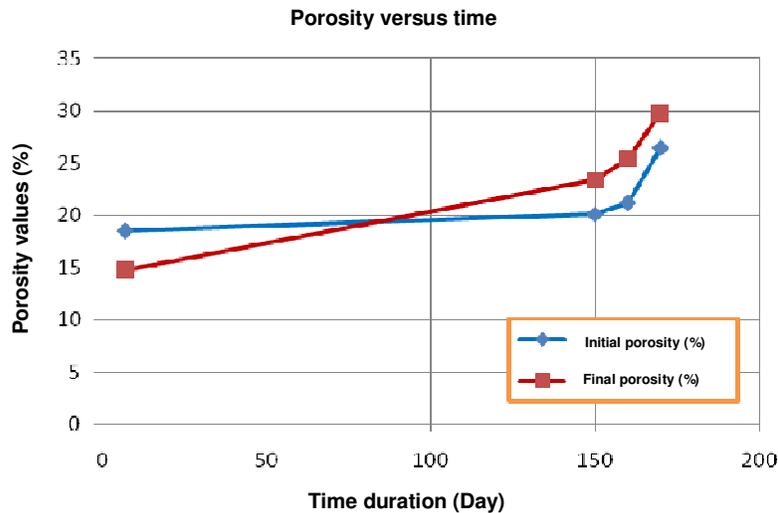


Figure 10. Porosity (initial and final) versus aging time.

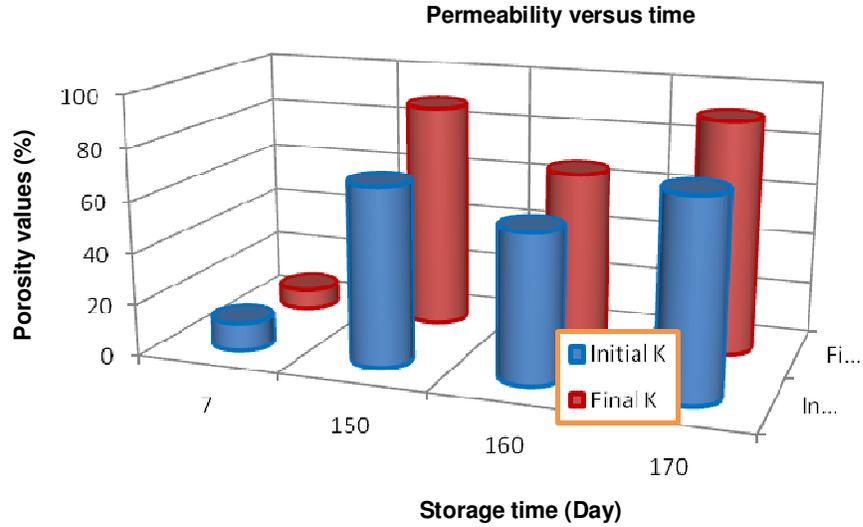


Figure 11. Initial and final permeability for different time intervals.

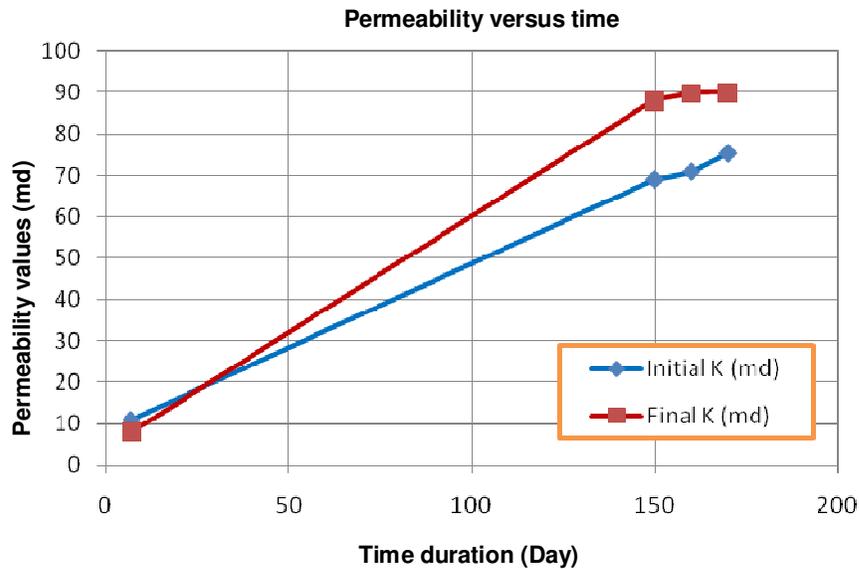


Figure 12. Permeability (Initial and final) versus storing time.

85 days.

For the carbonate rock permeability, the same trend is shown for permeability variation in Figure 11. By investigating this figure, we found that the permeability decreased in the first 27 days of storing CO₂, after that time, the rock permeability has increases with increasing the storing time remarkably as shown in Figure 12. These results can be attributed to the dissolution and precipitation processes that happened inside the pore space due to the geochemical reaction of CO₂ and the limestone and calcite that constitutes our core plugs.

Therefore, it is concluded that the carbon dioxide storage has to be for long time interval to provide enough

time for a complete carbonate dissolution process, which led to an increase in porosity and permeability. The attained data confirmed the results of Gupta (2010) that the porosity was decreased for some cores and improved for other ones.

Conclusions

This experimental study investigated the effects of brine salinity, pressure and temperature on carbon dioxide solubility plus the variation in porosity and permeability of carbonate reservoirs, the following conclusions can be

drawn;

1. The application of CO₂ sequestration and storage in carbonate reservoirs has significant potential to significantly reduce the emission of CO₂ into the atmosphere.
2. The CO₂ solubility in aquifer's brine decreases with an increase in salinity. The temperature effect diminishes and starts to have no effect, especially under pressures equal to or greater than 4000 psia.
3. The CO₂ solubility in brine decreases with increasing reservoir temperature.
4. The increase of pressure increases the CO₂ solubility for different reservoir temperatures and different brine salinities.
5. For reservoirs or aquifers of moderate porosity and good permeability, the porosity and permeability are decreased when CO₂ is stored in carbonate reservoir rocks for short period of time equals to 7 days and increase when stored for longer time intervals equal to or greater than 150 days.

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