An Experimental Study of the Response of the Galesville Sandstone to Simulated CAES Conditions

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July 1980

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AN EXPERIMENTAL STUDY OF THE RESPONSE
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PREFACE

Compressed air energy storage (CAES) is a technique for storing heated air in an underground reservoir; the air can subsequently be withdrawn, mixed with fuel, burned, and allowed to expand through turbines to generate peak power in electric utility systems. Several types of geologic reservoirs, including mined salt cavities, hardrock caverns, and aquifers, have been proposed for CAES. The technical and economic feasibility of CAES technology depends to a major extent on locating, developing, and maintaining an adequate subsurface reservoir. A fundamental uncertainty is the manner in which a reservoir might physically and chemically respond to new environmental conditions imposed by CAES operation.

The laboratory investigation discussed in this report addresses the possible interactions of air and water with an aquifer sandstone under environmental conditions anticipated for CAES. It is part of a Reservoir Stability Program designed to develop criteria for the long-term operation of underground reservoirs used for compressed air energy storage. The Compressed Air Energy Storage Technology Program at the Pacific Northwest Laboratory (PNL) is sponsored by the Department of Energy, Division of Energy Storage Systems. PNL is operated for the Department of Energy by Battelle Memorial Institute.

SUMMARY

The objective of this experimental study was to determine how the mineralogical and physical characteristics of host rock formations are affected by environmental conditions anticipated for compressed air energy storage (CAES) in porous, permeable rock. In this study, Galesville sandstone cores were reacted in autoclave pressure vessels supporting one of four environments: 1) dry air, 2) heated, air-water vapor, 3) heated, nitrogen-water vapor mixtures, and 4) heated, compressed, liquid water. The simulated CAES environments were maintained in autoclave pressure vessels by controlling the following independent variables: 1) temperature, 2) pressure, 3) time, 4) oxygen content, 5) carbon dioxide content, 6) nitrogen content, and 7) liquid volume. The dependent variables studied were: 1) apparent porosity, 2) gas permeability, 3) water permeability, and 4) friability. These variables were measured at ambient temperature and pressure before and after each sandstone sample was reacted in one of the CAES environments.

The dry air and gas-water vapor tests were designed to simulate CAES conditions. The liquid tests were designed to monitor groundwater chemical changes resulting from reaction of sandstone in an aqueous fluid. The liquid tests may also simulate isolated, low permeability zones which may remain saturated (irreducible water content) under actual CAES field conditions.

The experiments gave the following results:

- The Galesville sandstone exhibited excellent stability in dry air at all temperatures tested (50° to 300°C).
- Significant physical alterations occurred in sandstone samples exposed to liquid water above 150°C.
- Preliminary experiments had indicated that significant physical alterations occurred in sandstone samples exposed to gas-water vapor mixtures above 150°C. However, these alterations were later proven to be the result of water condensation inside the pressure vessel. In later experiments, samples shielded from dripping water exhibited excellent stability to 300°C.

- The porosity and friability of the sandstone samples increased as a function of increased temperature, humidity, and/or testing time.
- The sandstone also exhibited greater changes in gas and liquid permeability with increased temperature, humidity, and/or testing time. However, both permeability increases and decreases were observed.
- The changes in the physical stability of the sandstone were postulated to result from the dissolution of interstitial quartz cement binding the individual grains.
- Sandstone may be a suitable storage media for heated, humid air provided elevated temperature zones are relatively free of mobile liquid water.
- Observed changes in the physical properties of the rock may have been caused, in part, by the lack of confining stress on the sample. The inability to apply confining pressure is a severe limitation of autoclave experiments.

Future experiments will be conducted in the CAES triaxial/fluid flow test facility being completed at PNL. This facility will eliminate many of the limitations of autoclave pressure vessels and will allow a more accurate determination of physical and chemical property changes in porous media exposed to simulated CAES conditions.

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AN EXPERIMENTAL STUDY OF THE RESPONSE OF THE GALESVILLE SANDSTONE TO SIMULATED CAES CONDITIONS

INTRODUCTION

The Galesville sandstone (located at the Media natural gas storage field, Illinois) appears to be a reasonable candidate rock for compressed air energy storage (CAES). This sandstone is currently under consideration for a CAES field demonstration. However, it is not known how this sandstone will physically and chemically behave in the hot, humid conditions anticipated for CAES. This laboratory study addresses the possible interactions of hot air and water with the Galesville sandstone.

Although the chemical composition is regionally variable, the samples used in this study are from a clean sandstone (approximately 100% quartz) that exhibits gas permeabilities, parallel to the bedding planes, ranging from 20 to 2400 millidarcies (md). To determine how the reservoir rock may respond to anticipated CAES conditions, cylindrical cores of sandstone were reacted in autoclave pressure vessels. The physical rock properties of primary interest include apparent porosity, gas permeability, liquid permeability and friability. These physical properties for individual sandstone cores were measured before and after each core had been subjected to various combinations of pressure, temperature, humidity, gas composition, and experiment duration.

This report gives the results of the experiments, along with the procedures used. Appendix A lists the experimental data, and Appendix B details the analytical methods and procedures. The reported results are applicable only to the specific samples of Galesville sandstone used in these experiments. Experimental results and conclusions should be viewed in a qualitative sense; the diversity of physical and chemical properties for porous rocks generally does not warrant an extrapolation of quantitative results between different rock types. As CAES candidate sites are selected, each potential host rock should be evaluated with the tools and methodology developed in the Reservoir Stability Program.

CONCLUSIONS AND RECOMMENDATIONS

The following observations and conclusions may be deduced from the experimental results:

- The Galesville sandstone appears to be physically stable at temperatures between 50° and 300°C if exposed to dry air.
- Physical alterations of the sandstone exposed to liquid or vapor phase water at temperatures less than 150°C were minimal for the duration of these experiments. Above 150°C, the physical stability of the rock can be extensive, especially in a heated, liquid water environment.
- The porosity and friability of all sandstone samples increased as a function of increased temperature, humidity, or time.
- Gas and liquid permeability changes were also greater for those samples exposed to the higher temperature and humidity conditions. However, both increases and decreases in permeabilities were observed.
- Much of the scatter evident in the permeability results at high temperature was caused, in part, by the surface disaggregation of the sandstone cores. This disaggregation, especially for those cores reacted in heated groundwater, often prevented accurate determination of permeability. Applying a confining pressure to the sample to simulate the lithostatic pressure (weight of overburden) in a CAES reservoir may alleviate this problem.
- The chemical composition of the groundwater monitored during experiments in which sandstone was reacted in liquid indicated insignificant changes in the concentrations of Na, K, Mg, and Ca with time. The concentration of Si increased rapidly at first and then became constant with time. The change in the concentration of Si was most likely caused by dissolution of quartz. However, this chemical reaction was not detected in petrographic analysis of thin sections of reacted sandstone.

- The dissolution of quartz cement was the probable cause for the disaggregation of the sandstone. This disaggregation could result in increased rock permeability. However, it is possible that dislodged particles may ultimately reduce the permeability by plugging the formation. An increase in permeability may be beneficial, but the potential for subsequent particulate plugging cannot be dismissed.
- Sandstone may be a suitable storage media for heated, humid air provided elevated temperature zones are relatively free of mobile, liquid phase water.

The results and conclusions presented in this report are considered preliminary due to the many limitations of autoclave pressure vessels previously described. The observed changes in permeability, porosity, and friability of sandstone may be attributed partly to the inability to accurately simulate CAES reservoir environments. In the porous media flow facility currently being fabricated, the following conditions can be simultaneously monitored: 1) triaxial load on the sample, 2) flow of heated, dry or humid air through porous geologic materials, (e.g., sandstone), 3) axial and radial deformation of the rock, 4) the pressure drop along the axis of the core, and 5) effluent air humidity. Using this apparatus, it will be possible to determine changes in the physical properties of the sandstone at the temperature and pressure of the experiment. Samples of reservoir rock subjected to the above conditions will provide more useful data on the physical and chemical stability of sandstone subjected to environments characteristic of CAES.

EXPERIMENTAL FACILITIES AND PROCEDURES

The main objectives of the experiments were to identify: 1) independent variables that have significant impact on the experimental response of the rock, 2) independent variables that have a relatively small influence and can be held constant in future experiments, and 3) trends in the data that pertain to CAES reservoir design and/or operating criteria. The dependent variables of primary interest were: 1) gas permeability, 2) water permeability, 3) apparent porosity, and 4) rock friability.

EXPERIMENTAL EQUIPMENT

To adequately simulate field conditions anticipated for CAES, the sample should be heated, subjected to axial and radial confining pressure, and ventilated with air of known pressure, humidity, temperature, and flow rate. Measurements of the pressure drop along the axis of the rock core, axial and radial deformation, effluent air humidity and suspended particle load are of principal concern. A facility that will meet these criteria is currently nearing completion at PNL. In the interim, autoclaves are being used to obtain preliminary data for humid air conditions. Cyclical dry air tests are being performed at the University of Wisconsin at Milwaukee.

Figure 1 illustrates the existing autoclave facility. Cylindrical sandstone specimens are subjected to one of four environments; 1) heated, dry air, 2) heated, humidified air, 3) heated, humidified nitrogen, or 4) heated liquid. The following independent variables can be controlled in the facility: 1) temperature (25°-300°C), 2) pressure (1-175 bars), 3) time (unrestricted), 4) oxygen content (0-21%), 5) carbon dioxide content (0-1%), 6) nitrogen content (78-100%), 7) liquid volume (0-600 ml), and 8) fluid stirring frequency (0-250 rpm). Although autoclaves are an expedient means for obtaining preliminary data, certain limitations must be noted. The fundamental limitations of the autoclave experiments are: 1) no effective stress is imposed on the sample; 2) samples are subjected to an uncharacteristically large fluid/rock volume ratio; 3) no control of fluid transport is possible; and 4) permeability, porosity, and strength measurements are possible only at ambient temperature and pressure prior and subsequent to sample treatment.

FIGURE 1. Autoclave Pressure Vessel Laboratory

SAMPLE PREPARATION AND CHARACTERIZATION

The Galesville sandstone samples used in the experiments were supplied by Geo-Engineering Laboratories, Inc. and the Northern Illinois Gas Company. Cores with 10-cm diameters were obtained from the Media natural gas storage reservoir. The Galesville sandstone is white to grayish-white and has an estimated mean grain size of 0.5 mm. Petrographic examination revealed that the subangular clasts have equant to subequant forms. The rock is moderately well sorted and exhibits cyclic parallel bedding of thin, coarser-grained layers and thick, finer-grained layers.

Cylindrical cores (1.8 cm diameter x 3.0 cm in length) were drilled, parallel to the bedding planes, from four field cores obtained from a depth of 685 m. Each 1.8 cm x 3.0 cm core was labeled with a letter prefix (i.e., D, I, J, or K), identifying the parent core from which it was cut, and a consecutive number.

The mineralogy was estimated from microscopic and X-ray diffraction techniques. The results showed the Galesville sandstone to be an orthoquartzite with the approximate mineralogy listed in Table 1. The clasts are almost entirely quartz with trace amounts to 1% of feldspar that has been partially weathered to kaolinite. The cementing material is predominantly crystalline quartz and trace amounts of interstitial calcite. In addition to these minerals, the K-series parent core also contains small amounts of dolomite identified by X-ray diffraction analysis. Quartz was the only detectable phase present in X-ray diffractograms of the D, I, or J series core material. Photomicrographs of thin-sectioned Galesville (D-series) sandstone are illustrated in Figures 2a and b.

TABLE 1. Estimated Mineralogy of the Galesville Sandstone

<u>Mineral</u>	<u>Volume %</u>	<u>Mineral</u>	<u>Volume %</u>
Quartz	98	Dolomite	0-5
Feldspar	1	Mica	<1
Kaolinite	<1	Opakes	<1
Calcite	<1

a. Plane Polarized Light (50X)

b. Crossed Nicols (Mottled Kaolinite, Yellow Calcite, Other Quartz) (50X)

FIGURE 2. Unreacted Galesville Sandstone

Groundwater from the Galesville sandstone horizon was collected and its chemical composition determined. A synthetic aqueous solution was prepared to simulate the chemical composition of actual waters currently in equilibrium with the Galesville. The composition of the synthetic groundwater used in these experiments is listed in Table 2.

TABLE 2. Chemical Composition of Synthetic Groundwater

<u>Constitutents</u>	<u>Concentration (mg/l)</u>
Na	430
Ca	50
K	200
Mg	14
Si	<0.05
SO ₄ ²⁻	300
CO ₃ ²⁻	150
Solution pH = 8.0	

EXPERIMENTAL STRATEGY AND PROCEDURES

The experimental work was completed in two parts: 1) screening experiments, and 2) detailed experiments. In the screening experiments, a Plackett-Burman (1946) experimental design was selected to systematically investigate the response of the dependent variables to various combinations of maximum and minimum values of the independent variables. Using this method, insignificant independent variables could be eliminated from further consideration. The responses of the dependent variables were then determined as a function of intermediate values of the most significant independent variables in a set of detailed experiments. The total number of required experiments was significantly reduced by this experimental approach. The ranges of independent variables used in this study are listed in Table 3.

In all experiments, the sandstone samples were exposed to one of the following environments: 1) heated, dry air, 2) heated, air-water vapor or

nitrogen-water vapor mixtures, or 3) heated, liquid (ground) water. Measurements of gas permeability, water permeability, and friability were obtained before and after each core had been reacted in one of the above environments. Apparent porosity was later added as a fourth dependent variable. Details of the measurement techniques for the dependent variables are discussed in Appendix B.

TABLE 3. Range of Independent Variables

<u>Variable</u>	<u>Minimum Value</u>	<u>Maximum Value</u>
Pressure (bars)	60	120
Temperature (°C)	50	300
Time (months)	0.5	2.0
Oxygen Content (%)	0.0	21.0
Carbon Dioxide Content (%)	0.0	0.1
Nitrogen Content (%)	78.0	100.0
Stirring Frequency (per day)	0	8
Liquid Saturated Environment	No	Yes
Air and Vapor Saturated Environment	No	Yes

EXPERIMENTAL RESULTS

The experimental results for the screening and detailed experiments are discussed in the following section. Results of gas permeability, water permeability, apparent porosity, and friability measurements as well as other physical and chemical responses of the sandstone are summarized.

SCREENING EXPERIMENTS

During the parametric screening survey of the Galesville sandstone, gas permeability, water permeability, and matrix friability were analyzed. Table 4 summarizes the changes in relative percentage observed in values for the three dependent variables for each of 12 screening experiments. The experiments represent various maximum-minimum combinations of the independent variables (pressure, temperature, time, oxygen content, nitrogen content, carbon dioxide content, and humidity.)

Gas Permeability

The analysis of the gas permeability test matrix resulted in the following conclusions:

- Temperature and humidity are the dominant independent variables.
- The duration of the experiment has a moderate effect.
- Pressure, oxygen content, carbon dioxide content, nitrogen content, and autoclave stirring frequency have negligible effects and may be held constant in all future experimental strategies.

The gas permeability data listed in Table 4 are graphically presented in Figure 3. Changes in gas permeability are shown in the circles as either increasing (+) or decreasing (−) a certain percentage from the pre-test values. The independent variables represented by the x, y, and z axes are temperature, relative humidity, and time, respectively. Each sandstone core was reacted in a dry air environment (D), a gas-water vapor environment (V), or a liquid (ground)-water environment (L). Summary observations and conclusions for gas permeability tests are as follows:

TABLE 4. Test Matrix for Screening Experiments

Trial	Pressure (bars)	Temperature (°C)	Time (months)	Oxygen (%)	CO ₂ (%)	Stirring (Yes-No)	Liquid (Yes-No)	Vapor (Yes-No)	Permeability ⁽¹⁾ (% Change)		Friability ⁽¹⁾ Index (% Change)
									Gas	Liquid	
1	120	300	0.5	21	0.1	Y	N	N	+03	00	+ 27
2	120	50	2.0	21	0.1	N	N	N	+02	+04	+ 41
3	60 ⁽²⁾	300	2.0	21	0.0	N	N	Y	+31	+78	+222
4	120	300	2.0	0	0.0	N	Y	N	+65	+73	+474
5	120	300	0.5	0	0.0	Y	N	Y	+76	+38	+185
6	120	50	0.5	0	0.1	N	Y	Y	+17	+37	+ 29
7	60	50	0.5	21	0.0	Y	Y	N	+03	+08	+ 43
8	60	50	2.0	0	0.1	Y	N	Y	+18	+31	+ 26
9	60 ⁽²⁾	300	0.5	21	0.1	N	Y	Y	+96	+36	+171
10	120	50	2.0	21	0.0	Y	Y	Y	+11	+51	+ 61
11	60 ⁽²⁾	300	2.0	0	0.1	Y	Y	N	+12	+28	+480
12	60	50	0.5	0	0.0	N	N	N	+02	+04	+ 31

(1) Percent change calculated from pre-test and post-test values for each sample.

(2) Actual pressure increased to prevent flashing.

FIGURE 3. Percent Change in Gas Permeability

- The sandstone cores demonstrate good stability with respect to gas permeability if one or both of the following conditions exists; 1) temperatures are 150°C or less, and/or 2) water content is negligible.
- The average gas permeability change is approximately 20% for the environmental conditions of the 12 screening experiments.
- Observed changes in permeability may be attributed, in part, to erosion of the cylindrical core face. This erosion can result in the bypass of gas around the core during the post-test permeability measurement. The erosion may be due to the lack of confining pressure on the sample during the experiment and/or the high ratio of fluid to rock volumes (typical fluid to rock ratio in these experiments was 40:1).
- The number of experiments and the inhomogeneity of the parent rock core limit the precision of the data. Additional experimental replication is warranted.

Liquid Permeability

Liquid permeability data are illustrated in Figure 4. All experimental conditions are identical to those for the gas permeability tests. The analysis of the matrix data gave the following results:

- Temperature, time, and relative humidity are the dominant independent variables.
- Stirring frequency in the autoclave has a moderate effect.
- Oxygen content, nitrogen content, carbon dioxide content, and total interstitial pressure have negligible effects.
- For the range of conditions tested in the screening experiments, the average increase in liquid permeability is approximately 14%, which is similar to that observed for the change in gas permeability.

Matrix Friability

The matrix friability index is designed to measure the shear rupture modulus and grain-to-grain contact strength of sandstone cores before and after treatment at simulated CAES conditions. Figure 5 illustrates the friability data. The following observations were noted from the test matrix results:

- Temperature, time, and humidity are the dominant independent variables.
- Oxygen content and vapor saturation conditions also have moderate effects.
- Pressure, nitrogen content, carbon dioxide content, and stirring frequency are insignificant.
- All combinations of independent variables resulted in an increase in friability and in the disaggregation of the sandstone.
- The lack of confining stress on the cores contributed to the disaggregation of the samples.

FIGURE 4. Percent Change in Liquid Permeability

FIGURE 5. Percent Change in Matrix Friability

- The disaggregation of the sandstone may be caused by dissolution of the interstitial cements (quartz and carbonate). The large ratio of liquid to solid volume probably enhances this chemical reaction. Figures 6a and 6b contrast the varying degree of disaggregation, as a function of temperature and time, that occurred when the Galesville sandstone was subjected to a heated, liquid environment.

DETAILED EXPERIMENTS

The results of the screening tests indicated that temperature, time, and humidity are the dominant independent variables affecting the stability of the Galesville sandstone. Therefore, these variables were studied in more detail during the second phase of the experimental program. The insignificant independent variables (pressure, O₂ content, N₂ content, CO₂ content, and stirring frequency) were held constant in these experiments.

All experimental results for the Galesville sandstone obtained in this study are listed in Appendix A. Included in this Appendix are data for both screening experiments (EXP G-1 to EXP G-14) and detailed tests (EXP G-15 to EXP G-42). Beginning with experiment G-22, note that: 1) apparent porosity was selected as a dependent variable in addition to gas permeability, water permeability, and friability; and 2) two cores were reacted simultaneously. Simultaneous reaction of two cores per experiment improved the statistics of the dependent variable measurements. Only a maximum of two cores could simultaneously be reacted per autoclave because of the size limitations of the pressure vessels.

Physical Responses

The disaggregation observed (Figure 6) during the screening and detailed tests for some sandstone samples reacted in humid gas above 200°C was somewhat unexpected. If dissolution of quartz is mainly responsible for changes in physical rock properties and disaggregation of the cores, sandstone reacted in gas-water vapor mixtures should exhibit smaller changes in these properties than in 100% liquid experiments. This result is expected since the solubility of quartz in water vapor is immeasurably small relative to its solubility in

a. 50°C, 336 hours, Liquid

b. 300°C, 1488 hours, Liquid

FIGURE 6. Increasing Disaggregation of Galesville Samples Reacted in a High Humidity Environment at Elevated Temperatures

liquid water at temperatures and pressures below the critical point of water (Kennedy, 1950). Therefore, it was postulated that water was condensing above the core in cooler regions of the autoclave and dripping onto the core. If such were the case, the condensate would leach the sandstone and cause extensive surface disaggregation.

To investigate this hypothesis, gold foil was folded on top of two cores before they were placed in the pressure vessel (Appendix A, EXP G-26). The foil shielded the cores from any condensation forming at the top of the autoclave. The results of this experiment are illustrated in Figure 7a. The surface of the core (J13) on the right in this photograph is slightly friable compared to the extensive surface pitting and spalling of grains present in unshielded cores reacted under identical conditions (e.g., Figure 7b). The core on the left in Figure 7a (J19) exhibits obvious pitting and disaggregation over a small area of the core. As seen in the photograph, the gold foil was placed above the core with part of the foil in direct contact with the core. Because of improper shielding, water was transported from the area of contact along the length of the core. As a consequence, the surface pitting resulted. The shielded portion of this core exhibited no evidence of disaggregation.

Because the disaggregation appeared to be an experimental difficulty, some doubt remained as to the validity of the post-experiment physical properties measured on unshielded cores. Therefore, in experiments involving reaction of sandstone cores in the vapor phase conducted after experiment G-26, gold foil shields were used to prevent this anomalous disaggregation.

Figures 8 through 11 illustrate the response of each of the dependent variables as a function of temperature and humidity. In these figures, a distinction is made between shielded and unshielded cores reacted in a humidified air environment. The experimental results in Figures 10 and 11 indicate that porosity and friability increased in all simulated CAES environments. In contrast, both increases and decreases in rock gas and water permeability were noted in these experiments (Figures 8 and 9). Small changes in all physical properties were measured at temperatures less than or equal to 150°C. Changes in gas permeability, water permeability, and porosity increased at a greater

a.

b.

FIGURE 7. Results of a) Experiment G-26 (Cores J-19 and J-13), Run Conditions are 300°C, Vapor, 355 Hours, and b) Experiment G-5, Core D-5, Run Conditions are 300°C Vapor, 360 Hours, Core Unshielded

FIGURE 8. Percent Change in Gas Permeability with Temperature

FIGURE 9. Percent Change in Water Permeability with Temperature

FIGURE 10. Percent Change in Friability with Temperature

FIGURE 11. Percent Change in Apparent Pososity with Temperature

rate above 150°C. Values for friability exhibited a marked increase between 150° and 300°C. Overall, the greatest changes in physical properties occurred in those cores reacted in a liquid environment. However, this is the least likely environment to be encountered in an elevated temperature CAES reservoir. An important trend in the data is that insignificant changes in all physical properties were observed for 0% humidity (dry air) conditions, regardless of the temperature of the experiment. For the most part, similar observations were made for shielded cores reacted in water vapor environments.

Much of the experimental scatter in the data is the result of disaggregation of samples under conditions of high humidity and high temperature conditions. Many difficulties were encountered obtaining accurate measurements of physical properties for these delicate samples. In some cases, due to extensive disaggregation, no measurements could be obtained (indicated as ND in Appendix A). Therefore, changes in permeability or degradation of the Galesville sandstone under simulated CAES conditions cannot be accurately predicted if based solely on the data obtained in the autoclave experiments. The porous media flow facility currently being fabricated will eliminate many of the restrictions of the autoclave experiments. In this new facility, an accurate simulation of anticipated CAES environments will be possible and changes in many of the fluid flow and structural properties of the sandstone will be continuously monitored during the elevated temperature experiments.

Chemical Responses

The groundwater present in the autoclaves was sampled during several experiments in which sandstone was reacted in liquid. Table 5 lists the determined groundwater chemical compositions at the conclusion of six experiments. The sampling method and analytical techniques are summarized in Appendix B. The major water/rock interaction implied by the fluid chemistry data (Table 5) is dissolution of quartz as a function of temperature and time. Figures 12 and 13 illustrate the changes in concentration with time of the elements Ca, Mg, Si, K, and Na in the fluid reacted with the Galesville sandstone at 50° and 300°C, respectively. The curves represent eye fits to the experimental data and are included only to identify general trends. The data for Si concentration in Table 5 illustrate the positive correlation between concentration

TABLE 5. Chemical Composition of Groundwater

Run No.	Core No.	T°C	P(bars)	Time (hours)	Elemental Concentrations (mg/l)								Solution pH
					Si	Ca	Mg	Na	K	Cr	Fe	Mn	
					INITIAL GROUNDWATER								
					<0.05	50	14	430	200	0	0	0	8.0
G-8	D-8	50	60	357	6	29	12	486	251	<0.05	0.2	<0.05	7.5
G-11	I-16	50	60	335	1.7	50	11.5	444	238	0.3	0.3	<0.05	8.12
G-16	D-13	200	120	238	76	25.5	0.2	447	223	<0.05	<0.05	<0.05	-
G-1	D-3	300	120	335	201	13	3.1	443	196	45	1.1	0.05	6.66
G-6	D-6	300	120	1457	193	0.6	<0.05	454	185	0.03	<0.05	<0.05	8.40
G-7 ⁽¹⁾	D-4	300	120	2392	206	0.5	<0.05	291	115	51.1	0.2	<0.05	8.08

(1) Composition determined from sample collected after 1968 hours

FIGURE 12. Elemental Fluid Concentrations (mg/l)
Versus Time for EXP G-11

FIGURE 13. Elemental Fluid Concentrations (mg/l)
Versus Time for EXP G-1

and temperature. The changes in concentration with time of Ca, Mg, Na, and K (Figures 12 and 13) are considered insignificant. However, the concentration of Si exhibits an initial rapid increase and approaches a constant value. In addition, the concentration of Si in the fluid at 300°C is significantly greater than at 50° or 200°C. The concentrations of two anionic species in the fluid were also determined but are considered insignificant. Typical concentrations determined for SO_4^{2-} at 50° and 300°C were 240 mg/l and 200 mg/l, respectively, and 138 mg/l for CO_3^{2-} at 300°C. The concentrations of SO_4^{2-} and CO_3^{2-} in the initial groundwater were 300 mg/l and 150 mg/l, respectively.

Since the Galesville sandstone is composed of nearly 100% quartz, no major chemical reactions, other than dissolution of quartz in the aqueous fluid, were anticipated in the pressure-temperature range of this study. Evidence from thin sections, photomicrographs, X-ray diffractograms, and X-ray fluorescence analysis of reacted Galesville sandstone indicates that no observable mineral alterations occurred, even at high temperatures. X-ray diffractograms of unreacted material and cores D-5 (EXP G-5) and D-3 (EXP G-1) suggest only α -quartz is present (Table 6). Bulk chemical analyses of reacted and unreacted Galesville sandstone obtained by X-ray fluorescence also imply no perceptible change in the bulk rock chemistry (Table 7). Comparison of Figures 2a and b with Figures 14a and b illustrates that the trace amounts of calcite present in the unreacted material have disappeared, presumably dissolved in the fluid phase. Although data for the fluid geochemistry imply quartz dissolution, concentrations of silicon in the fluid are so small at these experimental temperatures and test durations that there is no observed petrographic evidence for quartz dissolution.

TABLE 6. Comparative X-ray Diffraction Data for Unreacted Galesville Sandstone and Cores D-5 and D-3

Unreacted Galesville Sandstone		Reacted Core D-5(1)		Reacted Core D-3(2)		α -Quartz(3)	
dÅ	Relative Intensity	dÅ	Relative Intensity	dÅ	Relative Intensity	dÅ	Relative Intensity
4.23	30	4.25	25	4.23	24	4.26	35
3.336	100	3.336	100	3.327	100	3.343	100
2.533	2	2.540	2	-	-	-	-
2.447	15	2.453	14	2.453	13	2.458	12
2.268	14	2.274	11	2.274	13	2.282	12
2.225	9	2.231	7	2.231	11	2.237	6
2.120	13	2.120	10	2.120	11	2.128	9
2.079	2	2.079	2	-	-	-	-
1.971	8	1.975	6	1.975	10	1.980	6
1.809	25	1.812	22	1.816	10	1.817	17
1.665	10	1.668	7	1.668	10	1.672	7
1.654	4	1.657	4	-	-	1.659	3
1.604	2	1.601	2	-	-	1.608	1
1.596	2	-	-	-	-	-	-
1.536	18	1.538	15	1.538	15	1.541	15

(1) Run Conditions: 300°C, 120 bars, 360 hours in air-water vapor mixture

(2) Run Conditions: 300°C, 120 bars, 335 hours in liquid water

(3) Joint Committee of Powder Diffraction Standards (JCPDS) File Card No. 5-490.

TABLE 7. Comparative X-Ray Fluorescence Data⁽¹⁾ for Sandstone Cores D-4 and D-5

Oxide	Unreacted Galesville Sandstone wt%	Reacted Core D-4(2) wt%	Reacted Core D-5(3) wt%
SiO ₂	99.20	99.51	99.42
Al ₂ O ₃	0.19	0.11	0.09
TiO ₂	0.01	0.01	0.01
Fe ₂ O ₃ *	0.02	0.00	0.01
FeO*	0.03	0.00	0.01
MnO	0.00	0.00	0.00
CaO	0.05	0.01	0.00
MgO	0.12	0.11	0.11
K ₂ O	0.04	0.02	0.00
Na ₂ O	0.34	0.21	0.32
P ₂ O ₅	<u>0.01</u>	<u>0.02</u>	<u>0.03</u>
	100.01	100.00	100.00

(1) Analyses performed by the Department of Geology, Washington State University, Pullman, Washington. All analyses normalized on a volatile-free basis.

(2) Run Conditions: 300°C, 120 bars, 2392 hours in liquid water.

(3) Run Conditions: 300°C, 120 bars, 360 hours in air-water vapor mixture.

* Total iron recorded as 44% Fe₂O₃, 56% FeO.

a. Plane Polarized Light (50X)

b. Crossed Nicols (50X)

FIGURE 14. Galesville Sandstone After Heating to 300°C
in an Air-Water Vapor Mixture

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APPENDIX A

EXPERIMENTAL DATA FOR GALESVILLE SANDSTONE

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EXPERIMENTAL DATA FOR GALESVILLE SANDSTONE

Run No.	Core No.	T°C	P(bars)	Time (hours)	Liquid(L) Vapor(V) or Dry(D)	Change in K _{GAS} (%) ⁽²⁾	Change in K _{LIQUID} (%) ⁽³⁾	Change in Friability(%)	Change in Porosity(%)
G-1	D-3	300	120	335	L	↑96	↑36	↑171	-
G-2	D-1	50	60	338	V	↑6	↑10	↑24	-
G-4	D-2	300	120	338	V	↑76	↑38	↑185	-
G-5	D-5	300	120	360	V	↑31	↑78	↑222	-
G-6	D-6	300	120	1457	L	↑12	↑28	↑480	-
G-7	D-4	300	120	2392	L	↑65	↑73	↑474	-
G-8	D-8	50	60	357	L	↑17	↑37	↑29	-
G-9	D-7	50	60	1440	V	↑18	↑31	↑26	-
G-11	I-16	50	60	335	L	↑3	↑8	↑43	-
G-12	D-9	50	120	2904	V	↑11	↑51	↑61	-
G-13	D-10	300	120	330	D	↑3	0	↑27	-
G-14	D-11	50	120	330	D	↑2	↑4	↑41	-
G-15	D-12	200	120	339	V	↑331*	↑51	ND	-
G-16	D-13	200	120	238	L	↑100*	↑10	↑171	-
G-17	D-14	250-300 ⁽¹⁾	75-120		D	↑9	↑3	↑93	-
G-18	J-1	100	60	340	L	↑5	↑21	↑45	-
G-19	J-2	100	60	341	V	↑14	↑20	↑40	-
G-20	J-3	150	60	340	V	↑20	↑25	↑145	-
G-21	J-4	200	60	340	V	0	↑5	↑265	-
G-22	J-6	300	120	456	V	↑7	↑2	↑1456	↑18
G-22	J-10	300	120	456	V	↑38	↑1		↑15
G-23	J-5	300	120	411	L	↑296*	↑89	↑173	↑17

A.1

EXPERIMENTAL DATA FOR GALESVILLE SANDSTONE (contd)

Run No.	Core No.	T°C	P(bars)	Time (hours)	Liquid(L) Vapor(V) or Dry(D)	Change in K _{GAS} (%)(2)	Change in K _{LIQUID} (%)(3)	Change in Friability(%)	Change in Porosity(%)
G-23	J-8	300	120	411	L	ND	ND		↑53*
G-24	J-7	150	120	410	L	↓33	↓62	↑205	↑9
G-24	J-12	150	120	410	L	↑3	↓40		↑8
G-25	J-9	200-300 ⁽¹⁾	50-120	456	L	↑85	↑14	↑869	↑27
G-25	J-11	200-300 ⁽¹⁾	50-120	456	L	↓4	↓39		↑2
G-26	J-13	300	120	355	V	↓17	↓26	↑71	↑24
G-26	J-19	300	120	355	V	ND	ND		ND
G-27	J-14	50	120	1343	V	↓5	↓51	↑33	↑2
G-27	J-20	50	120	1343	V	↓5	↓14		0
G-28	J-15	50	120	335	V	↓11	↓16	↑2	↑1
G-28	J-21	50	120	335	V	↓21	↓68		↑1
G-29	J-16	175	120	334	D	↓3	0	↑10	↑3
G-29	J-22	175	120	334	D	↓9	↓16		↑2
G-30	J-17	300	120	1388	V	ND	ND	↑1129*	ND
G-30	J-23	300	120	1388	V	ND	ND		ND
G-31	J-18	175	120	844	V	↑1	↓13	↑67	↑3
G-31	J-24	175	120	844	V	↑9	↑40		↑4
G-32	J-25	175	120	844	V	↓6	↓1	↑36	↑6
G-32	J-26	175	120	844	V	↓10	↓24		↑5
G-10	J-27	300	86	1032	V	↑28	↓17	↑320	↑22
G-10	J-28	300	86	1032	V	ND	ND		ND
G-33	J-29	300	120	309	V	↑48	↑75	↑985	↑24
G-33	I-15	300	120	309	V	↓26	↓31		↑1

EXPERIMENTAL DATA FOR GALESVILLE SANDSTONE (contd)

Run No.	Core No.	T°C	P(bars)	Time (hours)	Liquid(L) Vapor(V) or Dry(D)	Change in K _{GAS} (%) ⁽²⁾	Change in K _{LIQUID} (%) ⁽³⁾	Change in Friability(%)	Change in Porosity(%)
G-34	I-17	175	120	309	V	+4	+6	+244	+1
G-34	I-18	175	120	309	V	0	+3		0
G-35	I-19	175	120	309	V	+5	+2	+365	0
G-35	I-20	175	120	309	V	+1	+13		0
G-36	K-1	50	120	336	V	+26	+26	+12	0
G-36	K-2	50	120	336	V	+7	+20		0
G-37	K-3	175	120	312	V	+5	+18	+98	+3
G-37	K-4	175	120	312	V	+2	+16		+3
G-38	K-5	300	120	312	V	+19	+21	+1093	+8
G-38	K-6	300	120	312	V	+76	+46		+7
G-39	K-7	175	120	312	L	+29	+39	+21	0
G-39	K-8	175	120	312	L	+82	+16		+4
G-40	K-9	300	120	511	L	+292*	+371*	+156	+25
G-40	K-10	300	120	511	L	ND	ND		ND
G-41	K-11	175	120	1004	V	+18	+33	+2	+6
G-41	K-12	175	120	1004	V	+8	+5		+4
G-42	K-13	300	120	1032	V	+27	+47	+274	+9
G-42	K-14	300	120	1032	V	+41	+51		+23

ND Not determined because of extensive core degradation

* Anomalous values caused by extensive core degradation

(1) Cycled 5 times between the two temperatures listed

(2) K_{GAS} = nitrogen gas permeability

(3) K_{LIQUID} = water permeability

APPENDIX B

ANALYTICAL METHODS AND PROCEDURES

APPENDIX B

ANALYTICAL METHODS AND PROCEDURES

This section summarizes the analytical methods and procedures used in this study. The physical properties of the samples measured were permeability, apparent porosity, and friability. The extraction procedure of fluid and gas samples, as well as the determination of the chemistry of these samples, is also discussed.

PERMEABILITY AND POROSITY MEASUREMENTS

Prior and subsequent to each experiment, gas and liquid permeabilities were measured using factory-calibrated Ruska gas and liquid permeameters. Gas permeabilities were measured using nitrogen on desaturated cores that were heated to 105°C in an oven for a minimum of 24 hours before the measurement. Liquid permeabilities were obtained from cores that had been evacuated and saturated with deaired, distilled water.

Pre- and post-test apparent porosities (ϕ) of sandstone cores were measured using a saturation and caliper technique (Franklin, 1972) and the equation:

$$\phi(\%) = P_v/B_v \times 100$$

where P_v and B_v are the pore and bulk core volumes, respectively. The bulk volume is calculated from the dimensions of the cylindrical core. The pore volume is determined from the difference in weight of the core after heating to 105°C for 24 hours, relative to the core weight after vacuum saturation with distilled water. Figures B.1 and B.2 illustrate the pre-test apparent porosity and permeability of the Galesville sandstone samples used in this experimental study.

FRIABILITY MEASUREMENT

In an effort to measure differences in friability of reacted and unreacted samples, a quantity related to the modulus of rupture was measured on small

FIGURE B.2. Pre-Test Gas and Liquid Permeabilities of the Galesville Sandstone

A simple equation for the friability index (FI) is proposed as the bulk volume (V_b) of the disc divided by the weight (w) required to rupture the disc or:

$$FI \equiv \frac{V_b}{w} = \frac{\pi r^2 t}{w}$$

For two samples of approximately the same bulk volume, the sample that is more friable will require a smaller weight to cause rupturing and, therefore, will also have a larger value for FI. Although measurement of this empirical index may not rigorously represent a value for friability, it may be used for qualitative comparisons. Comparisons of this type may be useful in evaluating integrity changes that occur when a reservoir rock is subjected to simulated CAES conditions.

MISCELLANEOUS ANALYTICAL TECHNIQUES

Several comparative techniques were utilized to illustrate the physical and chemical changes that occurred by reacting the sandstone with elevated temperature and pressure air and/or fluids. Photographs of entire samples were obtained for several cores reacted under various temperature, pressure and fluid conditions (Figures 6a, b, and 7b) in order to illustrate the physical degradation of the cores. To determine what, if any, mineralogical changes occurred, thin sections of several reacted cores were optically examined using a petrographic microscope. X-ray diffractograms were obtained for powdered samples. Figures 2a and b illustrate an area of unreacted Galesville sample in plane polarized light and with crossed nicols, respectively. Figures 14a and b illustrate similar samples that have been reacted at 300°C in an air-water vapor environment. Bulk chemical analyses of sandstone samples using X-ray fluorescence techniques were also obtained and are listed in Table 7.

FLUID AND GAS SAMPLING

The three types of fluid samples collected during the autoclave experiments were: 1) a liquid sample used for determining elemental concentrations (general analysis), 2) a liquid sample for dissolved CO₂ determination, and 3) a gas sample. The sampling apparatus for liquids and gas are illustrated in Figures B.3a and b.

FIGURE B.3. Sampling Apparatus for Extracting Liquid and Gas Samples

Liquids for general analysis were sampled at run temperature and pressure with an apparatus that includes a micropore filter and a cooling bath (Figure B.3a). Prior to collecting a liquid sample, the sampling valve was opened in order to drain a volume of fluid approximately equal to the volume contained in the sampling system. This procedure removed any residual fluid remaining in the cooling bath from the last sampling. Two fluid samples, each of approximately 10 ml, were then collected in capped plastic bottles by slowly opening the sample valve and allowing the fluid to condense in the cooling bath. After sampling, the system was repressurized with compressed gas to the initial pressure selected for the test. One sample was acidified by adding 5 drops of concentrated HCl. The pH of the other sample was measured and recorded and both samples were refrigerated at approximately 2°C. The shelf life for an acidified sample is approximately 1 month.

The same sampling apparatus used for collecting the general liquid sample (Figure B.3a) was also used to extract a liquid sample needed for dissolved CO_2 determination. Before sampling a 10 ml graduated cylinder is filled with 2 ml of 0.1 N NaOH. The fluid sample, about 10 ml, is collected in the graduated cylinder below the fluid level of the NaOH to assure that no dissolved CO_2 escapes. The cylinder is then capped with a rubber stopper (size 0) and the total volume of fluid in the cylinder is recorded. The shelf life for these samples is generally several days.

The sampling apparatus for gases is illustrated in Figure B.3b. After sufficient fluid has been drained from the sampling system, a clean and evacuated sample bomb is attached in place of the cooling bath. The sample valve and valve A (Figure B.3b) are opened and the flow of gas is monitored by noting the pressure drop on the gauge. When the bomb is full, valve A and the sampling valve are closed and the autoclave is repressurized. The gas samples were analyzed within 5 days.

PROCEDURE FOR LIQUID AND GAS ANALYSIS

The following analytical procedures were employed for the analysis of the liquid and gas samples.

pH Determination

The pH of the solution was determined by measuring the potential of the solution between glass and reference electrodes. A dip-type combination Orion electrode and meter equipped with an automatic thermistor temperature compensator was used. The general procedure involved: 1) recording a reading that had been stable for 1 min, or 2) the average reading if minor oscillations persisted.

Carbon Dioxide Determination

Total dissolved CO_2 was determined by the American Public Health Association (APHA) standard method 407B.

Element Concentrations

The elements Al, B, Ba, Ca, Fe, K, Li, Mg, Na, P, Sr, Ag, As, Cd, Co, Cr, Cu, Mn, Ni, Pb, Sb, Si, Sn, Ti, U and Zn were analyzed using a Jarrell-Ash Plasma Emission Spectrometer. This instrument is capable of simultaneously determining the concentrations of 31 elements using sample volumes of 5-10 ml per analysis. A background correction system on the spectrometer allows for correction of interelement interferences.

Anion Concentrations

Compressed gases used in the experiments were analyzed using a Hewlett-Packard Gas Chromatograph. The thermal conductivity detector allows for the determination of hydrogen, carbon dioxide, oxygen, nitrogen and methane. The detection limit is 1000 ppm except for hydrogen which is 10,000 ppm with a precision of $\pm 1\%$.

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