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# Experimental Study on the Dissociation Behavior and Productivity of Gas Hydrate by Brine Injection Scheme in Porous Rock

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This study presents the experimental apparatus to analyze the dissociating phenomena of gas hydrate in porous rock. The experiments by brine injection scheme have been carried out in a way that can allow for the analysis of dissociation characteristics and how various brine concentrations and permeabilities affect the gas productivity. With the experiments, the pressure and flow behavior have been investigated during the dissociation period, as well as the productivity of dissociated gas from gas hydrate. As a result, it has been observed that, if the brine concentration is excessively high, the gas production rate tends to reduce significantly. This means that excessive NaCl molecules disturb the fluid flow between pores and, thus, significantly reduce the permeability. In the experiment with varying permeability, an increase in the permeability caused an increase in the gas production and a decrease in the dissociation rate. A difference in the production rate was also observed up to 35%. Therefore, it is experimentally verified that the permeability of the rock, which describes the connectivity of pores, is one of the most important factors that influence production from gas hydrates. It suggests that, during gas production from a gas hydrate reservoir, the dissociation methods must be conducive to increase or at least maintain the reservoir permeability.

## Introduction

To prepare for the depleting petroleum/natural gas reserves and also the consequent high oil price, research in new energy sources is imperative. To achieve this, both developed and developing countries are strengthening their investment in the research. Recently around the world, gas hydrate is being recognized as a potential strategic energy resource for finite resources, such as petroleum or natural gas. This is because gas hydrate is vastly distributed throughout both marine and permafrost areas, and its deposited amounts are immense.[1,2] To produce gas hydrate from deep sea sediments, more information on the physical characteristics, flow characteristics, and dissociation behavior of the hydrate-bearing formations is necessary.[3] However, because of technical difficulties and geo-environmental risks, commercial profit from production in these reservoirs is not possible at this stage. Production will involve facilitating the dissociation of solid gas hydrates and gathering the produced gas. Because the process will bring significant changes in sedimental structures, safety issues must be appropriately established.[4] A few of the suggested methods are as follows: depressurization, which decreases the reservoir pressure to a point lower than the equilibrium pressure, thermal stimulation, which heats the reservoir, usually with hot water or steam injection to induce dissociation, inhibitor injection, which involves the injection of chemical additives, such as methanol, glycol, or brine, and $CO_2$ swapping, which replaces the methane molecules in the gas hydrate structure with carbon dioxide molecules. Each method has its own strengths and weaknesses, but all of these methods involve the addition of energy into the sediment layer by an external source.

Depressurization is used most commonly because it is economic. Unlike thermal stimulation or the inhibitor injection, it does not require any additional costs. However, because it has a long dissociation time, it has a low production rate and, thus, a low productivity. Also, because the dissociation reaction is endothermic, there is a possibility of gas hydrate reformation.[5,6] Thermal stimulation is more efficient than other methods because hydrate reacts most readily to heat. However, the injected energy can spread throughout not only the gas hydrate-bearing layer but also the surrounding layers that may not have gas hydrates. Heat loss can range from 10 to 75% of the injected total energy.[7−9] This high

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(1) Kvenvolden, K. A.; Lorenson, T. D. The global occurrence of natural gas hydrates. In *Natural Gas Hydrates—Occurrence, Distribution, and Detection*; Charles, K. P., William, P. D., Eds.; American Geophysical Union (AGU): Washington, D.C., 2001; AGU Geophysical Monograph Series, Vol. 124, pp 3−18.
(2) Collett, T. S. Gas hydrates as a future energy resource. *Geotimes* **2004**, *49*, 24–27.
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percentage loss is considered to be the major weakness of the method. Inhibitor injection will infuse liquid inhibitors to shift the equilibrium curve of the gas hydrate. The gas hydrate begins to dissociate as the equilibrium condition is changed by the injected inhibitor. This method may be effective, but it has the downside of possibly polluting the environment and also the high cost of the inhibitor used in the process.[10,11] Few reports are found about the investigation of the dissociation behavior for methane hydrate in the porous media under hot brine injection. Kamath et al.[12] conducted a series of core experiments examining depressurization and injection of hot brine schemes. Li et al.[13] experimentally investigated the gas production behavior from methane hydrate in porous sediment by injecting hot brine with different concentrations and different temperatures. The results showed that the production efficiency was affected by both the brine concentration and temperature. However, the impacts of the concentrations of the injected brine solution in porous rock with different permeabilities have not yet been investigated. $CO_2$ swapping changes the molecular structure. It replaces the methane molecules with carbon dioxide molecules.[14] This is attracting attention not only as a potential approach to produce gas from natural gas hydrates but also as a technique that may allow for $CO_2$ sequestration. However, it is a field that requires further research to be practically and commercially viable.[15]

Figure 1 presents the equilibrium curve of the gas hydrate in relation to pressure and temperature and also the phase change involved in each production scheme. Of the listed approaches, brine injection will be mainly discussed in this study. When brine is injected, dissociation is induced as the gas hydrate equilibrium curve is shifted away from its stability zone.[7] A slight effect of heat injection can also be expected, because there is about a 15 K difference between the temperature in the upper part of the seawater and the lower part (below the sea floor). In this study, the experiments using the brine injection scheme in porous rock were carried out in a way that allows for the analysis of dissociation and production behavior. The objectives of this study are to investigate the impacts of the concentrations of the injected brine solution in porous rock with different permeabilities and the permeability of the rock for the gas hydrate production.

## Experimental Section

**Experimental Apparatus.** The study examines the gas hydrate formation and dissociation behavior within porous rocks and also gas productivity from gas hydrate by brine injection
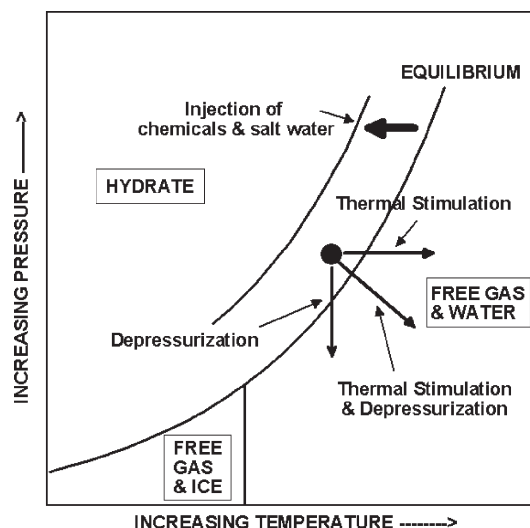


**Figure 1.** Dissociation mechanism of the gas hydrate.

scheme. The experimental apparatus was designed and set up to conduct gas hydrate research in porous media. Figure 2 shows the schematic diagram of the experimental system. The core holder, which is a main part of the system, was constructed with 316 SUS material, so that it will endure high-pressure and low-temperature conditions. The inlet and outlet of the core are held by end plugs, and the outside of the core is fixed by a Viton sleeve with confining pressure, which is a minimum of 1 MPa greater than within the core. It allows for the flow only in an axial direction. Including the core-in and core-out sections, there are total of seven pairs of sensors, with each pair consisting of one pressure transmitter (PT) port and one resistance temperature detector (RTD) sensor (Figure 3). The accuracy of the pressure and temperature measurements is within ±20 kPa and ±0.01 K. The core holder is covered with a cooling water jacket, so that the temperature can be controlled by the refrigerated circulator. Because the major component of naturally occurring gas hydrates is methane, methane gas of 99.95% purity was used in this experiment. Distilled water was used to keep the influence of impurities to a minimum. Brine is made using sodium chloride, purity grade 99.5%, from DC Chemical Co. Gas is injected from the gas cylinder to the core, while water and brine are injected using a syringe pump. A data acquisition system continuously records all of the information, such as the pressure, temperature, and flow quantity, as the experiment is conducted. Water and gas produced after dissociation are separated in the separator after it goes through the back-pressure regulator. The water is collected in the mass cylinder, and the dissociated gas is measured through the wet gas meter with 0.1% relative standard deviation. The cores used in this experiment are Berea sandstones, which have lengths of 304 mm and diameters of 381 mm. Berea sandstones are commonly used by the petroleum industry as standard porous media for laboratory experiments because they are relatively homogeneous.[16]

**Experimental Procedure.** To form gas hydrates in porous rocks, Berea sandstone is placed within the core holder. A vacuum pump is used to pump air and other impurities out of the core. The pore is then fully saturated with distilled water using a syringe pump. When this process is performed, methane gas is injected and circulated until the desired water saturation value is attained. The initial water saturation in this experiment is set at about 60%. Next, to begin the hydrate formation

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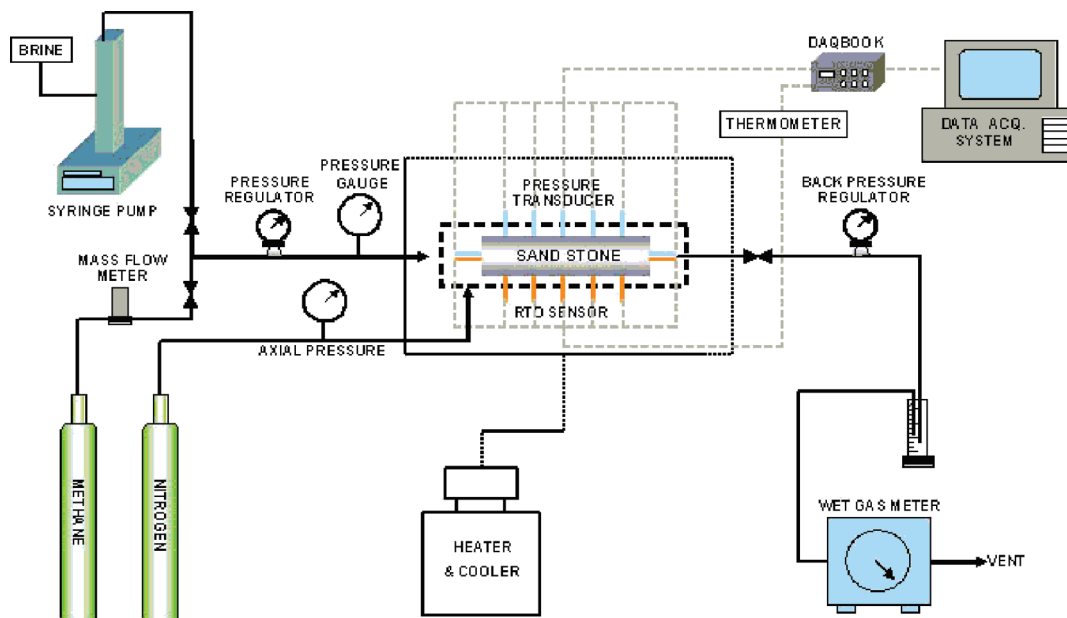
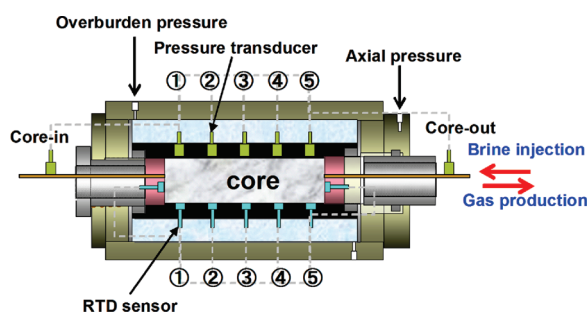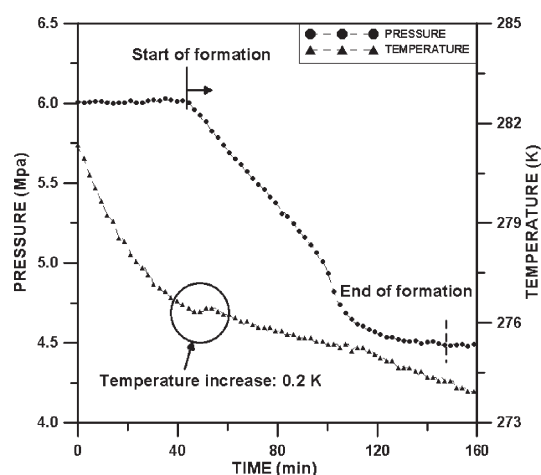**Figure 2.** Schematic diagram of the experimental system.



**Figure 3.** Details of the core holder and the point of brine injection and gas production.



**Figure 4.** *P−T* curve during hydrate formation.

experiment, methane gas is injected into the core to pressurize up to the initial pressure. The initial injection pressure of methane is set to 6.0 MPa, which is a point much higher than the equilibrium pressure (2.79 MPa at 273.76 K). The injection is continued until there is no more change in the overall core pressure. Then, the inlet valve is closed, and using the refrigerated circulator, the temperature is lowered to 273.76 K, so that gas hydrates can start forming. Figure 4 shows the pressure and temperature
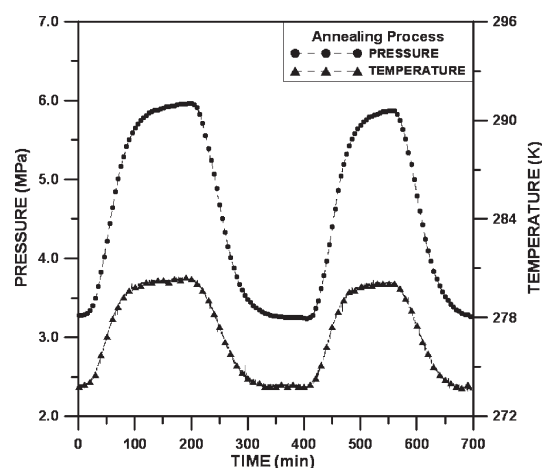


**Figure 5.** *P−T* behavior during the annealing process.

curve measured from the formation experiment. From this figure, it is observed that pressure decreases to 4.45 MPa from the injection pressure of 6 MPa and the exothermic temperature increases 0.2 K during the hydrate formation. The measurements are used to provide quantitative information on the amount of hydrate formation.

The amount of hydrate formed in the core samples is obtained using the following equation of state for real gases:

$$n_i - n_f = \frac{V}{RT}\left[\left(\frac{P}{z}\right)_i - \left(\frac{P}{z}\right)_f\right] \qquad (1)$$

where the amount of injection gas in the core holder is known by the measured gas mass flow during the injection period. When hydrate formation finished, the inner pressure in the core holder is measured. Thus, the amount of converted hydrate in core samples is calculated by substituting the measured gas mass flow and formation pressure for eq 1.

When the formation process is finished, the annealing process is conducted. The annealing process is a procedure that lets gas hydrates in a core distribute uniformly. It is performed by repeating the raising and the lowering of the core temperature, so that the gas hydrates go through the cycle of formation and

**Table 1. Salinity and Rock Properties for Experimental Runs**

| | experimental runs | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 1A | 1B | 1C | 2A | 2B | 2C | 3A | 3B | 3C |
| salinity (wt %) | 3.45 | 10.0 | 20.0 | 3.45 | 10.0 | 15.0 | 3.45 | 10.0 | 15.0 |
| permeability (md) | 71 | 71 | 71 | 89 | 89 | 89 | 169 | 169 | 169 |
| porosity (%) | 31 | 31 | 31 | 13.7 | 13.7 | 13.7 | 13 | 13 | 13 |
| water saturation (%) | 64 | 64 | 64 | 66 | 66 | 66 | 66 | 66 | 66 |

dissociation several times. The measured pressure and temperature during the annealing process are plotted in Figure 5. As shown in this figure, one can see that the pressure is almost the same before and after the annealing process, which indicates no more additional formation and redistribution of hydrate. In this experiment, the core temperature was set to 273.76 and 279.16 K alternately. This simulates the environment in which naturally occurring gas hydrates were formed.[17] Therefore, the annealing process is significant not only as a means to achieve even hydrate distribution for a better measurement and analysis but also as a means to replicate the natural formation process.

After the hydrate is distributed evenly along the core by the annealing process, the dissociation experiment through brine injection is performed. The brine of the required salinity was prepared in the brine reservoir. The temperature of brine was maintained at 288.15 K by the heater and cooler in the brine reservoir. To find a brine concentration that is the most conducive to gas hydrate dissociation, different concentrations of 3.45, 10.0, 15.0, and 20.0 wt % have been tested. A total of 15 mL of brine has been injected in the first 40 min and left for another 50 min of soaking time after closing the injection valve, letting the brine apply to all of the hydrates formed within the core. Next, the outlet valve was slowly opened to start off the hydrate dissociation process. All of the experiments were carried out in the temperature of 273.76 K and the dissociation pressure of 2.07 MPa and were left to dissociate until there was no more change in the pressure in each port. The amount of recovered gas was measured using the wet gas meter. Table 1 displays the salinity and physical properties of the used core samples in these experiments.

### Determination of Rock Properties

**Porosity.** The porosity is the fraction of the bulk volume of the material occupied by voids. It is described from the following equation:

$$\phi = \frac{V_b - V_s}{V_b} \times 100 \qquad (2)$$

Using eq 2, the total porosity of core samples is determined by measuring the bulk volume of core samples and the volume of the injected water. Here, it is assumed that the volume of the pores in the core sample is the same as the volume of the injected water.

**Permeability.** Permeability is the property of a porous material that characterizes the case with which a fluid may be made to flow through the material by an applied pressure gradient. The permeability of core samples is determined by following Darcy's equation.

$$k = \frac{\mu Q}{A}\left(-\frac{\Delta P}{\Delta L}\right) \qquad (3)$$

To obtain absolute permeability, the length and diameter of core samples are measured precisely. The core sample is placed within the core holder, and then distilled water is injected into the core sample by a constant flow rate until steady state. The flow rate of the inlet and outlet is the same.
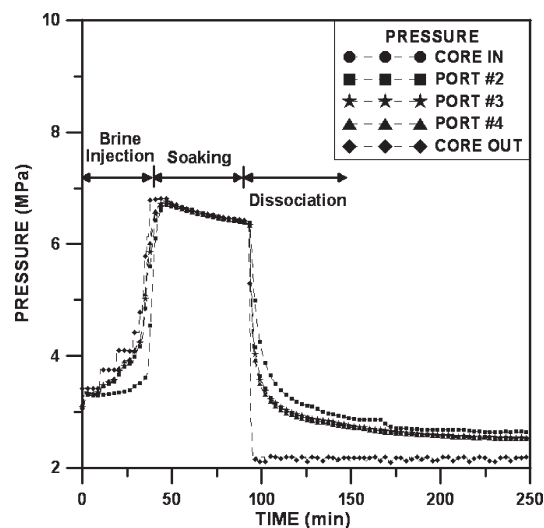


**Figure 6.** Pressure behaviors of run 1A recorded through brine injection, soaking, and the dissociation process.

If the fluid flow has reached a steady state, the flow rate and pressure difference of the inlet and outlet are measured. Absolute permeability of the core sample is calculated by substituting the measured flow rate, pressure difference, and water viscosity (1.0 cP) for Darcy's equation.

### Results and Discussion

Brine may be useful in the production of gas hydrate because it shifts the equilibrium curve of the gas hydrate from its stable zone to an unstable zone. The production can also slightly benefit from the effect of heat injection, because the higher seawater is about 15 K warmer than the sea floor. Figure 6 presents the pressure behavior in the inner core after brine has been injected and left for heat to soak. After gas hydrate formation has been thoroughly completed, brine has been gradually injected for 40 min using a syringe pump. The figure reflects that the pressure increase begins from the port closest to the point of brine injection; this implies that under real production conditions, the pressure increase will also begin from the layer in the immediate proximity to the point where brine injection occurs. The inner core pressure was raised until it reached 6.89 MPa. Next, the heat soaking process has been conducted for 50 min. As the soaking process advanced, the heat spread toward the periphery of the core and the inner core pressure decreased slowly to 6.45 MPa. Here, if the heat soaking is held for too long, the productivity may be aversely affected because the system will be influenced by the temperature of the surrounding area. Therefore, verifying the most appropriate heat soaking time is necessary.[18]

The sodium chloride (NaCl) component of brine has the tendency to shift the equilibrium point when mixed with gas hydrate. While the equilibrium point of pure methane hydrate is 2.79 MPa, mixtures with various concentrations of NaCl have different equilibrium points, as indicated in Figure 7. The NaCl concentration of 3.45 wt % changes the equilibrium point to 3.20 MPa, 10.0 wt % to 4.38 MPa, 15.0 wt % to 6.03 MPa, and 20.0 wt % to 9.41 MPa.[19] Because the purpose of this

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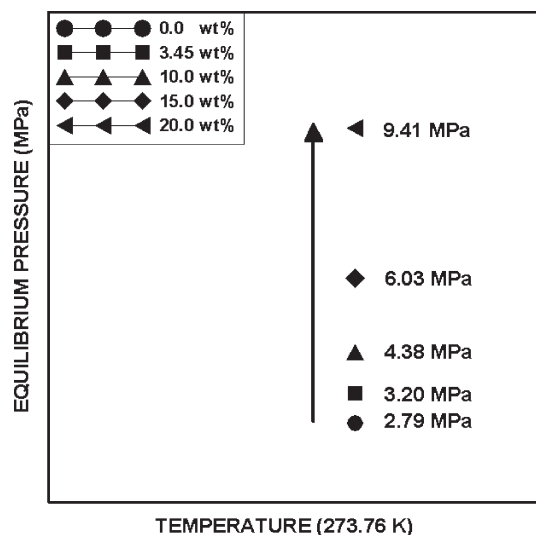
**Figure 7.** Change of the equilibrium pressure because of various salinities under isothermal conditions (273.76 K).



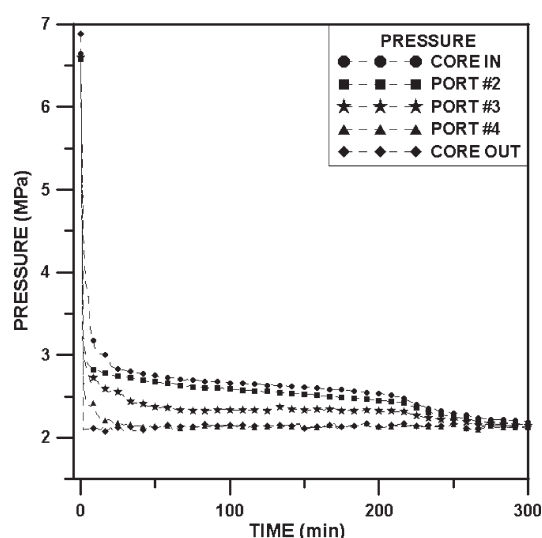**Figure 9.** Pressure behavior during the first 2 min (run 2C).



**Figure 8.** Dissociation pressure of run 2C.

experiment is to examine how brine facilitates the dissociation process and not the formation process, NaCl was not added while gas hydrate was being formed. It would have also been meaningless to have added NaCl during the formation process in the first place, because gas hydrates have special lattice structures that involve only $H_2O$ and $CH_4$ (or other gas) molecules.[20] Although, the salinity of brine before and after the run could be changed, the decrease of the salt concentration in these experiments was not measured because there was no purpose to obtain the quantitative correlation for the effect of residual salt. The field of observation of this study is the degree of influence that each concentration of brine has on the productivity of gas hydrates.

Figure 8 presents the dissociation pressure curve recorded in run 2C. Gas hydrate was formed in sandstone with a permeability of 89 md, porosity of 13.7%, and water saturation of 66%. Then, brine of 15.0 wt % concentration was injected, and the core underwent depressurization down to
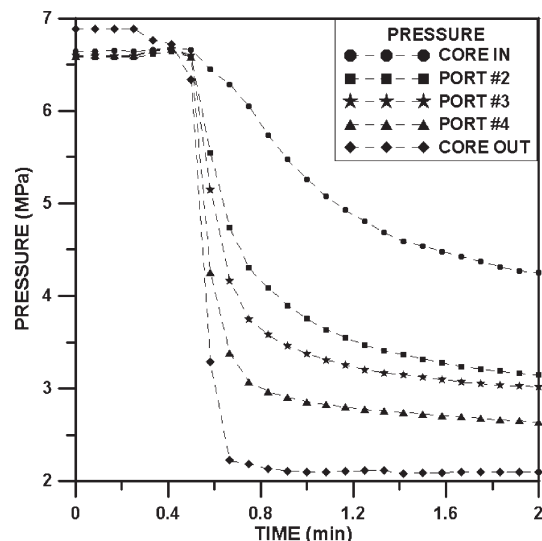
2.07 MPa. Observing the dissociation pressure, it was apparent that the dissociation occurred gradually in the direction from the core-out to the core-in. During hydrate dissociation, the hydrate can quickly reform because the reaction is endothermic; however, such an interval did not appear explicitly in this run. The pressure drop occurred in all ports as soon as the initial outlet valve was opened.

Figure 9 presents the pressure behavior of run 2C during the first 2 min. As indicated by the graph, as soon as the outlet valve was opened (that is, as soon as the production began), the desired gas that dissociated beforehand started to discharge near ports 4, 3, and 2 and the core-in, in the order listed. When taking these results to the actual production field, engineers must take care to prepare against such a "kick" effect that they could encounter. It is worthy to mention that the gas hydrates near the core-in [the point farthest from the core-out (the injection point)] did not seem to be much influenced by the brine effect. This implies that the gas hydrate in this area remained in its stable condition. Therefore, it seems difficult to fully dissociate gas hydrates using only the brine injection scheme. As for such a hydrate area that cannot sufficiently benefit from the brine injection, other methodology may be implemented.

**Effect of the Brine Salinity on Gas Production.** The addition of brine or chemical additives can shift the equilibrium curve of gas hydrates to promote dissociation, as shown in Figure 1. However, the production from such dissociation caused by shifting of equilibrium curves needs to be verified in terms of how it affects the real gas productivity. To validate its applicability to real productions, a production experiment using identical depressurization and varying brine concentrations has been conducted. Figure 10 shows the cumulative gas production from a gas hydrate injected with different brine concentrations in a 71 md core sample. Run 1A shows little production for the first several minutes. After 30 min, there is an abrupt increase in dissociation as the hydrate dissociated at the highest rate. The fluid flow within the core may be disturbed by the complexity of the microstructure of natural sandstone in initial dissociation. It seems that this phenomenon was shown by this reason. Run 1B had a slightly lower dissociation rate than run 1A. Its cumulative gas production is 0.93 L, which is identical to that of run 1A.

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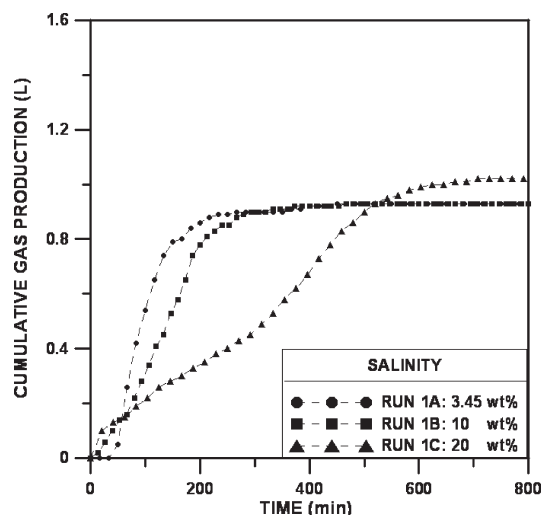
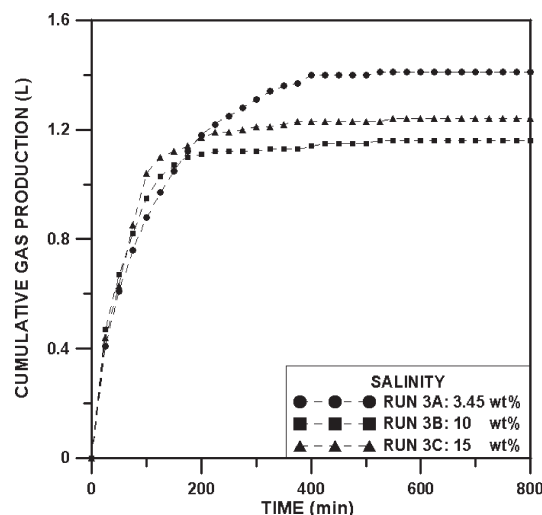**Figure 10.** Cumulative gas production by each salinity in a 71 md core sample.



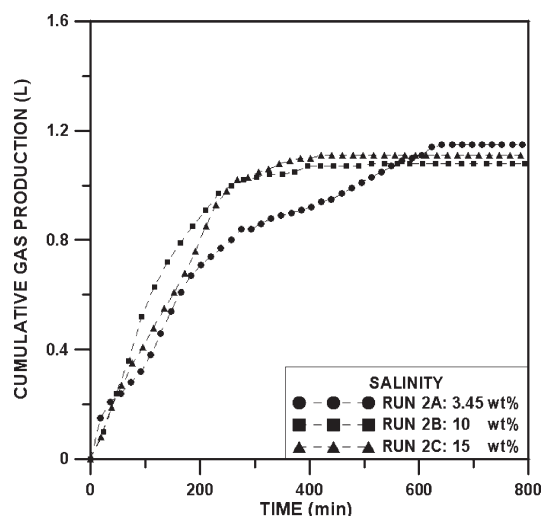**Figure 12.** Cumulative gas production by each salinity in a 169 md core sample.



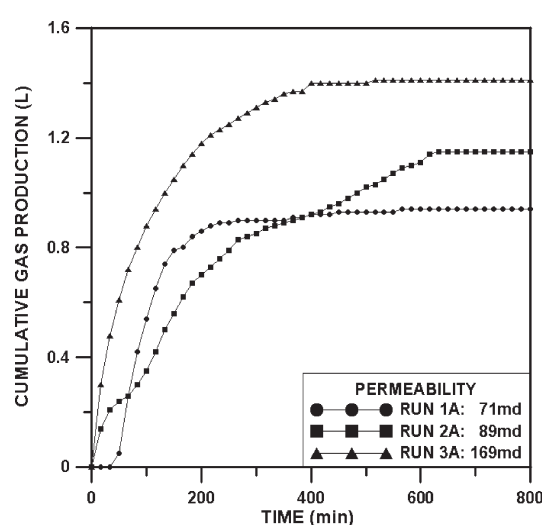**Figure 11.** Cumulative gas production by each salinity in an 89 md core sample.



**Figure 13.** Cumulative gas productions of runs 1A, 2A, and 3A with different permeabilities.

Run 1C, which was carried out with a brine concentration of 20.0 wt %, showed the highest cumulative gas production of 1.01 L. However, it takes about 3 times as long to fully dissociate as does run 1A. Although the cumulative gas production of run 3A is the highest of the three runs, its productivity is the lowest because it took the longest to complete its dissociation. The gas hydrate dissociation rate is one of the most significant factors in its production. The results indicate that, although NaCl in the brine speeds up the dissociation, if it is added excessively, it will aversely affect the process. When NaCl is abundant, NaCl remaining after it has promoted that dissociation can disturb the fluid flow within the cores, which can decrease gas production. In other words, NaCl molecules act as an impermeable layer that exists between gas phases within the core, which significantly reduce the observed permeability.

Figure 11 presents the cumulative gas production as a result of the brine concentration in an 89 md core sample. Unlike Figure 10, run 2A with 3.45 wt % shows the highest cumulative gas production of 1.11 L. Run 2C with 15 wt % is a little higher than run 2B with 10 wt %, but the difference is

not significant. Figure 12 shows the cumulative gas production as a result of the brine concentration in a 169 md core sample. It has a similar tendency, in which run 3A with 3.45 wt % shows the highest cumulative gas production, as Figure 11. However, it is noted that the dissociation time is shorter and the amount of gas production is much more than Figure 9. Here, the tendency of increasing gas production by each concentration is not fully consistent in each core sample group. The core sample with runs 1A, 1B, and 1C has 31% porosity, unlike the other runs. Unfortunately, we cannot use sandstone core samples with different permeability under the same porosity precisely, because natural sandstone has little heterogeneity basically. It may be assumed that this experimental limit causes different tendencies. Nonetheless, it is believed that the effect of the brine salinity on gas production has relevant results because all experimental conditions are the same, except brine salinity, in each run with the same core sample. In conclusion, the titratable range of the brine concentration matching the conditions of this experiment is estimated around 3.45 wt %. A certain concentration where the increase of performance does not
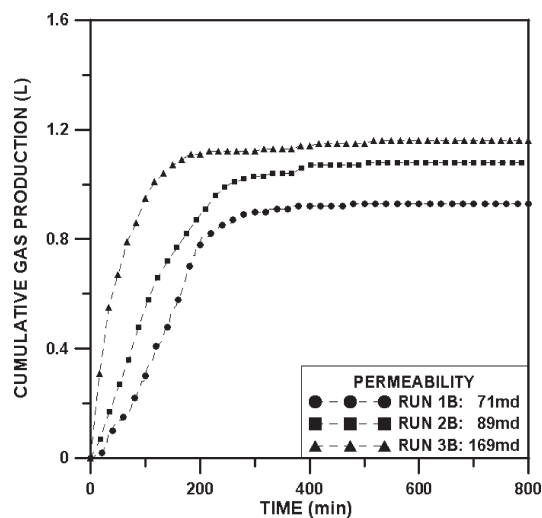
**Figure 14.** Cumulative gas productions of runs 1B, 2B, and 3B with different permeabilities.



**Figure 15.** Cumulative gas productions of runs 1C, 2C, and 3C with different permeabilities.

occur exists, and the increase of performance beyond this is rarely shown, just like the results of previous research.[13] However, the value in which salinity has little effect is not in consistency probably because the experimental environment and the conditions, such as brine temperature, rock properties of the sample, etc., are not fully united with their experiments.

**Effect of the Permeability on Gas Production.** To analyze how the permeability of the rock affects the gas production during brine injection, gas production experiments using identical brine salinity have been conducted in porous rocks with varying permeability. Figure 13 indicates the cumulative gas productions of runs 1A, 2A, and 3A, each with permeability of 71, 89, and 169 md, respectively. The recording was held only during the first 800 min. Even though run 1A had a relatively high porosity, it had a cumulative gas production of only 0.94 L, which was the lowest of the three runs. As for runs 2A and 3A, which have very close porosity, they had a cumulative gas production of 1.15 and 1.41 L, respectively. The former value is 81% of the latter. In the figure, only run 2A shows three inflection points during production. As for run 3A, it shows a continuous and gradual production. The only variable in these two runs was rock permeability. Figure 14 shows cumulative gas productions of runs 1B, 2B, and 3B with different permeabilities. From the figure, one can see the same tendency that, as permeability increases, the amount of gas production increases, as shown in Figure 13. It is observed that, as permeability increases, the dissociation time tends to gradually decrease. In the case of run 3B, which has the highest permeability of the three runs, it reveals 1.24 L of cumulative gas production. This value is approximately 10% higher than that of run 2B. Figure 15 presents cumulative gas productions of runs 1C, 2C, and 3C. It turned out that identical tendency is shown as in Figure 14. As permeability increases, the cumulative gas production increases and the dissociation time decreases. Especially, in the case of run 3C, shortening of the dissociation time was revealed, with a difference being 3 times more than run 1C. Figure 16 compares the gas productivity by the effect of permeability. Productivity has been defined by dividing the amount of cumulative gas production until the completion of dissociation by the time it took until the completion of dissociation.
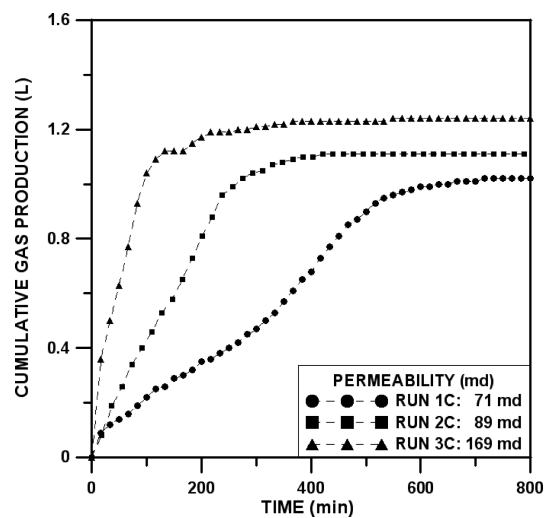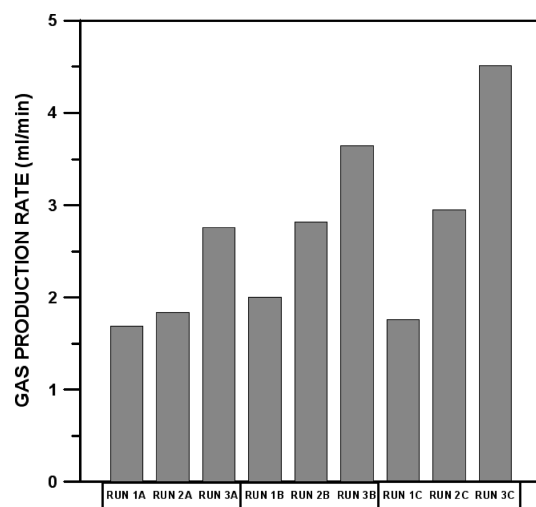


**Figure 16.** Comparison of the average gas production rate by each permeability.

This concept is equivalent to that of the average production rate from beginning to end of dissociation. As depicted on the figure, gas productivity increases as the permeability increases in each run group. Especially, runs 3A, 3B, and 3C, which have the highest permeability, have a value 23−35% higher than that of runs 2A, 2B, and 2C. From the results, it is experimentally verified that the permeability of the rock, which describes the connectivity of pores, is one of the most important factors that influence production from gas hydrates. It suggests that, during gas production from a gas hydrate reservoir, the dissociation methods must be conducive to increase or at least maintain the reservoir permeability.

## Conclusions

In this study, an experimental apparatus has been designed and set up so that gas recovery and production tests could be conducted in a porous rock. Production behaviors in two sets of assessments were observed: dissociation by injecting various concentrations of brine and dissociation in cores with varying permeability. The conclusions drawn from the research are as follows: (1) There is an abrupt pressure drop at

the early stage of gas hydrate production by brine injection. Although pressure fluctuations can occur because of hydrate reformation caused by the endothermic nature of dissociation during the dissociation period, no explicit oscillation in pressure was observed. (2) Recovery characteristics and the effect of brine salinity on gas production from brine injection with various concentrations have been analyzed. The results present that the brine concentration of 3.45 wt % suffice to significantly increase the gas production rate in porous rock. (3) It has been confirmed that, if the brine concentration is excessively high, the productivity is lowered because the average gas hydrate dissociation rate is reduced. This means that excessive NaCl molecules disturb fluid flow between pores and, thus, significantly reduce the permeability. (4) Considering the effect of permeability in productivity of gas hydrate, it is obvious that an increase in the permeability caused an increase in gas production and a decrease in the dissociation rate. A difference in the production rate was also observed up to 35%. This indicates that permeability is one of the most important factors that determine the gas hydrate productivity.

**Nomenclature**

$n$ = moles of gas
$V$ = volume occupied by gas
$R$ = universal gas constant
$T$ = temperature
$P$ = pressure
$z$ = gas compressibility factor ($z$ factor)
$\phi$ = porosity
$V_b$ = bulk volume of the sample
$V_s$ = volume of the solid
$k$ = permeability
$\mu$ = fluid viscosity
$Q$ = fluid flow rate, in volumes per unit time
$\Delta P$ = pressure difference across the length of the specimen
$A$ = cross-sectional area
$L$ = length

*Subscripts*

i = initial
f = final