

HIGH-TEMPERATURE WATER ADSORPTION ON GEOTHERMAL RESERVOIR ROCKS

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ABSTRACT

The mechanism of water storage in geothermal reservoirs was investigated by direct measurements of water adsorption on reservoir rocks under simulated reservoir conditions. Since the capacity for water adsorption was found to vary widely across the reservoir, multiple measurements are necessary to map water retention capacities and build reliable models. Isotherms of water adsorption on well cores from The Geysers (California, USA) and Awibengkok (West Java, Indonesia) geothermal fields were obtained at 150, 200, and 250 °C in the whole pressure range from vacuum to over 98% of the water saturation pressure at each temperature. Both adsorption (increasing pressure) and desorption (decreasing pressure) runs were made in order to investigate the adsorption hysteresis. The amount of water adsorbed was determined by direct *in situ* weighing of solid samples using the Oak Ridge National Laboratory isopiestic apparatus. Additionally, adsorption and desorption isotherms of nitrogen at its boiling point (−196 °C) were obtained for the same samples. The low temperature adsorption data were used to determine BET specific surface areas, pore volume distributions with respect to pore sizes, and porosities. Mercury intrusion porosimetry was also used to obtain additional information about pore size distributions extending to very large pores (macropores) and fractures. The results indicated that the dominant water storage mechanism at high temperatures is better described as multilayer adsorption rather than capillary condensation. The adsorption branches of the isotherms were essentially temperature independent in the range investigated, but the amounts of water retained on the desorption branches decreased with increasing temperature. At 250 °C capillary condensate exists only at pressures very close to the saturation pressure for bulk water. The remaining hysteresis loop is significantly reduced in area in comparison with its size at temperatures up to 150 °C. Water adsorption capacities are generally higher for the AWI 1-2 core than for any previously investigated well at The Geysers. One of the AWI 1-2 samples adsorbed more water at $p/p_0 = 0.5$ (12 mg/g) than any other geothermal well core sample, exceeding even water adsorption on pure powdered chlorite. The capacity of the rocks for water adsorption depends on both mineral compositions and pore system features. The BET specific surface areas for nitrogen adsorption were found to be a poor predictor of the capacity of geothermal rocks for water adsorption, since water adsorption on polar adsorbents involves not only weak dispersion forces, but also electrostatic and chemical interactions with the solid.

1. INTRODUCTION

The functioning of geothermal reservoirs depends on the presence of voids, fractures and pores in a wide range of dimensions extending over several decades. Conduits as large

as several centimeters are important for efficient mass, and consequently, heat transfer, while all the pores down to the molecular dimensions (less than 1 nm) may contribute significantly to water storage. The total amount of water retained by a solid is usually a sum of the fractions held in condensed state by different mechanisms of lowering vapor pressure. These include capillarity, responsible for creation of curved interfaces (menisci), multilayer adsorption occurring even on flat surfaces, micropore filling mechanism, and chemical bonding with the solid. In the last case the adsorbed water can form a well-defined surface layer, or be a part of the crystalline structure of the solid, for example in the case of clay minerals and zeolites. Understanding the mechanism of water storage in vapor-dominated geothermal reservoirs requires characterization of the pore structure in combination with chemical properties of the mineral components. Dissolution and deposition of solids carried with geothermal fluids provides for an abundance of varieties of rocks with different permeabilities and water storage capacities. Adsorption properties can depend strongly on the content of a minor component and the extent of geothermal alteration.

Recently, (Gruszkiewicz *et al.*, 1996) measurements of water adsorption on geothermal rocks were made for the first time at temperatures and pressures close to the conditions in a typical vapor-dominated reservoir (up to 250 °C). A few samples from The Geysers field were investigated. The results showed a correlation between water adsorption capacities and mineral compositions of the rocks. The relative importance of capillary condensation and multilayer adsorption was inferred from the temperature dependence of the desorption isotherms. It was found that at high temperature multilayer adsorption is the dominant water storage mechanism, while capillary condensate exists only at pressures very close to saturation ($p/p_0 > 0.9$). A much larger number of samples (36) from the same geothermal system were previously investigated by Shang *et al.* (1995) and Satik *et al.* (1996) at temperatures up to 120 - 130 °C. Bertani *et al.* (1998) measured water adsorption (but not desorption) on several samples of cuttings from different wells in the Monteverdi, geothermal field, Italy, at temperatures from 170 to 200 °C.

The present work is a part of the Awibengkok, Indonesia, Research Project focused on the continuous core provided for the U.S. Department of Energy's Office of Geothermal Technology by Unocal Geothermal Indonesia. The AWI 1-2 core is 1.1 km long, and nearly 100% complete (Hulen and Anderson, 1998). It is expected to provide a great insight into the properties of not only the Awibengkok field, but also other similar systems worldwide. Adsorption data for the Awibengkok field, together with the information already available on The Geysers, will be used in improved reservoir models. Reliable models based on a better understanding of water storage in geothermal reservoirs will contribute to performance forecasting, developing reservoir stimulation technology through effective and economical injection, and to analyzing chemical tracer data.

2. EXPERIMENTAL

2.1 Methods and procedures

The experimental approach adopted in this work, based on *in situ* weighing of solid samples, was described previously (Gruszkiewicz *et al.*, 1996, 1999; Holmes *et al.*, 1978). The apparatus was originally designed for measuring isopiestic molalities of aqueous solutions, but the principle of direct weighing has been employed since the beginning of the century for measuring adsorption of gases on solids at temperatures close to ambient.

The samples are placed in titanium pans inside a stainless steel autoclave with a sapphire window. A torsion-suspension electromagnetic balance with a platinum mirror attached to its beam is installed inside the autoclave. After sealing the autoclave it is possible to rotate the sample holder to put each of the twenty sample pans on the dish of the balance. The electric current through the balance coil is automatically adjusted by an electronic controller to equilibrate the balance beam. This current, proportional to the apparent mass of the sample pan, is measured. The position of the balance beam is detected by two photoelements placed on the path of the light reflected from the balance. The apparent mass is corrected for the effect of buoyancy in steam.

A cycle of adsorption measurements starts with drying the samples in vacuum while monitoring their weights, until the daily mass decrease is less than the resolution of the measurement (< 0.1 mg/g). The masses of dry solids are used to obtain all the water adsorption values in milligrams of water retained per gram of solid. Next, the densities of the samples are determined by weighing them in argon at pressures between 20 and 40 bar. It is assumed that the amount of argon adsorbed on the geothermal rock samples is negligible. This is indicated by the independence of the obtained densities from the pressure of argon. If this assumption is valid, and provided the samples don't contain residual water, the accuracy of the density determination is better than ± 0.01 g/cm³. However, some samples with very large active surfaces, like for example zeolites, may adsorb or entrap significant amounts of argon even at 150 °C. In such cases the solid densities needed for the buoyancy corrections must be determined by a different method.

Deionized water, freshly boiled and purged with helium, is admitted to the autoclave in steps, using a chromatography pump. Usually one day is allowed for equilibration, after which all the samples and the standards are weighed one to three times. After reaching the saturation pressure at the current temperature, the water is withdrawn in steps from the autoclave to obtain the desorption isotherm. After reaching vacuum the samples are weighed repeatedly again to obtain the mass of dry solid, which should normally be the same as that obtained before the cycle, unless the solid contained volatile components which can be removed by steam, or an irreversible chemical reaction with water took place. The overall uncertainty of the measurement is better than 0.1 mg/g.

The method used most often for obtaining adsorption isotherms is based on measurement of gas or vapor pressure in calibrated volumes, and using the mass balance to obtain

the amount adsorbed. Careful calibration of all spaces, good temperature control, and elimination of all leaks are required for accurate results. This method is perfected in automated commercial instruments designed for nitrogen or krypton adsorption measurements at liquid nitrogen temperature. In this work such devices were used to obtain full nitrogen adsorption/desorption isotherms and for multiple point BET surface area determinations (Micromeritics, Norcross, Georgia).

The direct weighing method has several advantages over the static volumetric method based on pressure measurement:

- Significant systematic errors are very unlikely. The mass of the sample is determined in comparison with standard weights. The result is not sensitive to errors in pressure measurement or small leaks.
- With the direct weighing method all the points on an isotherm are measured independently, so the errors of earlier obtained data points do not affect the following points. This allows for monitoring the weight of the sample for long times. With the volumetric method the amount adsorbed is accumulated during an adsorption/desorption cycle, so that the uncertainty increases.

Mercury intrusion tests were performed by Micromeritics using their instruments. The pressure range was from 0.039 bar to 414 MPa, corresponding to the pore size range from 0.3 nm to 3 nm

2.2 Samples

The fragments of the AWI 1-2 well core were obtained from the Energy and Geoscience Institute, Salt Lake City, Utah, USA. The samples covered the depth range from 871 to 1674 m. Two samples from The Geysers geothermal field, wells MLM-3 (1325 m), and CA1862-4 (1361 m), previously investigated by Satik *et al.* (1996), were obtained from the Stanford Geothermal Program, Stanford University, Stanford, California, USA. These samples were included in order to verify adsorption results obtained by two different methods, and in particular the hysteresis loop shapes.

The samples were prepared so as to obtain representative aliquots for the core fragments and to avoid unnecessary segregation of the components. Each of the AWI 1-2 core fragments (7 to 10 cm in length) was cut in two parts along the length of the well. One part was crushed to pass entirely through a 4.75 mm standard sieve (No. 4), so that no large grain-size fraction was produced. A No. 35 standard sieve was used to separate grains smaller than 0.5 mm. The amount of this fine fraction was relatively small, and its density (for the sample AWI 1-2 11) was only slightly higher than that of the main fraction (2.846 vs. 2.794 g/cm³). The same crushing and sieving procedure was applied to both Stanford samples, which were supplied in chunks larger than 4.75 mm.

The fractions between 0.5 and 4.75 mm were used for the experiment, but the fine fraction of one Awibengkok sample (AWI 1-2 11F) was also included in order to verify the conclusions of the previous work regarding the apparent irreversible adsorption by the fine fractions (Gruszkiewicz *et al.*, 1998, in print).

Pure Brazilian quartz, with grain sizes from 45 to 75 μm , was also included for reference. The results of density determination for this sample confirmed the accuracy of the method. The density obtained for this sample at 150 $^{\circ}\text{C}$ was 2.640 g/cm^3 , while the calculated value is 2.635 g/cm^3 .

3. RESULTS AND DISCUSSION

After the present work is completed, AWI 1-2 will become the geothermal well with the best-characterized adsorption properties. Although usually no more than one to three samples from the same well core were used previously for adsorption measurements, it became clear that adsorption properties are not correlated with depth in a simple manner, and they may vary widely with the lithology and the degree of alteration, even for samples taken from similar depths. Shang *et al.* (1994) measured adsorption isotherms at the temperatures up to 120 $^{\circ}\text{C}$ for Prati State 12 well cuttings taken from ten different depths between 1463 and 2865 m. Water adsorption capacities were not correlated with depth. The results indicated a correlation between nitrogen adsorption BET surface areas and water adsorption capacities. However, other results on mineralogically different materials provided many examples that in general do not support such a correlation. Two samples investigated by Satik *et al.* (1996) provide a good example. BET specific surface areas for two samples from The Geysers geothermal field, Sulphur Bank 15-D (west central Geysers, depth 472 m) and Aidlin-8 (northwestern Geysers, depth 3165 m) were 1.91 and 0.071 m^2/g , while the amounts of water adsorbed at $p/p_0 = 0.5$ were 0.500 and 1.74 mg/g , respectively. Examples of larger water adsorption capacity for samples with smaller BET surface areas can be also found in our present and recent results (Gruszkiewicz *et al.*, in press).

The well depths, densities (obtained by weighing samples in argon and in vacuum), BET specific surface areas (from multipoint nitrogen adsorption at -196°C), the amounts of water adsorbed at $p/p_0 = 0.5$, and porosities (from mercury intrusion) for all the samples included in this work are collected in Table 1. It is interesting to compare the adsorption of nitrogen and water in terms of the coefficients k also included in Table 1. Coefficients k are the ratios of the number of molecules (moles) of water adsorbed at a given relative pressure p/p_0 per molecule (mole) of nitrogen adsorbed as a monolayer by the same amount of solid, divided by the same ratio for nonporous, fully hydroxylated silica. To calculate the amounts of nitrogen from the BET surface areas, the most often used value of 0.162 nm^2 per nitrogen molecule, or $9.76 \cdot 10^4$ m^2/mol was used. The values of p/p_0 were chosen as 0.1 and 0.5. For nonporous silica $k_{0.5}$ is very close to unity and can be treated as a reference (one mole of either nitrogen at 77 K forming a monomolecular layer or water at ambient and higher temperatures at $p/p_0 = 0.5$ would occupy about 0.1 km^2 of the physical surface area of SiO_2). At $p/p_0 = 0.1$ for the nonporous silica $k_{0.1} = 0.33$. The values of $k_{0.1}$ should represent well the affinity of water to the mineral surfaces, while $k_{0.5}$ may also reflect the features of the pore system. Values of k close to unity indicate purely physical adsorption, due to the weak dispersion forces, while values less than one indicate the presence of surfaces more hydrophobic than silica (for example carbon). Very high values of k indicate minerals reacting with water specifically, such as clays and zeolites.

The difference between $k_{0.1}$ and $k_{0.5}$ reflects the role of multilayer adsorption. If k decreases with relative pressure, multilayer adsorption is less important. Such behavior of k points to processes obeying the Langmuir isotherm for chemisorption such as adsorption of water on zeolites. On the other hand, increasing values of k indicate that water may create closely spaced, rapidly growing clusters on the surface.

For the samples Aidlin-8, and Sulphur Bank 15-D from the work of Satik *et al.* (1996) the values of $k_{0.5}$ are 1.41 and 133 and they represent the extreme values for this parameter. The Aidlin-8 sample was rich in argillite while the Sulphur Bank sample was a coarse-grained metagraywacke, with a small fraction of matrix material. For geothermal rocks very high values of k are most often found in samples with very small surface areas - and vice versa - for this reason the capacities for water adsorption vary less than either the surface affinities of water k or the BET specific surface areas. On this basis it is possible to classify geothermal rocks into five types. For type A the capacity for water adsorption is due mainly to the large surface area with a possible contribution of micropores, while for type B the capacity for water adsorption is due mainly to the specific chemical interactions with water. Type C includes rocks with intermediate properties, while types D and E include, respectively, rocks with large hydrophilic surfaces, and those with small, hydrophobic surfaces, represented for example by the serpentinite and felsite samples from the Satik *et al.* (1996) study (NCPA B-5 and CA 958-6). Types D and E are not typical for geothermal reservoirs. It has to be kept in mind that rocks are mixtures of various minerals, and overall adsorption characteristics are averages of many contributions.

The values for water adsorption on the AWI 1-2 samples indicated rather large differences in capacities for water adsorption between the samples from the same well. For example, the sample AWI 1-2 5 adsorbed ten times as much water as AWI 1-2 6. In fact, AWI 1-2 5 is the most adsorptive sample of all previously investigated geothermal reservoir rocks. It stores twice as much water as the most adsorptive sample among the set of 36 in the study of Satik *et al.* (1996). While the sample of serpentinite in the above work was not typical for The Geysers, the results of this work indicate that very high water storage capacities are not uncommon for the volcanic tuffs and lahars of the AWI 1-2 well. The affinities for water $k_{0.1}$ of the Awibengkok rocks are intermediate, as they vary from 2.1 to 7.4 while the BET surface areas are high (up to 12.4 m^2/g). The adsorption properties of the AWI 1-2 rocks appear to belong to the types C and A - similar to those of the previously investigated samples from The Geysers wells MLM-3 ($k_{0.5}$ from 2.1 to 5.3, type C) and NEGU-17 ($k_{0.1} = 1.2$, type A). The samples from The Geysers well Prati State 12 interact with water much stronger ($k_{0.5}$ from 6.5 to 14.2), while the CA 1862-4 samples are clearly of type B ($k_{0.5}$ from 21 to 51).

The differences in surface - water interactions can be illustrated by comparing three samples - Brazilian quartz, CA 1862-4, and NEGU-17. The first two samples have similar BET specific surface areas - 0.14 and 0.17 m^2/g . However, the adsorption of water on Brazilian quartz is below the sensitivity of our experimental method (about 0.03 mg/g at $p/p_0 = 0.5$). Clearly, the enhanced adsorption of water on CA 1862-4 is due to the chemical properties of the minerals, assuming no micropore-filling contribution. Conversely, for the NEGU-17 metagraywacke $k_{0.1} = 1.2$, indicating adsorptive

properties close to those of silica. However, the amount of water adsorbed at $p/p_0 = 0.5$ is 1.17 mg/g, due to the substantial BET surface area for this sample (4.06 m²/g).

Figure 1 shows cumulative volume curves obtained by the mercury intrusion method. The curves are divided into two parts at the pore size of 10 μm . The volume of the macropores is set to zero at this point in order to make a more meaningful comparison of the part of the pore system that is most significant for adsorption. Very wide pores and cracks above 0.01 mm are important for fluid transport, but they contribute little to adsorption. AWI 1-2 10 was the sample with the largest volume of macropores and at the same time the smallest volume of mesopores (pores less than 50 nm wide), only 12 $\mu\text{L/g}$. At the opposite end of the spectrum, AWI 1-2 1 had roughly two times less volume of the macropores, and the largest mesopore volume (91 $\mu\text{L/g}$). All the Awibengkok samples had pore volumes exceeding those of the two Geysers samples. The AWI 1-2 samples formed the following sequence ordered by the volumes of pores below 10 μm : 2, 1, 7, 4 and 5, 6, 9, 11, 10. The correlation with water adsorption capacities was weak, but in general rocks with very high adsorption capacities and large pore volumes were found at the top of the well.

Porosities of the AWI 1-2 samples are given in Table 1 for all pores wider than 1 μm . Again, the widest pores were omitted in order to avoid random variations, but they may add another 3 to 10 percentage points to the total porosity. The porosities were larger than those measured previously for most of The Geysers rocks. There is a decreasing tendency with the depth of the well, consistent with the data supplied by Unocal Geothermal, which indicated porosities mainly between 15 and 20 % in the top portion of the core, and between 10 and 15 % in its bottom portion.

The adsorption results were consistent with the mineralogy of the AWI 1-2 corehole (Hulen, 1998). Water adsorption capacity results for the AWI 1-2 samples show large differences between samples from the same well. It appears that, as observed earlier, the proportion between the grain and matrix material is a good rough predictor of water adsorption capacity. The sample AWI 1-2 5, a tuff with a high illite content and a low plagioclase content, showed an extremely high water adsorption capacity (30 mg/g at $p/p_0 = 0.8$). Hydrothermal alteration mineralogy data compiled by Hulén show that sample 5 might contain less than 5 % plagioclase and over 50 % illite, while for the sample 6 these proportions could be reverse. Another interesting feature of the AWI 1-2 5 sample is a very wide low-pressure hysteresis loop persisting down to a relative pressure at least as low as 0.02 (Figure 2). The high-pressure hysteresis loop, due to capillary condensation, is narrower. This shape of the adsorption/desorption isotherm points to clay minerals interacting very strongly with water. Typical porous solids with hysteresis loops due to capillary condensation and with mostly physical adsorption have adsorption/desorption isotherms similar to those for the sample 7BT from the MLM-3 well in The Geysers (Figure 3). The pressure of the onset of the high-pressure hysteresis loop is clearly visible and it increases with increasing temperature, so that the high-pressure hysteresis loop contracts.

All the samples showed some gradual, continuous decrease in the adsorption capacity with temperature at high relative

pressures (Figures 2 and 3). The magnitude of this decrease varied from negligible to nearly 30 percent. Additionally, an abrupt decrease of adsorption was observed in several samples at 250 °C. At $p/p_0 = 0.5$ these samples showed a decrease of the amount adsorbed with increasing vapor pressure. It is likely that an irreversible reduction of the available surface area occurred, caused by dissolution of a mineral or a structural change. On the other hand, the capacity for adsorption at low relative pressures often increased (Figure 2). In some samples water or other adsorbent was apparently bonded to the surface very strongly, so that a further mass loss and an apparent increase of the available specific surface area accompanied evacuation at each higher temperature. For example, it appears that after prolonged evacuation of sample 9 at 150 °C more than half of its surface was still covered with an adsorbent, which was later removed at 250 °C. More detailed analysis of the changes observed during high-temperature adsorption experiments in the context of mineralogy will be made after detailed mineral compositions for our samples are available.

Previous adsorption measurements on three grain-size fractions have shown that there is no detectable effect of sample grain size as long as the grains are not very small (below 0.1 mm). The time needed to reach equilibrium did not depend on the grain size in the range investigated. The irreversible adsorption observed previously in the small grain size fractions was attributed entirely to the contamination of the samples with iron. The results for the small grain size fraction of the AWI 1-2 11 sample from the depth of 1674 m supported the earlier conclusion. The shapes of the adsorption isotherms for the two grain-size fractions of this sample were similar, with the specific surface area about 1.5 times greater for the fine fraction. No irreversible adsorption similar to that observed earlier (Gruszkiewicz *et al.*, 1998) was found.

4. CONCLUSIONS

The *in situ* weighing method was improved since the first adsorption measurements were made at temperature and pressure conditions found in vapor-dominated geothermal reservoirs. The measurements provide accurate water adsorption results. It is possible to obtain isotherms with sufficient resolution to show clearly the differences in the affinity for water even in the low-pressure region. As a rule the capacity for reversible adsorption tends to increase with increasing temperature at low vapor pressures and decrease with increasing temperature at high vapor pressures.

The accumulating water adsorption database shows that the capacity of most geothermal reservoir rocks for water adsorption varies less than the chemical affinity of the rock surfaces for water, their specific surface areas determined from nitrogen adsorption, or pore system volumes. This indicates that there is a correlation between the macroscopic properties of the pore structure and the nature of the component minerals. Geothermal rocks with small surface areas tend to feature minerals with higher affinities for water, probably as a result of hydrothermal alteration. It appears that it will be soon possible to develop correlations for estimating water adsorption capacities for "typical" geothermal rocks using simplified rock classifications, BET surface areas, and the information about pore structure.

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Table 1. Selected characteristics of the well core samples obtained by nitrogen adsorption, at 77 K, water adsorption at 423.15 K, and mercury intrusion.

Sample	Depth (m)	BET specific surface area (m ² /g)	Water adsorption at p/p ₀ =0.5 (mg/g)	k _{0.1}	k _{0.5}	Porosity (pores less than 1 μm wide, %)	Density (g/cm ³)
AWI 1-2 1	871.15 - 871.24	12.43	6.9	2.1	3.0	22.1	2.817
AWI 1-2 2	966.64 - 966.73	4.80	3.25	2.5	3.7	22.4	2.778
AWI 1-2 4	1088.42 - 1088.23	8.03	3.8	2.8	2.6	16.4	2.780
AWI 1-2 5	1171.16 - 1171.22	11.10	12	2.6	5.9	17.9	2.827
AWI 1-2 6	1254.59 - 1254.65	1.62	1.25	3.0	4.2	15.0	2.820
AWI 1-2 7	1339.26 - 1339.34	3.95	2.0	2.3	2.7	5.70	2.825
AWI 1-2 9	1547.99 - 1548.05	2.95	2.8	7.4	5.1	12.9	2.818
AWI 1-2 10	1658.51 - 1658.63	1.47	1.6	3.9	5.9	4.04	2.840
AWI 1-2 11	1673.89 - 1673.95	2.38	1.5	5.1	3.4	9.04	2.795
AWI 1-2 11F	1673.89 - 1673.95	3.47	2.25	5.9	3.5	-	2.846
CA 1862-4	1360.9	0.17 (0.32)	1.45	21	45	2.53	2.775
MLM-3 7BT	1324.72 - 1325.27	3.05 (3.15)	3.0	5.3	5.3	2.84	2.815
MLM-3	1322	1.31	1.25	4.3	5.1	6.96	2.744
NEGU-17	2600	4.06	1.17	1.2	1.6	4.56	2.778
Prati State 12	1909	0.36	0.95	15.7	14.2	3.95	2.749

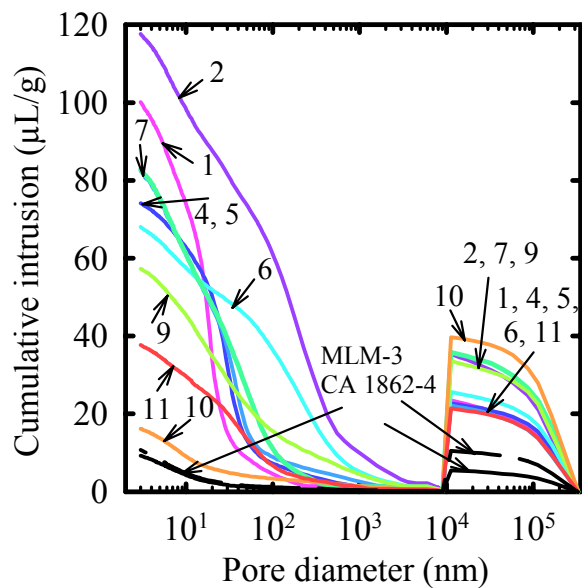


Figure 1. Cumulative pore volumes from mercury intrusion.

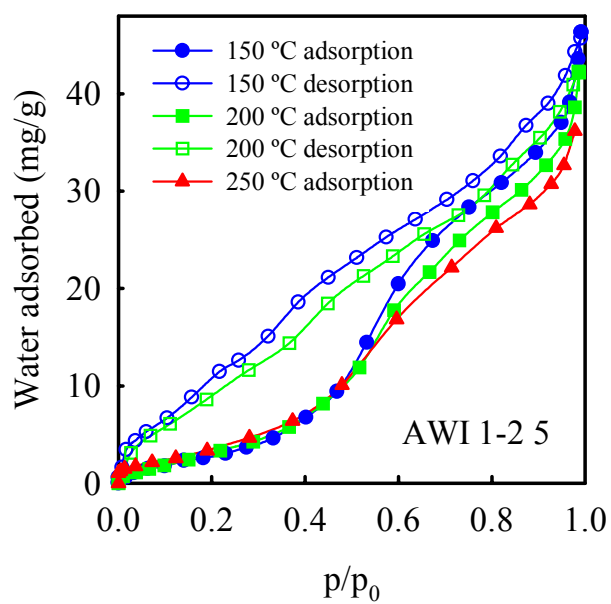


Figure 2. Adsorption and desorption isotherms for AWI 1-2 sample 5.

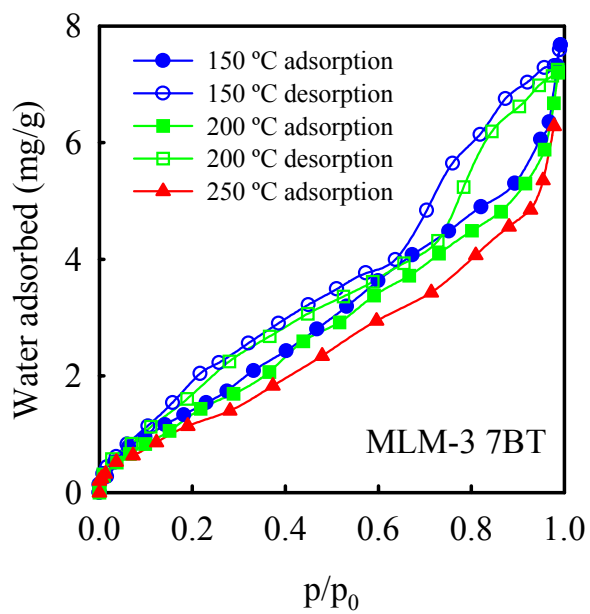


Figure 3. Adsorption and desorption isotherms for The Geysers sample MLM-3 7BT