WATER ADSORPTION AT HIGH TEMPERATURE ON CORE SAMPLES FROM THE GEYSERS GEOTHERMAL FIELD

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(423) 574-4965

CONF-980441--

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ABSTRACT

The quantity of water retained by rock samples taken from three wells located in The Geysers geothermal field, California, was measured at 150, 200, and 250 °C as a function of steam pressure in the range $0.00 \leq p/p_0 \leq 0.98$, where p_0 is the saturated water vapor pressure. Both adsorption and desorption runs were made in order to investigate the extent of the hysteresis. Additionally, low temperature gas adsorption analyses were made on the same rock samples. Mercury intrusion porosimetry was also used to obtain similar information extending to very large pores (macropores). A qualitative correlation was found between the surface properties obtained from nitrogen adsorption and the mineralogical and petrological characteristics of the solids. However, there was no direct correlation between BET specific surface areas and the capacity of the rocks for water adsorption at high temperatures. The hysteresis decreased significantly at 250 °C. The results indicate that multilayer adsorption, rather than capillary condensation, is the dominant water storage mechanism at high temperatures.

that adsorption can control the response of the reservoir to both production of steam and injection of water in the wells where the pressure falls significantly below its original level in the undisturbed reservoir.

The goal of this study was to expand our understanding of the adsorption/desorption processes occurring in geothermal reservoir rocks. The desired outcome is an improvement of the efficiency of the recovery of geothermal energy through more accurate inputs to both simple empirical reservoir performance forecasting schemes and comprehensive reservoir models, so that the life of the reservoir can be sustained for the most economical exploitation of the resource.

The reservoir pressure affects directly the economics of geothermal energy production. The rates of both pressure and flow rate decline during production are used as an indicator of the size of the resource and the expected life and performance of the reservoir (Sanyal *et al.*, 1992; Enezy, 1992). It was determined that the initial pressure at sea level in the central part of The Geysers reservoir was 35.4 bars (Lippmann *et al.*, 1978), which corresponds to a boiling temperature of 243.3 °C. Since this temperature is close to the measured well temperatures, the reservoir must contain some amount of free water present in wide pores and fractures (Pruess and O'Sullivan, 1992). The steam produced by a vapor-dominated reservoir initially comes from the evaporation of the excess liquid water. After the reserves of this bulk liquid are, at least locally, exhausted, the water retained in smaller pores and adsorbed on the surfaces starts to evaporate, and the pressure starts to decline. The process of depletion of a steam-dominated reservoir, assuming thermodynamic equilibrium over all the volume of a closed system (i.e. slow production rate), is shown schematically in Figure 1.

INTRODUCTION

The decline in reservoir pressures observed at The Geysers after a period of rapid development (Barker, 1992) prompted an increase in the research of the mechanisms of water storage. There is no doubt that at various locations in the reservoir and at various stages in production history, water can be present as bulk liquid, steam, capillary condensate and surface adsorbate. For the purposes of performance modeling and forecasting it is important to know the proportions between these storage mechanisms, since the expected reservoir responses to the production of geothermal steam would be different. It is expected

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Figure 1. Equilibrium pressure decline during depletion of a vapor-dominated geothermal reservoir.

The actual, dynamic response of the reservoir may not follow closely the adsorption isotherm if the production rate is high compared to the relaxation rate of the reservoir, or if the reservoir is being recharged. The flat (nearly horizontal) part of the solid curve in Figure 1 indicates that it would be rather difficult to accurately estimate the amount of water present initially in the reservoir by measuring the reservoir equilibrium pressure decline. At this stage, the withdrawal of even large amounts of water is associated with an immeasurably small (long-term) pressure drop. In the later stages of reservoir depletion, the amount of water left in the reservoir can be determined in principle from reservoir pressure data, if the water retention capacity as a function of pressure (i.e. an adsorption isotherm) and the reservoir size are known.

The shapes of the pressure decline curves can be significantly different from that shown in Figure 1, in particular in their high-pressure portions, where a hysteresis loop usually occurs. The particular shape depends on the properties of the reservoir rocks, such as mineral compositions, porosities, pore size distributions, and the predominant pore shapes. It can also depend on the way the pores are filled or emptied and on the temperature. Generally, adsorption, or any other mechanism of water pressure lowering, makes the pressure decline more gradual. Since steam pressure is the

driving force for further depletion, pressure lowering acts as a mechanism delaying a complete dry-out. Adsorption properties of geothermal reservoir rocks have the greatest impact on the behavior of vapor-dominated geothermal reservoirs, with the steam pressure substantially lower than the saturation pressure, but which still contain a considerable amount of water held by the solid-liquid interactions. The knowledge of adsorption characteristics can also help in better understanding of the reservoir's response to water injection. The time needed for the injected water to reach the production wells may be longer with adsorption since the injected water will at first readsorb on the surface of fractured rocks. The amount of water needed during injection may be different from the amount of water previously withdrawn, due to hysteresis, and to different conditions at the injection well.

Possible mechanisms of water storage based on solid-fluid interactions include micropore filling, cooperative adsorption (involving interactions between adsorbate molecules), monolayer and multilayer adsorption, capillary condensation, and interactions similar in varying degrees to chemical bonding. Since physical models developed to describe these phenomena are different, it would be desirable to determine the prevailing water storage mechanism at the actual reservoir temperature, so that an adequate model can be developed.

One of the reasons for measuring adsorption isotherms over a range of temperatures is to be able to distinguish between capillary condensation and multilayer adsorption. This distinction is possible because multilayer adsorption isotherms as a function of the relative pressure p/p_0 are expected to change only very little with temperature, while capillary condensation is temperature dependent. According to the established simplified model of multilayer physical adsorption, represented most often by the BET equation (Brunauer *et al.*, 1938), a single layer of adsorbed molecules is sufficient to screen the subsequent layers from interactions with the solid surface. The creation of an adsorption layer and condensation into bulk liquid are then thermodynamically similar processes. Since the associated enthalpy changes and volumetric properties are nearly the same, p/p_0 at a particular extent of adsorption is expected to remain constant with temperature.

In contrast to multilayer adsorption, the saturated vapor pressure over capillary condensate should be strongly affected by temperature as predicted by the Kelvin equation (Defay *et al.*, 1966). In fact, the surface tension of any fluid decreases to zero as the temperature approaches the liquid-vapor critical point. However, at a given temperature, the experimental relationship between relative pressure and the amount of water retained can be described equally well either in terms of the Kelvin equation or an adsorption equation like the BET adsorption isotherm. Hsieh and Ramey (1978) noted that experimental data are sometimes described in the literature in terms of capillary condensation, using the Kelvin equation, at conditions where the capillary condensation model is no longer valid. They suggested that the multilayer adsorption model (and the BET equation) would provide a more appropriate description. Even at conditions where capillary condensation certainly occurs, a substantial amount of the adsorbate may be more properly described in terms of the multilayer adsorption concept.

Studies of high temperature water adsorption in porous materials in general and rocks in particular have been rather scarce (Shang *et al.*, 1995). The temperatures did not exceed 150 °C, with most of the reported measurements done at 120 °C and below. Recently, Bertani *et al.* (1996) reported adsorption (but not desorption) isotherms on several geothermal reservoir rocks at temperatures between 170 °C and 200 °C. They found no temperature dependence of the amount of water adsorbed in this temperature range. Shang *et al.* (1995) found an increase in the amount of water adsorbed as the temperature increased from 80 °C to 130 °C. This increase was more pronounced at high relative pressures.

EXPERIMENTAL

The measurements were performed on core samples of rocks taken from the productive steam reservoir at The Geysers. Well numbers and approximate depths were as follows:

- MLM-3, 4336 - 4336.3 ft (1322 m)
- Prati State 12, 6261.7 - 6261.8 ft (1909 m)
- NEGU-17, 8530 - 8530.5 ft (2600 m).

The rocks were fine to medium grained graywacke, which had been intensely hydrothermally altered

during the time the reservoir was liquid-dominated, and the calcite, originally present in veins, was largely replaced by calc-silicates (Hulen *et al.*, 1991, 1992). Hydrothermal mineralization, alteration, mineral dissolution, and fracturing are the causes for the significant porosity and permeability of the reservoir rocks in comparison with the caprock (Gunderson, 1992).

The mineral compositions of the metagraywacke samples taken from the three wells were more diverse than expected. The percentages of the component minerals are given in Table 1.

Table 1. Mineral percentages of the metagraywacke samples (Hulen, 1997).

	MLM-3	NEGU-17	PS-12
quartz	38	19	45
albite	30	16	21
chlorite	14	31	13
illite	11	14	4
epidote	1	2	11
adularia	traces	8	3
sphene + anatase	2	2	2
calcite	2	-	traces
ferroxinite	-	2	-
organic	2	6	1

All three core samples contained traces of actinolite, apatite, and zircon, and traces of rutile were found in the MLM-3 and NEGU-17 cores. Additionally, prehnite was found in the MLM-3 sample, and pyrite, garnet, and clinopyroxene were found in the Prati-State sample. The compositions shown in Table 1 indicate that the fine grained NEGU-17 metagraywacke differs significantly from the other two more coarse grained samples in the relative amounts of quartz, albite, and chlorite. NEGU-17 also contains much more organic matter. The core from the Prati State 12 well contains more quartz and epidote but less illite than either NEGU-17 or MLM-3.

The rocks were crushed and screened into three fractions with the following grain sizes: 2.00 - 4.25 mm ('coarse'), 0.355 - 2.00 mm ('medium'), and 0 - 0.355 mm ('fine'). Two aliquots of each of the medium fractions were loaded in the autoclave in order to evaluate possible random differences due to the heterogeneity of the samples.

The ORNL high-temperature isopiestic apparatus has been described previously. (Holmes *et al.*, 1978; Gruskiewicz *et al.*, 1996). Briefly, the samples were placed inside a high-pressure, high-temperature autoclave in titanium pans fitting in holes in a disk which was rotated by the operator. The pans were positioned in turn on the torsion-suspension electromagnetic balance and weighed *in situ* by adjusting the electric current through the balance coil. Since the weighings were made by comparison with a set of standard weights placed inside the pressure vessel together with the samples, the method's susceptibility to significant systematic errors was greatly reduced.

In addition to the high temperature water adsorption experiments, nitrogen adsorption and desorption measurements were made across the whole pressure range for selected samples. Multipoint BET specific surface area analyses and pore size distributions were obtained from these data. Adsorption results were supplemented with mercury intrusion tests. Mercury pressure scans extended from 0.11 bar to 4.14 kbar, providing a nominal pore diameter range from 114 μm to 3 nm.

RESULTS

Since the properties of the rocks vary significantly with depth, it is difficult to compare directly the results obtained on different samples, even if they come from the same well. In this work, the samples are named by the well numbers, but they do not represent the entire respective well cores. There was very good agreement between the present results and those obtained by Satik and Horne (1995) where a direct comparison could be made for an adsorption isotherm (Gruskiewicz *et al.* 1996). However, the agreement of the desorption isotherms was not satisfactory, as the hysteresis loops obtained in this work were closed or nearly closed, while extensive low pressure hysteresis and irreversible water retention were observed by Satik and Horne (1995) and Shang *et al.* (1995) on all their samples.

Figure 2 shows the experimental results for water sorption on the medium fractions of MLM-3, NEGU-17 and Prati-State 12 cores, respectively, at 150, 200 and 250 $^{\circ}\text{C}$.

In all the figures open symbols represent experimental results on the adsorption branches, and filled symbols represent desorption branches.

Figure 2. Adsorption and desorption isotherms of water on the medium grain size fractions of the core samples; a) MLM-3, b) NEGU-17, c) Prati State 12. Offsets of either 1 and 2 mg/g, or 2 and 4 mg/g, were added to the 200 and 250 $^{\circ}\text{C}$ isotherms to prevent overlapping.

The following features of physical adsorption of water on the rocks investigated can be seen in Figure 2:

- There is essentially no temperature dependence of the adsorption branches;
- As the temperature increases, the hysteresis loop shrinks in both the amount of the adsorbate and the pressure range (this is most clear in the NEGU-17 isotherms);
- Water adsorption is reversible (the mass of the sample in vacuum after an adsorption-desorption cycle remains the same), but;
- The hysteresis loops persist to very low pressures in the case of MLM-3 and Prati-State 12, while more distinct closure points at an intermediate pressure are present for NEGU-17. The closure points for the 'main' hysteresis loop due to capillary condensation are also visible in MLM-3 and Prati-State 12 isotherms.

While on the adsorption branch the water is largely multilayer adsorbate, in principle all of the excess retention on the desorption branch is due to capillary condensation. This explains why the desorption branch is temperature dependent, while the adsorption branch is nearly temperature independent.

The Kelvin equation accounts for the observed behavior of the hysteresis loop. An analysis of the Kelvin equation indicates that the relation between the relative pressure p/p_0 and the temperature at a constant pore diameter is approximately linear. The experimentally determined hysteresis loop inception points at 150 °C and 200 °C for NEGU-17 are consistent with corresponding relative pressures calculated from the Kelvin equation. At 250 °C the hysteresis loops are narrow, and the inception points are not well defined. At this temperature the hysteresis is present only on the steep part of the isotherm and it is significantly reduced. (Figure 2). It appears that only a small fraction of the water is adsorbed as capillary condensate at 250 °C, close to the main reservoir temperature at The Geysers.

The causes for the low-pressure hysteresis (Figure 2a and 2c) on very heterogeneous rock samples are difficult to identify with certainty. It has been known (Arnell and McDermott, 1957) that swelling of some sorbents, caused by the presence of adsorbates, can lead to low-temperature

hysteresis as new surface sites open up during desorption. Sorbent swelling occurs to a detectable extent even in materials with a rigid structure. Low-pressure hysteresis of water on calcite was observed by Gregg and Gammage (1972) and attributed to the penetration of water deep into molecular-size crevices between adjacent surfaces of the lamellar solid, from where it can escape only very slowly. Finally, dissolution of the solid in the adsorbent can probably cause both positive and negative hysteresis, depending on whether new adsorption sites are created or destroyed. The components that are most likely responsible for the low-pressure hysteresis observed in the altered graywacke are chlorite and other minerals related to clays, or calcite.

All the nitrogen adsorption isotherms obtained in this work had well defined hysteresis loops closing at $p/p_0 \approx 0.45$, a rather typical value for nitrogen at temperatures close to its boiling point. Figure 3 shows nitrogen adsorption (bold lines) and desorption (regular lines) isotherms for medium grain size samples of the three cores investigated.

The following characteristics of the samples can be seen in the above diagram:

- There are large differences in the amounts adsorbed by the three cores (the ratios MLM-3 : NEGU-17 : Prati-State 12 at $p/p_0 = 0.4$ are 1 :

Figure 3. Adsorption and desorption isotherms of nitrogen at 77 K on the medium grain size fractions of the three cores investigated.

2.56 : 0.29; and the BET specific surface areas are 1.31, 4.06, and 0.36 m²/g, respectively);

- NEGU-17 metagraywacke has the largest specific surface area and its pore area distribution is shifted towards smaller pores compared to the other two cores;
- The MLM-3 metagraywacke adsorbs the largest amount of nitrogen at high pressures, which is likely due to the presence of more macropores.

Figure 4 shows water adsorption isotherms of the three cores (averages of the medium and coarse fractions) at 200 °C. The following characteristics of water adsorption, in contrast to nitrogen adsorption, are evident from a comparison of Figures 3 and 4:

Figure 4. Average adsorption and desorption isotherms of water at 200 °C for the three cores investigated.

- The increase in the amount of the adsorbate in the very low-pressure range, which corresponds to monolayer formation, is small in comparison with the increase in the intermediate pressure range when the subsequent layers are formed. This indicates that solid-fluid interactions are relatively weaker (as compared to the fluid-fluid interactions) for water than for nitrogen. As a consequence, molecular-size pores, which may be wide enough to admit either N₂ or H₂O molecules, are filled more easily with nitrogen than with water. Water molecules can minimize their energy by forming hydrogen bonds and through strong dipolar interactions between each other. Relatively weak

dispersion forces in nitrogen cannot compete with the interactions with the solid surface.

- In contrast to nitrogen adsorption, the amounts of water retained by the three cores at low relative pressures are similar (the ratio MLM-3 : NEGU-17 : Prati-State 12 at $p/p_0 = 0.4$ is 1 : 0.90 : 0.74;
- The NEGU-17 isotherm has a distinctly different shape from the other two isotherms.

Figures 3 and 4 show that the standard BET surface areas cannot be used directly to predict relative capacities of various rocks for water adsorption, even if these rocks are mineralogically similar. There is convincing evidence, obtained by all three methods (high temperature water adsorption, low temperature nitrogen adsorption, and ambient temperature mercury intrusion), indicating that NEGU-17 metagraywacke has significantly more micropores and also fewer very wide macropores than either MLM-3 or Prati-State 12. Pore-volume distributions calculated from both nitrogen and water adsorption isotherms show a distinct difference between NEGU-17 and the other two cores. Average pore diameters (27.2, 6.6, and 28.6 nm for MLM-3, NEGU-17, and Prati-State 12, respectively) calculated from nitrogen adsorption BET surface areas and total pore volumes also support the conclusion that NEGU-17 metagraywacke has a finer pore structure than the other two cores. The Langmuir specific surface area analysis yields even lower average pore diameter for NEGU-17 (4.7 nm).

Both nitrogen adsorption and mercury intrusion results indicated that changes in the pore structure had taken place during the high temperature adsorption experiments. Pore volume distributions showed a shift towards wider pore sizes in NEGU-17 and MLM-3 samples, while Prati-State 12 pore volume distribution remained unchanged. Since the smallest pores are most affected by changes occurring on the surface or by solid dissolution and subsequent deposition, the Prati-State 12 metagraywacke apparently has by far the fewest very narrow pores. Dissolution of the finest crystals and sealing of the finest pores was also indicated by the decreases of 20 to 30 per cent in the BET specific surface areas and increases in the average pore sizes observed for all the cases where such comparisons were made. In a sample of silica gel the fine pore structure was nearly completely destroyed upon exposure to steam even at 150 °C, and the specific surface area decreased from 542

m²/g by the BET method, (782 m²/g by the Langmuir method) to 3.0 m²/g (BET). This was likely due to the dissolution of SiO₂ in adsorbed water. Similar effects can be expected to occur, to a lesser extent, in reservoir rocks containing silica and other minerals that become increasingly soluble in water at elevated temperatures. Since there is a significant random variability between the samples, obtaining definitive evidence for these changes would require more tests performed on exactly the same aliquots before and after high temperature adsorption measurements.

Total pore volumes (and hence total porosities) are difficult to measure unambiguously by either adsorption or mercury intrusion. The adsorption isotherms of the rock samples were characterized by a steep increase of the amount of water retained with the increase in pressure towards saturation, so that the isotherms were apparently asymptotic to the $p/p_0=1$ axis. Such behavior is not universal in porous materials, since often a plateau at high relative pressures is found. The lack of a plateau indicates that the pore volume distribution is relatively even in the upper end of the pore size range, and there is no distinct upper pore size limit. Such shapes of adsorption-desorption isotherms are often observed with aggregates of plate-like particles or sheets giving rise to slit-shaped pores, and they seem to be consistent with porosity caused by fracturing and hydrothermal rock alteration.

The porosity results (8.4, 10.9 and 12.3 % in the sequence NEGU-17, Prati-State 12, and MLM-3) were significantly higher than those reported by Satik *et al.* (1996) for similar materials (up to 5.5 per cent for MLM-3 metagraywacke) or by Gunderson (1990) (1.1 to 5.6 per cent for NEGU-17 core of approximately the same depth as in this work). Since the widest pores are randomly distributed and may have a relatively large volume, total pore volume determinations are subject to a significant random error. On the other hand, the average density of the rocks can be determined fairly accurately (it was 2.75-2.78 g/cm³ for the samples included in this work, with a maximum error of about 2 per cent). The main difficulty in finding porosities by the mercury intrusion method seems to lie in measuring the total volumes of the porous solids. The differences in mercury intrusion procedures (immersion pressure), sample grain sizes, and random differences between the

samples can significantly impact total porosity results.

The differences in adsorption properties between MLM-3, NEGU-17, and Prati-State 12 cores were qualitatively consistent with the differences in mineral compositions (Table 1). Enhanced microporosity of the NEGU-17 samples was concomitant with more than double the content of chlorite and the largest content of organic matter. Chlorite is a mineral related chemically to montmorillonite and other clay minerals, which form, charged molecular layers able to interact with each other and to form stable structures involving water. The strength of such interactions is intermediate between that of dispersion forces and typical chemical bonding. On the other hand, quartz and feldspars, which constitute more than 65 per cent of the MLM-3 and Prati-State 12 samples, but only 45 per cent of the NEGU-17 samples, have more rigid, three dimensional structures, which do not accommodate water or small cations as easily. The highly carbonized organic matter found in the NEGU-17 samples might contribute to the observed microporosity and hydrophobic adsorption, in a manner similar to that observed in water adsorption on charcoal (Gregg and Sing 1982, p. 262). More detailed study focusing on the properties of the constituent minerals could lead to more precise assignments of the contributions to the observed adsorption behavior, which would be useful for predicting adsorption properties of rocks.

ACKNOWLEDGEMENT

Research sponsored by the Office of Geothermal Technology (Office of Energy Efficiency and Renewable Energy), and the Office of Basic Energy Sciences, (Division of Chemical Sciences), US Department of Energy, under contract DE-AC05-96OR22464 at Oak Ridge National Laboratory operated by Lockheed Martin Energy Research Corporation.

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