

4-28-2022

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10.1029/2022GL098261

Sedev, R., Akhondzadeh, H., Ali, M., Keshavarz, A., & Iglauer, S. (2022). Contact Angles of a Brine on a Bituminous Coal in Compressed Hydrogen. *Geophysical Research Letters*, 49(8), p. 1-6, e2022GL098261. <https://doi.org/10.1029/2022GL098261>

10.1029/2022GL098261

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Geophysical Research Letters[®]



RESEARCH LETTER

10.1029/2022GL098261

Key Points:

- The surface of a bituminous coal is weakly water-wet in a hydrogen environment
- Hydrogen pressure increases the brine contact angles at 25°C but has no impact at 50 or 70°C
- Wettability changes are attributed to the hydrogen adsorption on the coal surface

Supporting Information:

Supporting Information may be found in the online version of this article.

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Citation:

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Received 13 FEB 2022
 Accepted 12 APR 2022

Author Contributions:

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Data curation: Rossen Sedev
Funding acquisition: Alireza Keshavarz, Stefan Iglauer
Investigation: Rossen Sedev
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Contact Angles of a Brine on a Bituminous Coal in Compressed Hydrogen

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Abstract Hydrogen storage is a main issue in the establishment of a hydrogen economy. Geo-storage could be a viable solution if hydrogen could be injected into and withdrawn from suitable geological formations, reversibly and reliably. Rock wettability is a major factor as it affects injectivities, withdrawal rates, storage capacities, and containment security. We report here the contact angles of a brine on the surface of a bituminous coal in a pressurized hydrogen atmosphere. Under realistic geo-storage conditions the coal surface was weakly water-wet. Hydrogen pressure increased brine contact angles at 25°C but did not have an impact at 50 or 70°C. We present a thermodynamic model that describes the observed behavior. Our results would support the development of large-scale geo-storage technologies for hydrogen.

Plain Language Summary The wide use of hydrogen as a clean fuel will require significant storage capacities. Above ground facilities are expensive and the possibility of underground storage must be considered. It is technologically feasible as it is already practiced with natural gas and carbon dioxide. Wettability, which reflects the interactions between the liquids, gases and rocks, plays an important role in understanding and controlling the behavior and movement of gases inside rocks partially filled with brine. We provide here measurements of the brine wettability of coal in the presence of hydrogen. These results will feed into the development of new technologies for underground storage of hydrogen.

1. Introduction

Hydrogen is a highly calorific yet clean fuel. Its role in decarbonizing the economy and developing renewable energy sources is expected to grow vastly (Zohuri, 2019). Hydrogen provides large amounts of energy per unit mass but much lower ones per unit volume. This makes large-scale hydrogen storage a serious problem (Scipioni et al., 2017). There is a growing interest in the geological storage of hydrogen (Heinemann et al., 2021; Panfilov, 2016). Currently hydrogen is stored underground but mostly in salt caverns (Hashemi, Blunt, & Hajibeygi, 2021; Hashemi, Glerum, et al., 2021). A recent report states that using salt caverns for storing hydrogen is more than 20 times cheaper in comparison to above ground facilities (Anonymous, 2021). However, salt caverns are not that common. Research suggests that depleted oil or gas fields, or sedimentary aquifers could be used for that purpose (Flesch et al., 2018; Heinemann et al., 2018; Tarkowski, 2019; Yekta et al., 2018). We have recently argued that hydrogen geo-storage in coal seams should be researched along with deep aquifers and exhausted oil and gas reservoirs (Arif et al., 2022; Iglauer, Abid, et al., 2021; Iglauer et al., 2022; Keshavarz et al., 2022; Pan, Yin, Ju, & Iglauer, 2021).

Rock wettability is of major importance in developing strategies for underground gas storage as it affects initial fluid distribution, fluid transport, storage capacity, and containment security (Al-Khdheawi et al., 2017; Anderson, 1987a, 1987b, 1987c; Broseta et al., 2012; Donaldson & Alam, 2008; Iglauer, 2017). We consider the possibility of storing hydrogen in coal seam gas reservoirs. Typical geological conditions would be: temperature 27–52°C, pressure 1–120 bar (Pashin & McIntyre, 2003), salinity 0.5%–3.5% (Pashin et al., 2014), and pH = 6.3–9.3 (Meng et al., 2014). However, hydrogen geo-storage is a novel topic and there is a severe lack of wettability data obtained under realistic reservoir conditions.

The recent work of Hashemi et al. (Hashemi, Blunt, & Hajibeygi, 2021; Hashemi, Glerum, et al., 2021) emphasizes the importance of wettability data for a successful pore-scale modeling of the brine/hydrogen flow in porous media. Yet very little is known about the influence of hydrogen on rock wettability (Heinemann et al., 2021). We have recently researched aspects of hydrogen geo-storage (Ali et al., 2021; Hosseini et al., 2022a, 2022b;

Writing – review & editing: Rossen Sedev, Alireza Keshavarz, Stefan Iglauer

Iglauer, Ali, & Keshavarz, 2021). Our current work fills some of the knowledge gap in the wettability of coal in a hydrogen environment.

We have reported the brine wettability of quartz surfaces modified with stearic acid (to represent realistically the rock surfaces in a sandstone reservoir) under hydrogen at pressures up to 250 bar at 23, 50, and 70°C (Iglauer, Ali, & Keshavarz, 2021). The contact angle of brine (10% NaCl) increased with hydrogen pressure and decreased with temperature. A study by Hashemi et al. (Hashemi, Blunt, & Hajibeygi, 2021; Hashemi, Glerum, et al., 2021) showed that Berea and Bentheimer sandstones remained water-wet in the presence of hydrogen. However, no clear trend in wettability was established when varying the temperature (20–50°C), pressure (20–100 bar), or salinity (0%, 0.5% and 5% NaCl).

We report here the wettability of a bituminous coal probed with a brine (2% NaCl) under pressurized hydrogen (5–90 bar) at three temperatures (25, 50, and 70°C). The coal surface was weakly water-wet under the experimental conditions. The role of hydrogen pressure was significant only at the lowest temperature tested (25°C). We propose a thermodynamic model that accounts for the experimental observations.

2. Materials and Methods

2.1. Materials

A sample of coal (Coal bituminous KG, Morgantown, West Virginia, USA) was sourced from Ward's Science (Rochester, NY). Key parameters of the sample are listed in Table 1.

The model brine was 2% aqueous NaCl (pH = 5.8). Compressed hydrogen was obtained from Coregas (Hydrogen 5.0: hydrogen \geq 99.999%, moisture \leq 5 ppm, oxygen \leq 2 ppm).

2.2. Methods

Five rectangular samples were cut from the same piece of coal and dry filed into shape. They were wet polished under flowing tap water (with sandpapers P120, P400, P600, and P1200) and then air-dried. The samples were reused after a light repolish (with P600 and P1200). Optical images of the polished coal surfaces are shown in Figure S1 in Supporting Information S1. Clearly, the samples were rough, heterogeneous, and have some macroscopic defects. Nevertheless, they were reflective enough to enable a correct use of the tilted drop method for contact angle measurement (Figure S2 in Supporting Information S1). The hysteresis, $\theta_A - \theta_R$, directly reflects the nonideality of the coal surface. The contact angles measured with water, under ambient conditions, were $\theta_A = 73^\circ \pm 3^\circ$ (advancing) and $\theta_R = 65^\circ \pm 2^\circ$ (receding). Thus, the wetting hysteresis on the coal surface was manageable (Figure S3 in Supporting Information S1).

A high-pressure high-temperature apparatus (CoreLab, IFT-10) was used for contact angle measurement (Sarmadivaleh et al., 2015) (a schematic is shown in Figure S4 in Supporting Information S1). The coal sample was placed on a tilted stage ($15^\circ \pm 2^\circ$). The pressurized hydrogen was introduced in the cell and a small drop of brine (25 μ l) was dispensed from a height of about 4 mm by pumping the brine at 0.2 ml/min. When the drop hits the surface it deforms, vibrates intensely but briefly, and then settles. The average contact diameter was 5.2 mm. Because gravity pulls the drop down, that is, sideways on the inclined coal surface, it is asymmetric. The contact angle at the leading edge is largest and is considered to be the advancing contact angle, θ_A ; the one at the trailing edge is the smallest and is labeled the receding one, θ_R . The whole process was filmed and selected images were used to measure θ_A and θ_R using the software ImageJ (Schneider et al., 2012).

Measurements were carried out at 25, 50, and 70°C.

3. Results and Discussion

The brine contact angles measured at 25°C are plotted in Figure 1. The error bars represent the uncertainty of the individual measurement (estimated at $\leq 2^\circ$). The overall scatter is larger and reflects the roughness and heterogeneity of the coal surfaces. Both factors affect the value of the contact angle and contribute to the contact angle

Table 1
Properties of the Bituminous Coal Studied (Maximum Vitrinite Reflectance 0.89%)

Proximate analysis (%)			
Inherent Moisture	Ash	Volatile Matter	Fixed Carbon
1.7	4.8	4.8	88.7
Ultimate Analysis (%)			
C	H	N	S
78.8	5.30	1.58	1.44
Maceral Composition (vol%)			
Vitrinite	Liptinite	Inertinite	Minerals
81.0	5.2	12.0	1.8

hysteresis, that is, the difference between the advancing and receding angles. An extensive study of drops on tilted surfaces suggested that roughness is of secondary importance and hysteresis is governed by the chemical heterogeneity of the surface (Extrand & Kumagai, 1995).

There is a gradual increase in both advancing and receding contact angles as gas pressure increases. We propose a simple thermodynamic description for this trend. Most theoretical considerations of the equilibrium contact angle, θ , are based on Young equation which describes the thermodynamic equilibrium at the three-phase contact line (Adamson & Gast, 1997):

$$\gamma_{12} \cos \theta = \gamma_{S1} - \gamma_{S2} \quad (1)$$

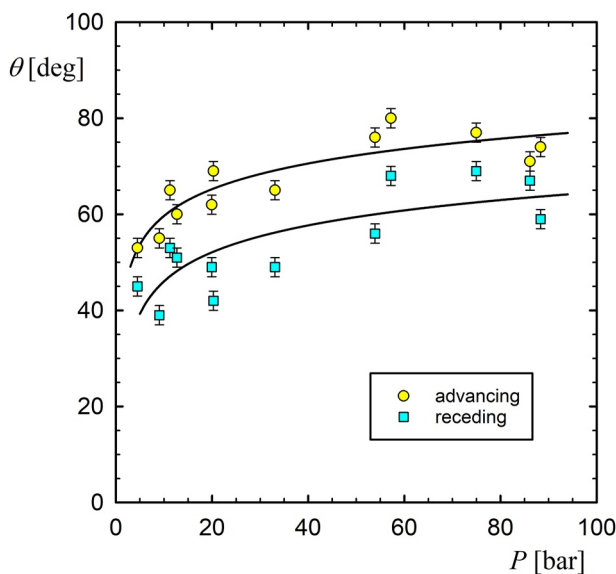
The three interfacial tensions pertain to the three interfaces brine-hydrogen, γ_{12} , coal-hydrogen, γ_{S1} , and coal-brine, γ_{S2} . Under our experimental conditions, the compressibility of the gas is significant and exceeds by far the values for the solid and liquid phases. It is therefore reasonable to assume

that only the liquid-gas and solid-gas interfacial tensions could be affected by the varying gas pressure. Thermodynamics relates the pressure dependence of the interfacial tension to a length parameter, h (Adamson & Gast, 1997):

$$\left(\frac{\partial \gamma_{12}}{\partial P} \right)_T = h \quad (2)$$

The parameter h can be interpreted as the thickness of the interfacial layer, $\sim 10^{-10}$ m (Neumann et al., 2011) or the distance between the two equimolar planes (Hansen, 1962). In any case, it has a very small value and thus the surface tension of a pure liquid shows a weak dependence on the system pressure. We consider this to be applicable to the brine used as the salt is not surface active and the solution is not very concentrated (~ 0.3 mol/l). Therefore variations in γ_{12} can be ignored. We suggest that the main influence of pressure is on the solid-gas interfacial tension which is given by the Gibbs adsorption equation (Adamson & Gast, 1997), where μ_1 is the chemical potential of the hydrogen and Γ is the hydrogen adsorption on the coal surface:

$$\frac{\partial \gamma_{S1}}{\partial \mu_1} = -\Gamma \quad (3)$$



It follows from this equation that the coal-hydrogen interfacial tension, γ_{S1} , decreases with pressure due to the increased adsorption of hydrogen on the coal surface, Γ . Recently, Pan et al. (Pan, Yin, & Iglauer, 2021) and Ali et al. (Ali et al., 2022) used Neumann's equation of state approach (Neumann et al., 2011) to extract the pressure dependence of the solid-gas interfacial tension from literature data. The interfacial tensions of quartz-hydrogen, basalt-hydrogen and mica-hydrogen all decreased with pressure. Here, we adopt the direct thermodynamic approach based on the Gibbs adsorption equation.

Using the standard expression for the chemical potential of the gas, $\mu_1 = \mu_1^0 + RT \ln P$, we obtain:

$$-\frac{\partial \cos \theta}{\partial \ln P} = \frac{\Gamma RT}{\gamma_{12}} \quad (4)$$

In other words, the cosine of the contact angle should be a linear function of the logarithm of the gas pressure. This is indeed the case (See Figure S5 in Supporting Information S1). We therefore use the following equation (with a and b derived from the fitted lines in Figure S5 in Supporting Information S1) to draw the lines in Figure 1:

Figure 1. Contact angles of brine (2% NaCl), θ , on bituminous coal in hydrogen as a function of pressure, P (25°C). The solid lines are the best fits of the equation (5) for advancing and receding contact angles.

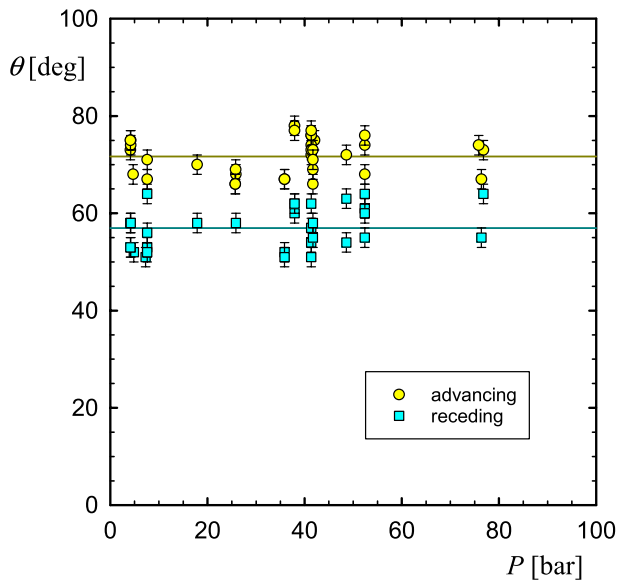


Figure 2. Contact angles of brine (2% NaCl), θ , on bituminous coal in hydrogen as a function of pressure, P (50°C). The solid lines indicate the mean values of the advancing and receding contact angles.

we reported the adsorption of hydrogen on a sub-bituminous coal is in the pressure range of 30–120 bar (Iglauer, Abid, et al., 2021). The amount of hydrogen adsorbed more than doubled with pressure at 30 and 45°C. However, at 60°C, it grew by 30% only.

The underground storage of hydrogen bears physical similarities to the geologic sequestration of carbon dioxide. The importance of capillary trapping for CO₂ is well-documented (Krevor et al., 2015) and should play an equally important role for hydrogen. The capillary pressure at the brine–hydrogen interface, P_c , is given by (Adamson & Gast, 1997)

$$P_c = \frac{2\gamma_{12}}{r} \cos \theta \quad (6)$$

It is highly relevant for the trapping of a continuous phase (Green & Willhite, 2018). For a fixed rock and brine combination (i.e., fixed interfacial tension, γ_{12} , and pore radius, r) the role of wettability is given by $\cos \theta$. In our case, at a temperature of 25°C (say a reservoir depth of 250 ft, (Pashin & McIntyre, 2003)), the wettability of coal decreases from 0.69 at 10 bar to 0.42 at 100 bar hydrogen pressure. This is about 40% loss in capillary pressure opposing hydrogen migration into the pores filled with brine (we consider the receding contact angle as in this scenario the brine is receding). At 50°C (say a depth of 5,200 ft, (Pashin & McIntyre, 2003)), the wettability is 0.60 and essentially independent of hydrogen pressure. In both cases, the scatter around these averages is significant: 25%–30%. This is due to the natural heterogeneity of the coal surface and implies significant space variations in the capillary configurations on the microscale. In the case of a trapped drop, two brine–hydrogen interfaces come into play (Green & Willhite, 2018) and therefore both advancing and receding contact angles must be taken into account. Thus our measurements provide a physical basis for realistic simulations of the distribution of a hydrogen plume in a coal reservoir.

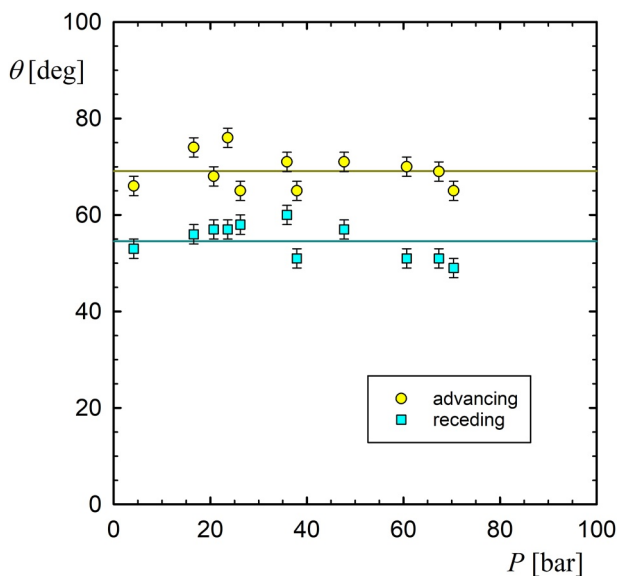


Figure 3. Contact angles of brine (2% NaCl), θ , on bituminous coal in hydrogen as a function of pressure, P (70°C). The solid lines indicate the mean values of the advancing and receding contact angles.

$$\cos \theta = a - b \log_{10} P \quad (5)$$

The description is reasonably good. We also note that the behavior of the advancing and receding contact angles is essentially the same. This suggests that the thermodynamic explanation is valid and affects similarly θ_A and θ_R (which themselves are not equilibrium parameters). Our approach of randomizing the coal surfaces tested resulted in robust results, which reveal both the heterogeneity of the coal surface and the thermodynamic reason for the observed behavior.

The contact angles measured at 50°C are shown in Figure 2. While the scatter is of the same order of magnitude, there is no statistically significant influence of the gas pressure on the brine contact angles.

This result was further confirmed by experiments conducted at 70°C (see Figure 3).

Hydrogen is physisorbed on coal due to weak van der Waals interactions. The average adsorption energy on various carbon-based surfaces is 4–5 kJ/mol (Ströbel et al., 2006). Also hydrogen desorbs quickly with increasing temperature (Panella et al., 2005). Given that adsorption is an exothermic process, the value of Γ declines with temperature (Yang & Saunders, 1985). It appears that in our study, in the temperature range 50–70°C, the variations of hydrogen adsorption on coal are too small to have a measurable impact on the brine contact angle (within the studied pressure range). In a recent study

4. Conclusions

This work reveals the variations of the brine contact angle on bituminous coal under pressurized hydrogen. The scatter in the data is due to the heterogeneous nature of the coal surface. The contact angle hysteresis measures the non-ideality of the coal surface. At all times the coal surface remains weakly water-wet (i.e., moderately hydrophobic). At 25°C, the brine contact angle increases with hydrogen pressure. We relate this change to the increased hydrogen adsorption on coal. At temperatures 50 and 70°C, the contact angle of the brine is essentially independent of the hydrogen pressure. We attribute this to the reduced hydrogen adsorption, which is an exothermic process. The wettability parameters measured here contribute to the realistic understanding of hydrogen trapping in depleted coal reservoirs and the development of underground hydrogen storage technologies.

Data Availability Statement

The data presented in this work are original and available from the Edith Cowan University institutional repository (<https://doi.org/10.25958/nhre-2m52>).

Acknowledgments

This research was supported by the Australian Government through the Australian Research Council's Discovery Projects funding scheme (project DP220102907). R. Sedev thanks Faisal Awan for engaging discussions on the properties of coal and coal reservoirs. Open access publishing facilitated by Edith Cowan University, as part of the Wiley - Edith Cowan University agreement via the Council of Australian University Librarians.

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