Experimental Studies on Retardation Properties of Granite Specimens from Grimsel Test Site, Switzerland - 9368

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ABSTRACT

Geological formations of igneous rocks, like granites, have been considered as candidate formations for geological disposal of high level radioactive wastes in many countries. Transport of radionuclides through an igneous rock formation is principally controlled by fluid flow along fracture networks, permeation and diffusion in the directions perpendicular to fracture surfaces, sorption onto geological materials and chain decay. Among these mechanisms, permeation and diffusion in the directions perpendicular to fracture surfaces, and sorption onto geological materials control the retardation properties of a geological formation. In our study, systematic laboratory experiments and analyses aimed to characterize the retardation properties of rock specimens taken from the Grimsel Test Site, Switzerland, have been planned. This paper presents the results obtained from the primary stage of the study, which include chemical and physical properties, and effects of stress and stress history on permeability of test specimens. The results illustrated that: 1) The porosity and wave velocities of Grimsel granite are relatively low compared to average values of igneous rocks indicating that micro-cracks can potentially exist within grain minerals. This inference is further supported by the X-ray scanning visualization. 2) The permeability of Grimsel granite is very sensitive to stress condition. Under the test condition of confining pressures up to about 60MPa, the intrinsic permeability of Grimsel granite ranged from the order of 1E-15 to 1E-19 m^2. 3) Permeability is not a simple function of effective confining pressure. It is significantly sensitive to confining pressure at low pressure levels. Besides, the permeability is hysteretic depending on stress history. 4) Similar to the permeability, diffusion coefficient of rock specimens can also be significantly affected by the confining pressure conditions. Laboratory diffusion tests should be designed to incorporate the effects of stress conditions.

INTRODUCTION

Geological formations of igneous rocks, like granites, have been considered as candidate formations for geological disposal of high level radioactive wastes in many countries, such as Canada, Sweden, Switzerland, Finland, Czech, China, India, Bulgaria, Ukraine, France and Japan [1]. Safety assessment of facilities associated with geological disposal of high level radioactive waste is generally performed through mass transport simulations combined with uncertainty and sensitivity analyses. For an assessment to be reliable and/or to reduce the uncertainty of a simulation, mathematical models describing the mechanisms of mass transport in a geological formation should be appropriate, and the values of parameters input for the simulation should be representative of in situ conditions.

The transport of radionuclides through an igneous rock formation is principally controlled by fluid flow along fracture networks, permeation and diffusion in the directions perpendicular to fracture surfaces, sorption onto geological materials and chain decay. Among these mechanisms, permeation and diffusion in the directions perpendicular to fracture surfaces, and sorption onto geological materials control the retardation properties of a geological formation.

Besides *in situ* tests which are generally cost intensive and time consuming, laboratory determination of the permeation and diffusive properties of a rock matrix can be performed by means of permeability [e.g., 2-7] and diffusion tests [e.g., 8-14], respectively. Since these techniques have long been used in practice, numerous valuable publications can easily be found through literature surveys. Although both laboratory permeability and diffusion tests are considered to be well-established and widely adopted approaches for characterizing the transport properties of geological materials, they do not necessarily provide accurate values of parameters to be determined without careful examination of calculation and/or test conditions. For example, a simple mistake in calculation associated with logarithm and natural logarithm functions was made in a frequently cited literature published in the Journal of Geophysical Research by Brace et al. in 1968 [15]. This miscalculation underestimated the values of permeability of Westerly granite by a factor of 2.3[3, 16]. The calculated value of permeability from a transient pulse permeability test is also proportional to the value of fluid compressibility which is a function of the compressibility of water, compressibility of the tubing system and pore pressure imposed within it. The use of a nominal value of compressibility for pure water to calculate the value of permeability from a transient pulse permeability test may underestimate the value of permeability by a factor of several to several hundred times depending on the testing system and test conditions[4, 17]. Errors in determining the diffusive properties from a laboratory diffusion test may also occur. Recent theoretical evaluation of a conventional through diffusion test illustrated that disregarding the increase of tracer concentration in the downstream measurement cell may lead to underestimation of both the effective diffusion coefficient and rock capacity factor [18]. Moreover, most diffusion tests are performed under atmospheric conditions except for a few case studies [7, 19, 20]. From the author’s viewpoint, detailed studies on both theory and experimental techniques for accurate determination of the transport properties of rock matrix are still of fundamental importance for increasing the reliability of safety assessments.

In this study, systematic laboratory experiments and analyses aimed to characterize the retardation properties of rock specimens taken from the Grimsel Test Site, Switzerland, have been planned. This paper presents the results obtained from the primary stage of the study, which include chemical and physical properties, and effects of stress and stress history on permeability of test specimens.

EXPERIMENTAL PLAN

To systematically evaluate the chemical, physical, hydraulic and diffusive properties of Grimsel granite, a series of laboratory analyses and experiments are planned. Analysis and/or test items planned in this study are summarized and tabulated in Table 1 together with the methods to be used.

The comprehensive experimental program planned in this study aims not only to characterize quantitatively individual properties of Grimsel granite, but also to find possible correlations between different properties. Such kind of correlations may be very useful for predicting one property by measuring another correlated property, and for interpreting results obtained from field investigations. For example, measurements of the physical properties of rock specimens, such as the wave velocities and resistivities, are especially useful because they may provide correlations with properties routinely measured in the field during site selection and/or site characterization. Laboratory measurements of other physical parameters, such as effective porosity and density, are also of fundamental importance because effective porosity is a basic parameter that controls fluid flow and mass diffusion within the rock, and density is required for calculating the ultrasonic dynamic elastic constants of rock from wave velocities to assess rock strength and for assessing the rock capacity factor. In addition, if the correlation between changes in permeability and changes in effective diffusion coefficient due to changes in stress condition can be determined, it would be very useful in practical applications. This is because the time required for performing a permeability test is significantly shorter than that required for a diffusion test, and a permeability test is much easier to be performed under high confining and pore pressure conditions that simulate ground pressures at depths.

Table 1 Analysis and/or test items planned for characterizing chemical, physical, hydraulic and diffusive properties of Grimsel granite.

Test item		Test method
Chemical property	Mineral composition	X-ray diffraction analysis (XRD)
	Chemical composition	X-ray Fluorescence Analysis (XRF)
Physical property	Porosity & density	Water saturation method
	Velocity	Ultrasonic wave measurement (P&S waves)
	Resistivity	Four terminals/electrodes method
	Specific surface area	Gas adsorption method, BET
	Micro structure	High resolution X-ray scan
Hydraulic property	Air permeability	Permeability tests under high confining pressure conditions
	Water permeability	Permeability tests under high confining & pore pressure conditions
Diffusive property	Effective diffusion coefficient	Diffusion tests under atmospheric & high confining pressure conditions
	Rock capacity factor	

Although currently available data to be presented in this paper are not sufficient to verify the last point, the author thinks that it could be an effective way to predict the effective diffusion coefficient at high pressure conditions through a diffusion test under atmospheric condition, and experimentally determined stress-dependent variations in permeability.

EXPERIMENTAL PROGRAM AND RESULTS

Test Specimens

Rock samples were taken from the underground research and development laboratory at the Grimsel Test Site, Switzerland, where a Long Term Diffusion (LTD) test which aims to obtain quantitative information on matrix diffusion in fractured rock under *in situ* conditions is currently in progress (see http://www.grimsel.com/ltd/ltd_intro.htm for reference). Fig. 1 shows a picture of granite rock samples taken from a drillcore numbered LTD 06.001 as an example.

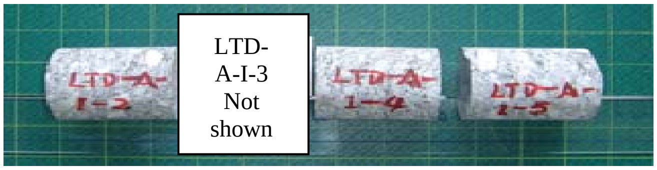

Fig. 1 Granite rock samples taken from the Grimsel Test Site, Switzerland.

Test specimens were prepared from these samples by simply polishing the ends of individual samples to make them parallel. Non-destructive tests such as physical and hydraulic tests were first performed, and chemical analyses were performed later because rock samples have to be crushed and powdered for chemical analyses.

Chemical Analyses

XRD analyses illustrated that minerial composition of Grimsel granite mainly contains quartz, feldspars and biotite. Quantative XRF analyses illustrated that Grimsel granite contains the follow chemical compositions.

SiO_2 : 72.98% Al_2O_3 : 14.37% Na_2O : 4.92% K_2O : 3.51%
Fe_2O_3 : 2.40% CaO : 1.88% MgO : 0.34% TiO_2 : 0.30%

Physical Experiments

The effective porosity was determined by means of the water saturation method. To avoid thermal-induced cracking, drying was performed below $60\,^oC$ for more than 48 hours until there was no more loss of mass. As for saturation, test specimens were first evaluated for more than 24 hours and then saturated for more than 48 hours until there was no more increase in mass. To ensure accuracy, a high resolution electronic balance with a resolution of 0.001g was used for weighing specimens lighter than 300g. The effective porosity, η_e, and density, ρ, were then calculated from the difference in weight between over-dried and water-saturated specimens with the following equations:

$$\eta_e = \frac{W_3 - W_2}{W_3 - W_4} \times 100\% \tag{1}$$

$$\rho = \frac{m}{V} = \frac{W_2}{W_3 - W_4} \tag{2}$$

Where, W_2, W_3 and W_4 are the weight of an oven-dried specimen in air, the weight of a saturated specimen in air and the weight of saturated specimen in water, respectively. The effective porosity and density of Grimsel granite were determined to be 0.69% and $2.71 g/cm^3$, respectively.

The standard test method of ASTM International (D2845) was adopted to determine the pulse velocities of the specimens. The resonance frequencies of the compression and shear wave transducers used in this study were 200 kHz and 100 kHz, respectively. The average velocities of the compression wave (P wave) in saturated and oven-dried Grimsel samples were 4,991m/s and 3,051m/s, respectively, and the average velocities of the shear wave (S wave) in saturated and oven-dried Grimsel samples were 3,647m/s and 2,811m/s, respectively.

Four terminals/electrodes method was used to measure the electrical resistivity. Since the samples were partially dried during transportation from the Grimsel Test Site, test specimens were first completely dried and then saturated with tap water having a resistivity of $37\,\Omega m$. The resistivity determined for saturated Grimsel granite ranged from $4{,}522\,\Omega m$ to $4{,}804\,\Omega m$, with an average of $4{,}797\,\Omega m$.

The gas adsorption method was used to determine the specific surface of, and the volume of micropores and average diameter of micropores within Grimsel granite. The results obtained were $2.41 m^2/g$, $3.86X10^{-4} cm^3/g$ and 77.15nm, respectively, for the three physical parameters.

A high resolution X-ray CT scanner was used to visualize the microstructure within Grimsel granite. Fig. 2 shows the scanned results with a resolution of 13nm.

Permeability Tests

Test Apparatus

Permeability tests were performed by using a gas and water permeability test system, as shown in Fig. 3, for testing rocks in the laboratory [21]. The system consists primarily of the following:

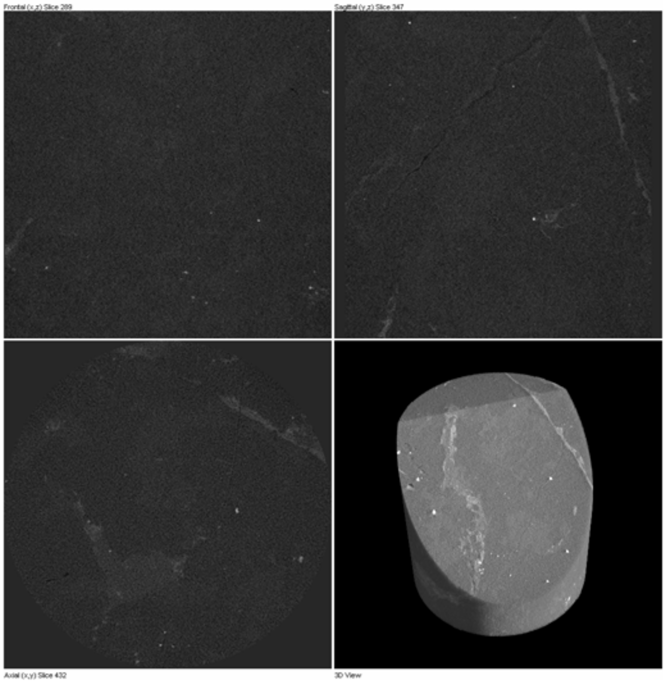

Fig. 2 Visulization of microstructures within Grimsel granite (resolution : 13nm).

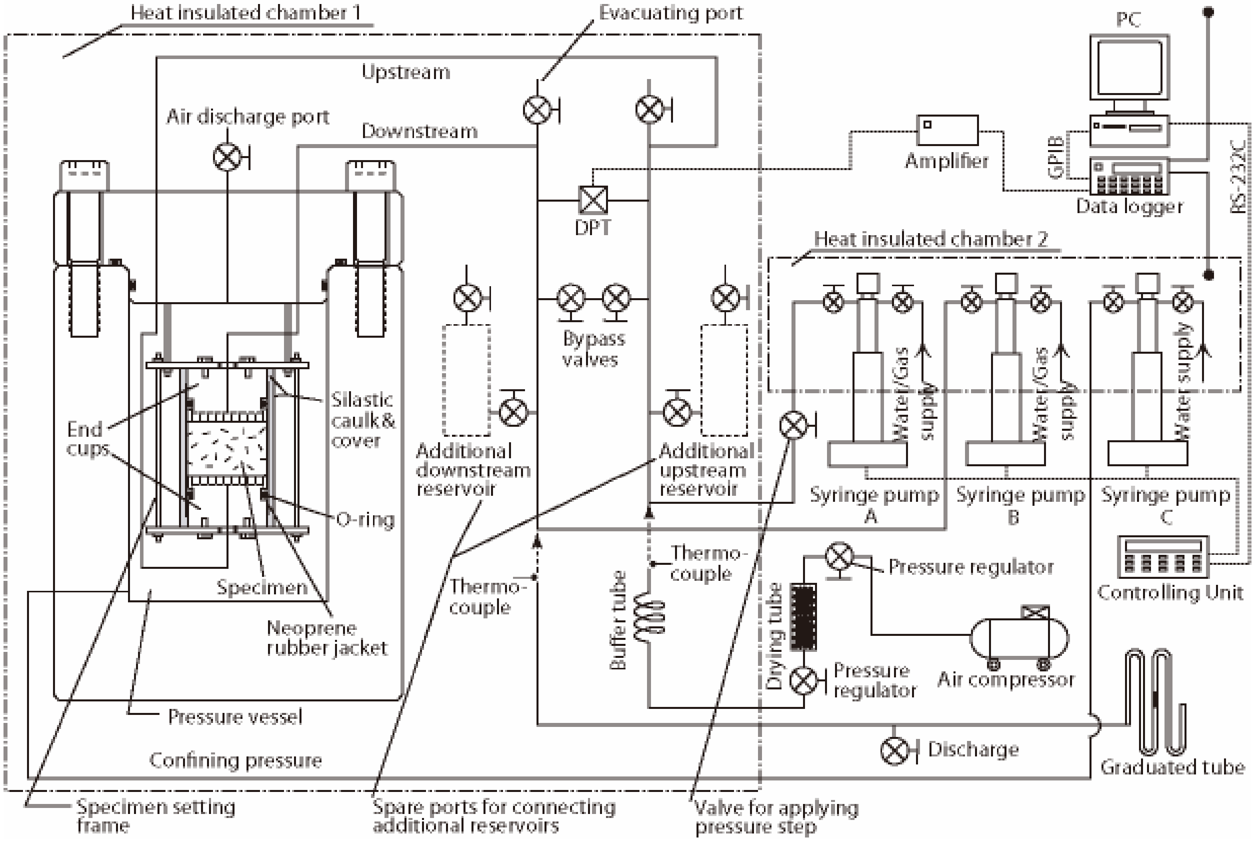

Fig. 3 Schematic of the gas and water permeability test system for rocks.

a pressure vessel and confining pressure generator (syringe pump C) for hydrostatic testing; syringe pumps A and B used for water permeability tests; an air compressor, pressure regulators, drying and buffer tank along with a graduatedtube for the air permeation test; a data logger and a personal computer (PC) along with the associated sensors, such as a differential pressure transducer and thermocouples, for data acquisition and monitoring; and the necessary valves with associated stainless steel or plastic tubing.

The pressure vessel can support hydrostatic pressures higher than 100 MPa. The syringe pumps (ISCO 100DX) pumps have a volumetric capacity of 103 ml, can stand fluid pressures up to 69 MPa, and can generate the same values for confining and pore pressures that simulate ground pressures at depths with a resolution of 1 psi (1 psi=6.9 kPa). A high-linearity differential pressure transducer (DPT) was used to monitor the induced hydraulic head difference across the entire length of the specimen during the air permeation or water permeability test. The computer-based data acquisition and monitoring system can record all of the physical parameters to be measured and monitored at any specified time interval during a water permeability test.

Flow rates of air during the air permeation test were measured by the movement of a plug of manometer oil in the graduated tube.

To eliminate the influence of temperature variation on permeability tests, the pressure vessel, the ancillary plumbing, the buffer tank, and the cylinders of the syringe pumps were insulated in the chambers. The buffer tank was equipped to stabilize the entrance pressure and temperature of air during the air permeation test.

Pressure Conditions for Air Permeation Tests

The effluent pressure was kept free at atmosphere pressure and a relatively small entrance pressure, less than 0.02Mpa, was used for air permeation tests. To investigate the stress and stress histroy on permeability of Grimsel granite, confining pressure was first increased stepwise from 0.069MPa to 57.96MPa, and then decreased stepwise to 0.069MPa.

Results of Air Permeation Tests

Air or gas permeability of the test specimen was calculated from the measured flow rate at steady state with the following equation :

$$K_g = \frac{2\mu_g Q_g p_a l}{A\left(p_0^2 - p_1^2\right)} \tag{3}$$

Where K_g is gas permeability, μ_g is viscosity of air, Q_g is steady-state flow rate of air at atmospheric pressure, l is the length of the specimen, A is the cross-sectional area of the specimen, p_a is atmospheric pressure, and p_0 and p_1 are the absolute pressures at the upstream and downstream ends of the specimen, *i.e.*, the entrance and atmospheric pressures, respectively.

To compensate for the effects of temperature variations during a series of permeability tests, the values of permeability were converted to those at a temperature of 15°C by the following conversion equation:

$$K_{15} = K_T \frac{\mu_T}{\mu_{15}} \tag{4}$$

where k_{15} and k_T are the permeability values at 15°C and the temperature during testing, respectively, $\mu_{15}$and μ_T are the viscosity of air at 15°C and the temperature during testing, respectively.

The results obtained from air permeation tests on Grimsel granite under a series of confining pressure conditions are tabulated in Table 2 and illustrated in Fig. 4.

Table 2 Permeability of Grimsel granite under loading and unloading conditions.

Increasing confining pressure		Decreasing confining pressure	
Confining pressure (MPa)	Permeability (m^2)	Confining pressure (MPa)	Permeability (m^2)
0.069	8.71E-16	57.96	5.33E-20
0.0966	6.73E-16	38.64	9.76E-20
0.1449	4.11E-16	19.32	1.49E-19
0.1932	2.49E-16	9.66	3.99E-19
0.2898	6.61E-17	4.83	7.19E-19
0.3864	2.00E-17	1.932	1.11E-18
0.483	7.63E-18	0.966	1.55E-18
0.966	3.51E-18	0.483	1.88E-18
1.932	3.00E-18	0.3864	1.85E-18
4.83	2.00E-18	0.2898	1.91E-18
9.66	9.56E-19	0.1932	1.83E-18
19.32	4.20E-19	0.1449	1.92E-18
38.64	3.08E-19	0.0966	2.52E-18
57.96	5.33E-20	0.069	2.83E-18

Note: Values of permeability are converted into intrinsic permeability at a temperature of 15°C.

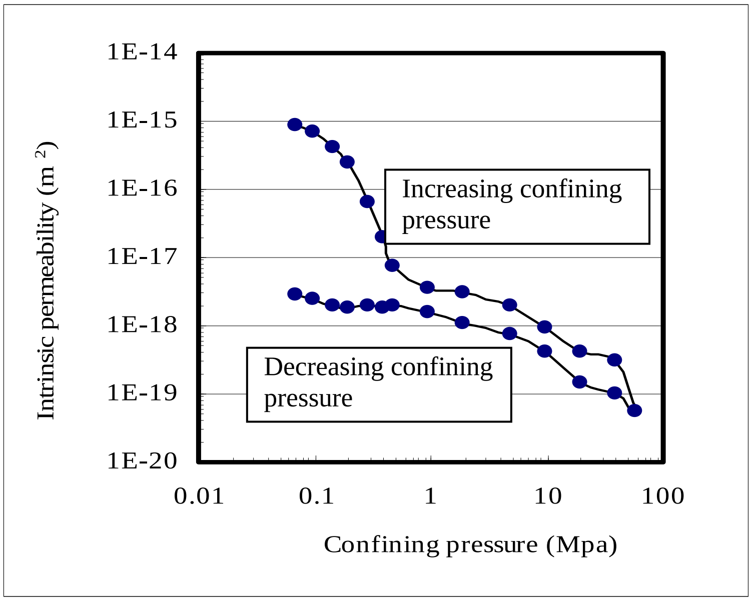

Fig. 4 Effects of stress and stress history on permeability of Grimsel granite.

DISCUSSION

The results of XRD analyses did not show any minerals of weathered rocks, meaning that the granite samples taken from the Grimsel Test Site, Switzerland, were relatively fresh. Since no expansive minerals are contained in Grimsel granite, saturation of a dried rock sample will not alter the pore structure within the specimen. This further means that the permeability of a completely-dried Grimsel granite induced by air permeation test can provide equivalent values for water permeability of a completely saturated Grimsel granite [21].

Chemical compositions of granites are typically 70-77% silica, 11-13 alumina, 3-5% potassium oxide, 3-5 soda, 1% lime, 2-3 total iron, and less than 1% magnesia and titania. Chemical compositions of Grimsel granite obtained from the quantitative XRF analyses are consistent with those of typical granites.

The porosity and wave velocities of Grimsel granite are relatively low compared to the average values of igneous rocks indicating that micro-cracks can potentially exist within grain minerals. This inference can be reinforced by visualizing the microstructures within Grimsel granite by means of high resolution X-ray scanning (Fig. 2).

An independent gas adsorption experiment on Inada granite which is a well-known and widely used intact granite in Japan by many researchers was performed. The results of the specific surface, the volume of micropores and the average diameter of micropores within Inada granite were $2.98m^2/g$, $3.69X10^{-4}cm^3/g$ and 51.76nm, respectively. Compared to the Inada granite, Grimsel granite has a slightly larger diameter of micropores, and a slightly higher volume of micropores. These findings are consistent with the above observations that micro-cracks within grain minerals exist in Grimsel granite. From the author's viewpoint, such in-grain micro-cracks may contribute a lot on the capacity of rock matrix for trapping and absorbing radio-nuclides.

The permeability of Grimsel granite is very sensitive to stress condition. Under the tested condition of confining pressures up to about 60MPa, the permeability of Grimsel granite ranged from the order of 1E-15 to 1E-19 m^2, i.e. about 4 orders of magnitude (Table 2 and Fig. 4). The permeability of the test specimen decreased monotonically with increases in confining pressure, while the rate of decrease fell at higher confining pressures. The reduction in permeability was due to the closure of micro-cracks that control fluid flow at low confining pressure [22]. The patterns of reduction in permeability due to increase of confining pressure are also dependent on rock types [21]. The reduction in permeability is more sensitive at low confining pressure conditions for the rocks having relatively more micro-cracks and/or having relatively low strengths.

The permeability of a rock specimen is not a simple function of confining pressure but is hysteretic depending on the stress history. Reduced permeabilities due to increases in stress levels cannot rebound to their initial values even if the stresses are released (Table 2 and Fig. 4).

Although the results of water permeability tests are not available at the present time for the Grimsel granite, the author believes that similar results to those obtained from air permeation tests on the Grimsel granite will be obtained.

Basically, permeation phenomena are similar to diffusive phenomena. Both permeability and effective diffusion coefficient are functions of the effective porosity that can be significantly affected by the stress condition. Currently, most diffusion tests are performed under atmospheric conditions, and the results cannot be directly used for simulating the diffusive properties at depths. Although a few case studies have been reported, the author thinks that conclusions drawn from such case studies do not necessary provide a general conclusion. For example, literature [20] reported the effects of confining pressure on diffusive properties of Opalinus Clay. By comparing the diffusive properties obtained under 1 and 5 MPa of confining pressures, the author concluded that confining pressure had only a small effect on the diffusion coefficient and almost no effect on the porosity. To some extent, this conclusion is true, but only for talking diffusive properties under confining pressures between 1 and 5 MPa, and for soft rocks like clay. A reduction in permeability over 2 orders of magnitude occurs when confining pressure is increased from about atmospheric condition to 1 MPa (Table 2 and Fig. 4). Even for a granite specimen like Grimsel granite in this study, permeability does not change so much when confining pressure is limited to the range of 1 and 5 MPa. Hence from the author's viewpoint, laboratory diffusion tests should be designed to incorporate the effects of stress conditions.

CONCLUDING REMARKS

Geological formations of igneous rocks, like granites, have been considered as candidate formations for geological disposal of high level radioactive wastes in many countries. Transport of radionuclides through an igneous rock formation is principally controlled by fluid flow along fracture networks, permeation and diffusion in the directions perpendicular to fracture surfaces, sorption onto geological materials and chain decay. Among these mechanisms, permeation and diffusion in the directions perpendicular to fracture surfaces, and sorption onto geological materials control the retardation properties of a geological formation. In our study, systematic laboratory experiments and analyses aimed to characterize the retardation properties of rock specimens taken from Grimsel Test Site, Switzerland, have been planned. This present paper presented the results obtained from the primary stage of the study, which includes chemical and physical properties, and effects of stress and stress history on the permeability of test specimens. Preliminary results obtained from this study indicate the following concluding remarks:

1) The porosity and wave velocities of Grimsel granite are relatively low compared to the average values of igneous rocks indicating that micro-cracks can potentially exist within grain minerals. This inference is further shown by the X-ray scanning visualization.

2) The permeability of Grimsel granite is very sensitive to stress condition. Under the tested condition of confining pressures up to 60MPa, intrinsic permeability of Grimsel granite ranged from the order of 1E-15 to 1E-19 m^2.

3) Permeability is not a simple function of effective confining pressure. It is significantly sensitive to confining pressure at low pressure levels. Moreover, the permeability is hysteretic depending on stress history.

4) Similar to permeability, the diffusion coefficient of rock specimens can also be significantly affected by confining pressure conditions. Laboratory diffusion tests should be designed to incorporate the effects of stress conditions.

To further exam and confirm the conclusions drawn from this study, a series of water permeability tests and diffusion tests which consider the effects of stress and stress history are currently in progress.

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