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Analysis of Pore Structure and Water Permeation Property of a Shale Rock by Means of X-Ray CT

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Abstract

In this study, pore structures and water permeation property of a shale rock are analyzed by means of X-ray CT. The target samples are produced Kushiro district in Hokkaido, Japan. Underground of this area is consisted by Cretaceous formation. Firstly, one dimensional permeation tests were performed by using core sample retrieved from Cretaceous formation, and intrinsic permeability was evaluated. Secondly, the internal structure of rock samples was observed by X-ray CT scanner, and porosity distributions were also evaluated by comparing CT image data between the dry and the water-saturated conditions. It was found that Cretaceous formation has relatively low permeability as $k=10^{-19} \sim 10^{-18}$ m². It was also found that the porosity of each sample was approximately 7%~13%, however, porosity distribution was not uniform, and was strongly influenced by density distribution in samples.

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1. Introduction

Natural gas/oil has been developed all over the world, and most of gas/oil has been supplied from so called conventional natural gas/oil deposits for long time. However, as technology and geological knowledge is advancing, many unconventional natural gas/oil deposits have been discovered, and total amount of supplied energy is increasing [1]. The representative of unconventional natural energy deposits is shale rock layers [1]. In Japan also,

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there are several promising shale rock layers as gas/oil reservoirs [2], and certain amount of gas/oil deposits are inspected.

In this study, pore structures and water permeation property of a shale rock are analyzed by means of X-ray CT. The target samples are produced Kushiro district in Hokkaido, Japan. Underground of this area is consisted by Cretaceous formation. This is mainly formed by sandy shale and it is spreading under Kushiro coal seam. It is estimated that huge amount of methane gas exists in the Cretaceous formation [3], however, the origin of the methane and the storage process in the formation including its total amount are still not clear. The purpose of this study is to obtain fundamental characteristics or properties of the shale rocks located at Kushiro, such as nominal porosity, permeability, porosity and density distributions. Here, nominal porosity is evaluated through water absorption tests. One dimensional permeation tests are performed, and intrinsic permeability of the shale samples are evaluated. In order to inspect the internal structure of the shale, X-ray CT method is applied. From the CT image data, the internal structure of rock samples is discussed, and porosity distributions are also evaluated by comparing CT image data between the dry and the water-saturated conditions.

2. Rock sample and its porosity

The target samples are produced at Kushiro district in Hokkaido, Japan (Fig. 1). Here, Kushiro Coal Mine Co. Ltd. is producing approximately 500 000 tons of coal every year, and the bed rock below the coal seams is consisted by Cretaceous formation. This is mainly formed by sandy shale and the shale layer spread up to 4 000 m depth. In this shale layer, it is expected a certain amount of methane gas is stored, however, precious characteristics of the shale is still unrevealed.

The rock samples used in this study are drilled from 160m to 250 depth of this site. An example photo of the sample in dry condition is shown in Fig. 2. This is dilled vertical direction. As this figure shows, the grain size is very small. Some sedimentary layers are also observed perpendicular to the core axis, that is horizontal direction, in some samples. Here seven pieces of rock samples (named Sample A to G) are used for following permeation tests and X-ray CT analysis.

In order to evaluate porosities of samples, water absorption tests are conducted, and the porosity is evaluated from the weight differences between dry condition and fully saturated condition. Here, water absorption tests are conducted under the vacuum condition, and the saturation process is also monitored. The temporary change of weight is shown in Fig.3. The vertical axis represents the normalized weight and that the temporal weight M is normalized by initial weight M_0 . As this figure shows, the characteristic point of the samples is that the weight gradually and slowly increases at the beginning of absorption process, and that it takes more than 20 to 25 days until the sample is fully saturated. The evaluated porosities together with weight change during absorption tests are summarized in Table 1. The evaluated values of porosities are approximately in the range between 7% and 13%, and the values are almost same as the ones of shales which product oil and gas [4, 5, 6].



Fig. 1. Location of Kushiro.



Fig. 2. View of Kushiro shale sample.

Sample	Dry Weight	Weight in saturated	Volume	Porosity, %
А	146.78 g	152.69 g	59.50 cm ³	9.92 %
В	165.85 g	174.80 g	71.53 cm ³	12.51 %
С	164.76 g	170.38 g	64.91 cm ³	8.66 %
D	172.09 g	177.96 g	67.58 cm ³	8.69 %
Е	150.43 g	156.26g	60.61 cm ³	9.66 %
F	202.22 g	208.01 g	78.97 cm ³	7.33 %
G	198.86 g	205.12 g	79.16 cm ³	7.91 %

Table 1. Weight change between dry and water saturated conditions and evaluated porosity.



Fig. 3. Temporal change of weight during water absorbing tests.

3. One-dimensional permeation tests

Authors have been introduced a specific type of permeation apparatus and rock samples in order to evaluate intrinsic permeability as shown in Fig. 4 and 5 [7, 8]. Main part of the pressure vessel is made by CFRP (carbon fiber reinforced plastic). Therefore, the total weight of the vessel part is only 7kg, and it is very easy to handle. Rock samples are processed as Fig. 5 shows. The side is completely sealed by resin. Drain pipe is installed at one end, and this part is connected to the pressure vessel (Fig. 4). Another end is free, and the water is permeating into a rock sample from this free surface. Since the rock sample is installed in the vessels as Fig.4 shows, confining pressure and inlet pressure is simultaneously acting on the rock sample. One-dimensional permeation tests were conducted under the pressure constant condition. Here, 1MPa of pressure difference is given to the rock sample and measure the flow rate which penetrate rock sample.

The evaluated intrinsic permeability of samples is shown in Fig. 6. As this figure shows, intrinsic permeability is in the range of 10^{-19} m² to 10^{-18} m² except for sample E, and it is found that generally Kushiro shale has relatively lower permeability.



Fig. 4. Experimental apparatus for one dimensional permeation tests. This system consists of pressure vessels made by CFRP (Carbon Fiber Reinforced Plastic) and syringe pumps.



Fig. 5. Rock sample for one-dimensional permeation tests.



Fig. 6. Evaluated intrinsic permeability of Kushiro shale.

4. Pore-structure analysis by means of X-ray CT

4.1. X-Ray CT system

In order to analyse the internal structure or pore distribution of Kushiro shale samples, X-ray CT method is applied in this study. As is well known, X-ray CT is one of the most useful and powerful tool among non-destructive imaging techniques. It is also important that X-ray CT is the method to detect density distribution inside of materials.

Rock mechanics group of Kumamoto University have been operating X-ray CT systems almost 20 years, and we have applied for various kinds of analysis [9, 10]. Here, industrial use X-ray CT scanner system (TOSCENNER-20000RE) is used (Fig. 7). The maximum X-ray tube voltage of the system is more than twice as higher as the medical use (300kV/2mA). Therefore, it is suitable for the visualization of relatively high density materials such as rock samples. Other important factor of X-ray CT scanner is the number of the pixels which composes a CT image. This has strong relation with the spatial resolution of CT images. Generally an image is consisted by $n \ge n$ pixels, and the maximum number is n = 2.048 in the case of our system. As for the visualization area, two modes such as $\phi = 150$ mm and 400mm can be selected. If the pixel number of n = 2.048 and visualization area of $\phi = 150$ mm are chosen, the minimum pixel size of 0.073mm is available. CT image is generally expressed by CT values, that is, each pixel/voxel which consists an image has one CT value. In the case of the X-ray CT system of Kumamoto University, CT values *CT* are given by the following equation.

$$CT = K \cdot \frac{f_m - f_m^{water}}{f_m^{air} - f_m^{water}}$$
(1)

where f_m is a X-ray attenuation coefficient at arbitrary position, f_m^{air} is the X-ray attenuation coefficient of air, f_m^{waterr} is the X-ray attenuation coefficient of water. These values are proportional to density, and the heaver material has higher value of X-ray attenuation coefficient. *K* is the coefficient determined by each CT machines or its settings. In the case of CT scanner in this study, the value of *K* is set to be 1 000. That is, 1 CT value represents 1/1 000 of the density difference between the air and water, and it corresponds to the density resolution of the scanner.

4.2. Evaluation of porosity distribution

In order to obtain porosity distribution, it is also necessary to take two images of a sample in different conditions, that is, dry condition and water saturated condition as same as porosity evaluation stated above. Therefore, CT images are taken in both conditions for each rock sample.



Fig. 7. Outlook of industrial use X-Ray CT scanner operated by Kumamoto University (TOSCANNER-20000RE).

As stated before, a CT image is consisted by $n \ge n$ pixels, and the address (i, j) is given to each pixel. Here, the CT value at this address is denoted by $C_d(i, j)$ and $C_w(i, j)$. The subscript "d" and "w" represent the CT value taken under dry and water saturated condition respectively. Porosity of a sample is obtained from the weight difference between dry and water saturated conditions, the same idea is also applied to the CT image analysis. The increment of CT value at address (i, j) under dry and water saturated conditions is denoted by

$$\Delta C(i, j) = C_{w}(i, j) - C_{d}(i, j)$$
⁽²⁾

As shown in equation (1), CT value is defined as $1/1\ 000$ of the density difference between water and air. When the porosity at the address (i, j) is denoted by $\phi(i, j)$, this is given by

$$\phi(i,j) = \frac{\Delta C(i,j)}{1000 \times \rho} = \frac{C_w(i,j) - C_d(i,j)}{1000 \times \rho}$$
(3)

where ρ is the specific gravity of water.

4.3. Evaluation of porosity distribution

The representative results of CT images of rock samples in dry condition and the porosity distribution obtained by equation (3) are shown in Fig. 8 and 9. As for each CT image ((a) of Fig. 8 and 9), brighter region represents the higher density region. Porosity distribution ((b) of Fig. 8 and 9) is expressed in the range between 0% and 15% as the scale on the left hand side shows. From these figures, it is found that the porosity distribution is strongly influenced by density distribution.

In the case of sample B (Fig. 8 (a)), left hand side of the sample is obviously blighter, and it represents the density on the left hand side is higher than right hand side. It was hardly confirmed from visual inspection on the sample surface, and it is proved that X-ray CT is very useful tool to detect internal structure of the sample. Porosity distribution (Fig. 8 (b)) also shows same tendency. The lower density region, that is right hand side, has relatively higher porosity. The mean value of porosity obtained from the result of Fig. 8 (b) is 11%. This is almost same as the value which is obtained from weight difference between dry and wet conditions as shown in Table 1.

In the case of sample F (Fig. 9), the relation between density and porosity distributions shows same tendency as sample B. In this case, CT image is brighter than sample B in whole area. It represents the density of sample F is higher than the one of sample B. As Table 1 shows, mean porosity of sample F is about 7% and obviously lower than the one of sample B. By comparing porosity distribution ((b) of Fig. 8 and 9), it is also confirmed. In the region inside of dotted square, darker line surrounded by blighter region can be confirmed. This is the lower density region surrounded by higher density part. However, the porosity of the lower density region is evaluated as lower than the one of the surrounding higher density region. If it is assumed that the high density region has lower permeability, the region of lower density is isolated from the other region. Therefore, water can hardly permeate into this region during absorption process. As a result, the porosity of the region is evaluated lower by taking difference between dry and saturated condition.

As stated above, mean porosity of sample B is larger than the one of sample F. However, the value of intrinsic permeability of sample B is smaller than the one of sample F as shown in Fig.6. This might represent that the pores in the sample are connected by very narrow throats. In this case, water permeation is strongly governed by the narrow connections of pores rather than nominal porosity. At this moment, it is impossible to visualize the connections or networks of pores by our CT system. We authors are planning to introduce new nano-focus CT scanner system, and we would like to try more precise analysis in near future.



Fig. 8. CT image and porosity distribution for sample B.



5. Conclusions

In this study, pore structures and water permeation property of a shale rock which is produced in Kushiro, Hokkaido, are analyzed by means of X-ray CT.

The porosity is evaluated by the weight differences between dray and water saturated conditions. The evaluated values of porosities are approximately in the range between 7 % and 13 %, and the values are almost same as the ones of shales which product oil and gas.

One-dimensional permeation tests were conducted and intrinsic permeability was evaluated. The intrinsic permeability is in the range of 10^{-19} m² to 10^{-18} m² except for one sample, and it is found that generally Kushiro shale has relatively lower permeability.

Internal density and porosity distribution were analyzed by X-ray CT method. It was found that the porosity distribution is strongly influenced by density distributions, and that the lower density region tends to show higher porosity region. However, even though the region where density was lower, some regions were evaluated as low porosity regions. It might happen when the low density region is isolated by low permeable regions.

References

- Website of US Energy Information Administration, The page of "World Shale Resource Assessments", https://www.eia.gov/analysis/studies/worldshalegas/
- [2] R. Kamitsuji, T. Okajima, S. Yokoi, Acid stimulation of Onnagawa tight oil formation in Ayukawa field, Japan, Proceedings of Asia Pacific Unconventional Resources Conference and Exhibition, vol. 2, 2013, pp. 954–957.
- [3] N. Sutekazu, T. Toshio, Several Cretaceous Oil and Gas Indications, Recently found in Hokkaido, The Japanese Association for Petroleum Technology 34 (1969) 240–248.

- [4] W. Kanitpanyacharoen, R. Vasin, H.-R.Wenk, D.N. Dewhurst, Linking preferred orientations to elastic anisotropy in muderong shale, Australia, Geophysics 80 (2014) C9–C19.
- [5] S. Morsy, J.J. Sheng, R.O. Ezewu, Potential of waterflooding in shale formations, in: Society of Petroleum Engineers 37th Nigeria Annual Int. Conf. and Exhibition, NAICE 2013 - To Grow Africa's Oil and Gas Production: Required Policy, Funding, Technol., Techniques and Capabilities, vol. 1, 2013, pp. 114–122.
- [6] J. Zhao, D. Yang, Z. Kang, Z. Feng, A micro-CT study of changes in the internal structure of Daqing and Yan'an oil shales at high temperatures, Oil Shale 29 (2012) 357–367.
- [7] A. Sato, K. Tanaka, T. Shiote, Analysis of residual CO2 trapping in the porous media by means of X-ray CT, Journal of MMIJ 126 (2010) 640–646.
- [8] A. Sato, H. Yoshitake, T. Shiote, K. Sasa, Development of the confined pressure type of multi-purpose rock flow system and its application to the evaluation of permeability of various geomaterials, in: ISRM International Congress on Rock Mechanics - Harmonising Rock Engineering and the Environment, vol. 1, 2011, pp.1357–1360.
- [9] A. Sato, O. Aydan, An X-Ray CT Imaging of Water Absorption Process of Soft Rocks, Unsaturated Soils: Research & Applications 1 (2014) 675–678.
- [10] A. Sato, K. Ikeda, Visualization of diffusion phenomena in the porous media by means of X-ray CT, Canadian Geotechnical Journal, 2015.