Development of anti-contamination coring system for microbiological studies

Yusuke Yano*, Tomoya Inoue*, Kazuyasu Wada*, Masanori Kyo* and Noriaki Masui**

Abstract In the coring operation carried out in science drilling or oil / gas exploration, core samples should be recovered without being contaminated to preserve the conditions at drilled point. Therefore, development of a coring system, which reduces a core contamination caused by contact with drilling fluid as much as possible, has been strongly required. In this research, a coring system, which can coat the core surface with gel having physical/chemical characteristics with the progress of coring, was developed. After the fabrication of prototype system, the fundamental functions and the reduction effect of core contamination were confirmed by on-land drilling test using it.

Keywords: Core barrel, Core contamination, IODP, Microbiology, Mud circulation

1. Introduction

After 1980's, the biosphere of underground and under the seabed became one of the important research fields. At present, it is going to progress not as only one field of microbiology but also as interdisciplinary research field which relates closely earth-science, geology, oil field and so on. In recent years, microbiology has been one of the important research fields in the Integrated Ocean Drilling Program (IODP) and the International Continental Scientific Drilling Program (ICDP). However, microbiological or chemical contamination while coring operation is a serious problem to analyze the recovered core for microbiological studies. Therefore, a method which estimates the quantitative contamination level of core caused by contact with drilling fluid and coring system, which can minimize the core contamination, have been required. In this research, a core barrel which can coat the core surface with gel that has anti-contamination effect to prevent the recovered core from invasion of other microbes existing in drilling fluid. In the performance test by using on-land drilling equipment, fundamental functions of the core barrel and state of coating gel on core surface were checked. At the same time, quantitative contamination level of core inside was analyzed by fluorescence beads, which imitated the microbe.

In this report, the basic structure of this coring system from a technical viewpoint, and the result of on-land test are described.

2. Core contamination by drilling fluid

In drilling operation, characteristics of drilling fluid such as specific gravity, viscosity, chemical composition, etc. are adjusted in accordance with the condition of target formation. Cuttings can be removed from the drilled spot by sending of drilling fluid from the drill bit, then a borehole wall is also stabilized. Although, drilling fluid takes on a important role to proceed drilling operation, the contamination of core due to the contact with drilling fluid is a serious problem. Especially, the influence of this contamination must be considered seriously, in the case that the core sample is analyzed for microbiological studies.

In the development stage of standard coring tools for the “OD21” program (i.e., “Ocean Drilling in the 21st century”), the contamination level due to the contact with drilling fluid was analyzed by use of the “Chloride tracer method” and the “Iodine potassium tracer method” in order to evaluate the level of contamination in a core quantitatively.

2.1 Outline of “Chloride tracer method” and the result of analysis

The contamination level test procedure of “Chloride tracer method” is described as follows.

(1) Saturate a gap of rock sample with brine. (Concentration of Chloride ion : 39,000ppm)
(2) Coring operation is performed by using the drilling fluid which does not contain chlorine ion.
(3) Cut out the prescribed parts from recovered core.
(4) The concentration of chloride ion of brine before the drilling operation and that of chlorine ion contained in the interstitial water of recovered core are analyzed by ion chromatograph.

The contamination level is evaluated quantitatively by use of the equation (1).

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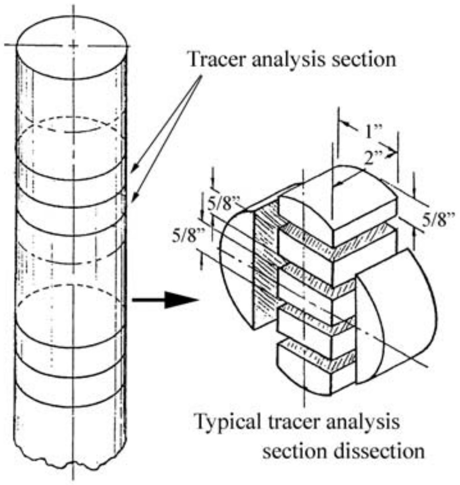

Figure 1: Sampling parts for analysis of core contamination

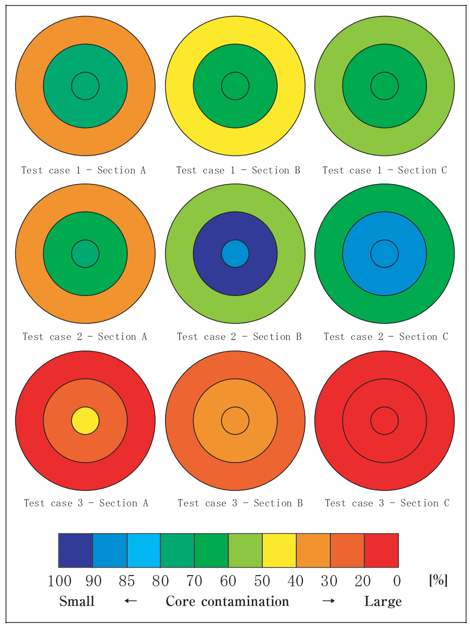

Figure 2: Test result of “Chloride tracer method”

Table 1: Conditions of conditions of the drilling test

Core barrel	SD-RCB (Developed in OD21 project)			
Core Diameter	83mm (3.27”)			
Rock sample	Castlegate sand stone			
	Porosity	27%		
	Density	1.95 g/cm^3		
Drilling condition	Confining pressure	17.5 MPa		
		Case 1	Case 2	Case 3
	Rotary speed	60 rpm	60 rpm	120 rpm
	Flow rate	97 L/min	97 L/min	97 L/min
	Weight on Bit	1050 kg	1050 kg	1050 kg

$$\text{Contamination level} = \frac{C_1}{C_2} \times 100\ \% \qquad (1)$$

where

C_1 : Concentration of chloride ion of interstitial water in recovered core

C_2 : Concentration of chloride ion of the brine

In the first stage of this test, inside of a core is completely saturated with the brine, which sodium chloride is dissolved in, and it is assumed that the chlorine ion concentration, which remains in core sample after this test shows the level of uncontaminated. Sampling parts for analysis of core contamination are shown in Figure 1.

Figure 2 shows an example of the measured result of contamination level of core by “Chloride tracer method”. And Table 1 shows physical properties of rock samples and conditions of the drilling test. Figure 2 shows that the contamination level of the outer part of recovered core is larger than that of inner part.

2.2 Outline of “Iodine potassium tracer method” and the result of analysis

The other contamination level test procedure of “Iodine potassium tracer method” is described as follows.

(1) Dissolve iodine potassium into drilling fluid (Concentration of iodine ion : 300ppm)

(2) Coring operation is performed by use of the drilling fluid, which contains iodine ion.

(3) Cut out the prescribed parts from recovered core.

(4) The concentration of iodine potassium in the interstitial water of recovered core are analyzed by ion chromatograph.

The contamination level is evaluated quantitatively by use of the equation (2).

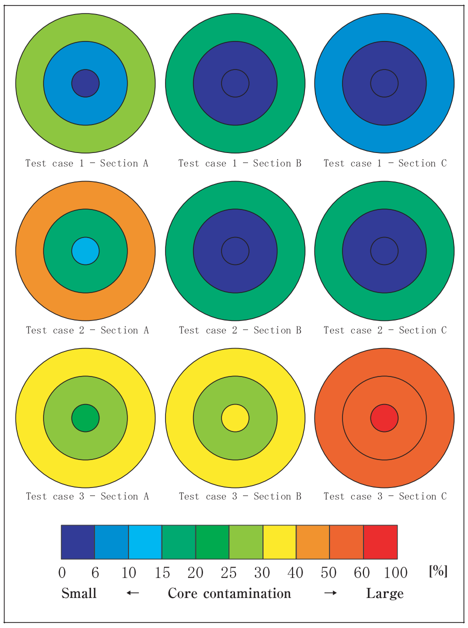

Figure 3: Test result of "Iodine potassium tracer method"

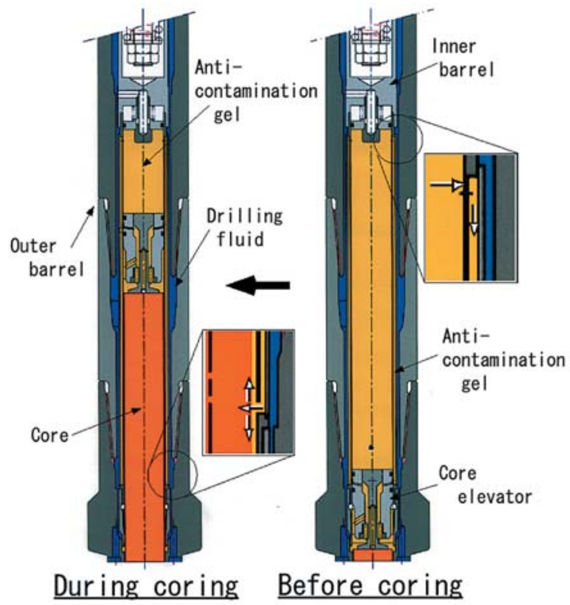

Figure 4: Schematic drawing of the anti-contamination coring system

$$\text{Contamination level} = \frac{C_3}{C_4} \times 100\ \% \qquad (2)$$

where

C_3 : Concentration of iodine potassium of interstitial water in recovered core

C_4 : Concentration of iodine potassium in the drilling fluid

The invading drilling fluid containing iodide potassium shows the contamination level, since the drilling fluid at the beginning does not contain an iodide at all. Therefore, compared with the "Chloride tracer method", this method shows the level of contamination more directly. Sampling parts for analysis of core contamination are shown in Figure 1.

Figure 3 shows an example of the measured result of contamination level of core by "Iodine potassium tracer method". And Table 1 shows physical properties of rock samples and conditions of the drilling test. Figure 3 shows that the contamination level of the outer part of recovered core is large than that of inner part.

3. Anti-contamination coring system

As mentioned before, since the inside of core recovered by using mud circulation method is contaminated by other microbes containing in drilling fluid, recovered core does not correctly represent the environment at the drilled point. Especially, for microbiological research, it is very important to specify the contaminated parts in core and reduce the core contamination as much as possible. In order to overcome with this contamination problem, a core barrel, which has the mechanism to coat the outer surface of recovered core with flow-able coating material composed of gel (Anti-contamination gel) when the core is drilled, was developed. This anti-contamination gel has the function to protect core from contamination with adventitious nonindigeneous microbes. By coating the core surface immediately after coring, the core contamination caused by contact with drilling fluid can be minimized.

Optimization of flow way of gel (the diameter, exit form, quantity, etc.) is indispensable to the reduction of contamination of recovered core and to prevent wrong functioning of core elevator. In the design phase of gel outlets at tip part of inner barrel, an examination using the visualization method was used to analyze the flow velocity / direction of gel on the surface of core. Then the optimum tip shape was determined in accordance with the examination results. Schematic drawing of the anti-contamination coring system developed in this research is shown in Figure 4.

The columnar core portion formed by drilling the surrounding thereof enters the core receiving space through the central opening in the drill bit and the opening for inserting the columnar core portion relatively to downward movement of the outer barrel and inner barrel with the progress of the drilling. At this time, the flow-able coating material ejected inwardly in the radial direction from the ejecting opening of the gel is sprayed on and

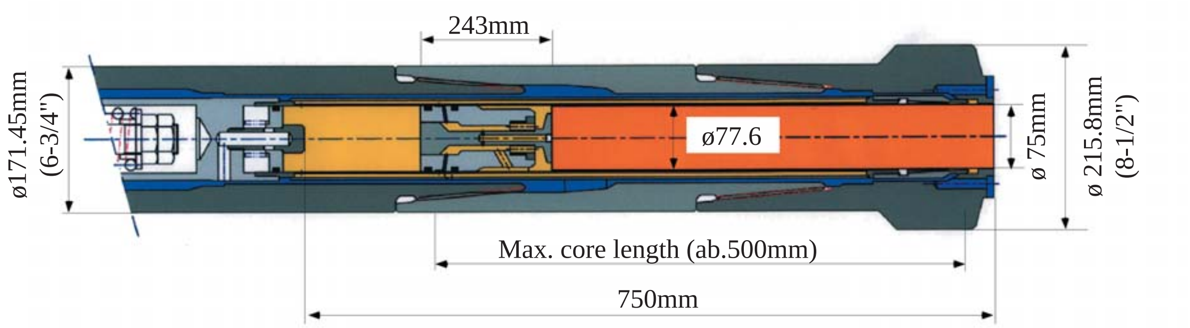

Figure 5: Principal dimensions of prototype coring system

caused to adhere to the outer peripheral surface of the columnar core portion. As a result, the whole outer peripheral surface of the columnar core portion is coated with the gel.

The feeding rate of the gel can be selected in view of various constructional conditions on the core barrel, for example, an area of a section perpendicular to the running direction of the gel in the gel-running channel, an inner diameter of the reservoir, the total opening area of the inlet openings or ejecting openings, and various operational conditions of the core barrel, such as drilling speed.

The ejection speed of the gel ejected from the gel-running channel to core receiving space should be suitably selected according to various factors such as the kind or condition of geology in the core to be drilled, research objects on the core sample taken, and physical properties of the drilling fluid and drilling speed. This ejection speed of the gel can be achieved by suitably setting the total opening area and opening form of the ejecting openings and the number of the ejecting openings, etc. according to the feeding rate of the gel.

Principal dimensions of prototype coring system fabricated in this research are shown in Figure 5.

4. Outline of on-land drilling test

On-land drilling test was carried out 11 cases in total by changing condition such as the kind of gel, various drilling parameters, number of gel ejecting openings, etc.. After the drilling test, coating state of recovered core surface by gel was observed by visual inspection. Change of color of core surface was very little, therefore it was confirmed that coating condition was very well. An example of the recovered core after drilling operation is shown in Figure 6. Since it was impossible to analyze the fluorescence beads quantitatively at the drilling site, the analysis was carried out later at laboratory.

Figure 6: Recovered core immediately after drilling operation (Case 4)

4.1 Outline of drilling equipment

Actual drilling test was carried out at atmospheric pressure by use of the drilling machine shown in Figure 7, and the drilling parameters in the test is shown in Table 2. Although the drilling equipment used in the test was very simple, fundamental method is not different from that used in oil / gas exploration called “Mud circulation method”.

4.2 Physical characteristic of the gels used at drilling test

In the on-land drilling test, several kinds of gel having different chemical characteristic were used, but their hydrodynamic characteristics were almost same. In the design phase of the core barrel, its dimensions and details were determined in accordance with the results of model tests such as the coating effect and the flow characteristic in the gel-running channel of inner barrel. Therefore, the gel used at the drilling test was adjusted its density of 0.94 g/cm^3 and its viscosity of 5,000 mPa·sec so that the coating effect on the core surface might be optimum. In actual drilling, the flow characteristic of the gel changes, because of the up and down of formation temperature and pressure. Therefore, changes of gel viscosity were measured by changing temperature

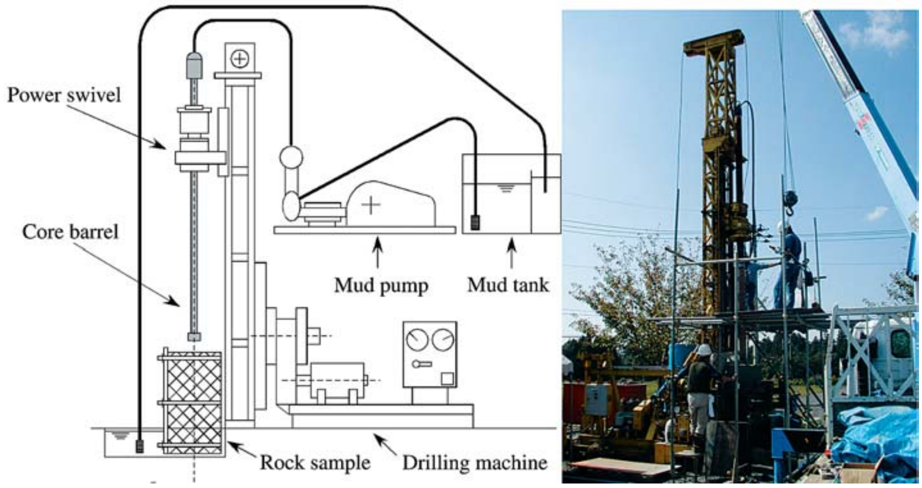

Figure 7: On-land drilling machine

Table 2: Drilling parameters in the drilling test (Ohya-tuff)

	Gel type	Number of bit rotation	Flow rate of mud	Weight on Bit	Recovery rate
Case 1	Without gel	25 ~ 47 rpm	100 L/min	1,200 kg	88%
Case 2	Gel A	25 rpm	100 L/min	1,200 kg	88%
Case 3	Gel A	25 ~ 47 rpm	100 L/min	1,500 kg	100%
Case 4	Gel B	25 rpm	100 L/min	1,200 kg	82%
Case 5	Gel A	25 rpm	100 L/min	1,200 kg	82%

Remarks) Gel A : Crosslinked polyacrylate
Gel B : Crosslinked acrylamide copolymer

(5 ~ 90 degreeC) and pressure (Atmospheric pressure ~ 65MPa). The test results show that there is no great change which will cause problem on the gel running in core barrel within the objective temperature and pressure range.

4.3 Physical properties of a rock sample

If the porosity, permeability and progress of weathering of formation are large, contamination level of recovered core may be also large. Therefore, rock sample such as sand stone with large porosity and permeability is suitable for contamination level test for microbiological studies, because the biological contamination is thought to be larger than that of the other types of rock. In the drilling test, a kind of tuff (Ohya-tuff) from Ibaraki Prefecture, Japan was chosen as main sample rock, and a kind of crystalline schist (Sanba-crystalline schist) has different physical properties was also chosen as comparative rock.

The porosity of sample rock was measured by the helium type pycnometer, and the permeability was measured by the fixed water level method for the Ohya tuff, and the flow-pump method for the Sanba-crystalline schist. The average porosity and density of the Ohya-tuff are estimated to be 40.79% and 1.61 g/cm^3, respectively. The permeability of the Ohya-tuff is roughly $\times 10^{-7}$ and decreases with an increase in effective confining pressure P_c^{eff} (P_c - P_p) from 1 to 9 MPa (Refer to Figure 8), which is considerably larger than that of the Sanba-crystalline schist. The measurement results of average value of porosity and density of the rock samples as mentioned before are shown in Table 3. Measurements of these physical properties were carried out at room temperature environment (15 degreeC).

5. Evaluation method of core contamination and the results

Generally, a method using radioactive isotope such as tritium and a method adding chemical substances have been applied in order to evaluate core contamination quantitatively. However, there have been pointed out the possibility of environmental pollution and restriction on analysis procedure. In addition to these reasons as mentioned, the size of added substance as tracer and its behavior are needed to be similar to those of microbe. Therefore Latex fluorescent microspheres with the

Table 3: Physical properties of sample rocks

	Ohya-tuff	Sanba-crystalline schist
Porosity	40.79%	1.99%
Density	1.61 g/cm^3	2.83 g/cm^3
Permeability	Measured by P_c^{eff} of 1, 5, 9 MPa (Refer to Fig.8)	

diameter of 0.2 μm was added to the drilling fluid as imitation of microbe in this drilling test. Since the size of Latex fluorescent microspheres and the surface electric charge are similar to microbe, the behavior of the microspheres in the drilling fluid is expected to the same as that of microbe existing in drilling fluid.

5.1 Evaluation method of core contamination

Latex fluorescent microspheres were added in the mud tank before the start of drilling operation. The final concentration of the microspheres was roughly adjusted to $\times 10^7$ beads/ml. After the adding of the microspheres to the tank, drilling operation was carried out while agitating in the tank. After the drilling operation, the gel adhering to the core surface was removed, then prescribed parts in the recovered core were grinded as the sub-samples for the contamination analysis. The details of analysis procedure are shown as below. (Refer to Figure 9)

(1) A grinded sub-sample from the prescribed parts is atomized by a mortar. This work procedure is sufficiently investigated so that the samples for every part might not contact with each other at this process.

(2) A grinded sample is dipped to brine with concentration about 30% to separate the microspheres from the sample by difference of their densities. After separation is completed, supernatant liquid is filtered by membrane filters with pore diameter of 0.45, 0.2, and 0.1 μm.

(3) The number of microspheres remaining on each membrane filters are counted by using an optical microscope. Measurement point on whole region of membrane filter was about 100.

(4) The total number of microspheres contained in the confined amount of grinded sub-sample is determined by multiplying the number of microspheres on each measurement point and the observation area.

5.2 Result of contamination analysis of core

The contamination level of inside of core is evaluated by using the analysis method as mentioned before. The density (contamination level of core) of the microspheres shows the tendency, which contamination level

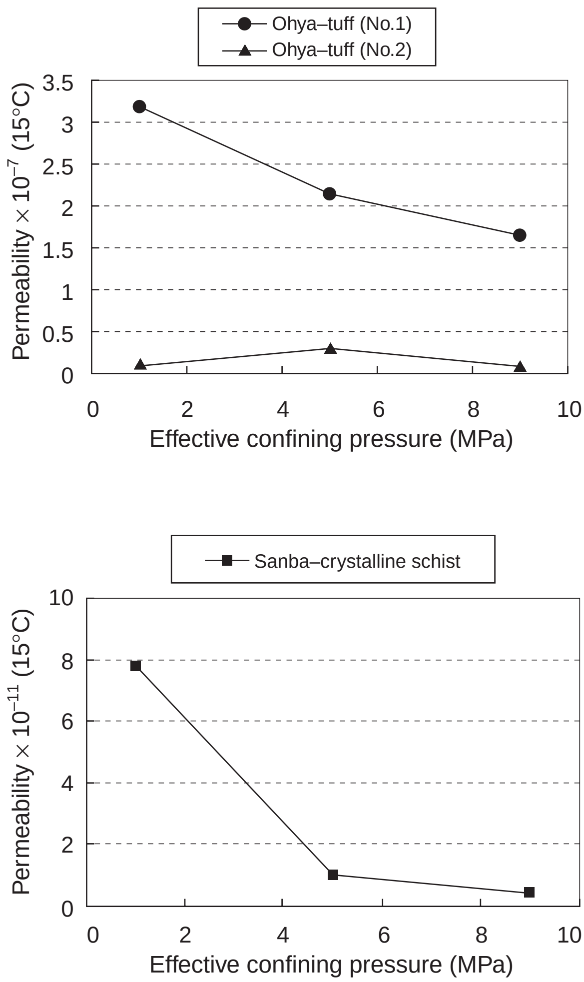

Figure 8: Permeability of the sample rocks by variety of effective confining pressure

of the inside becomes smaller than the outside in the almost all samples. In case of the Sanba-crystalline schist, whose porosity and permeability are considerably small compared with the Ohya-tuff, the number of counted microspheres was extremely few, especially the microspheres were not detected at all in the center part of core. Therefore, surface part (< 1 mm) of the Ohya-tuff considered that its contamination level was the largest, was analyzed particularly, then the relationship between the contamination level of core and the kind of applied gel was verified. Examples of analyzed result are shown in Figure 10. The number of counted microspheres per 1 g of grinded rock sample is shown as a logarithmic indication method in this figure.

The drilling test without gel (Case 1) shows the contamination level of recovered core by using standard drilling with mud circulation method. In this case, the contamination level of core is large compared with the core recovered by anti-contamination method. Even in the drilling cases with anti-contamination gels, it is observed that the each contamination level also changes

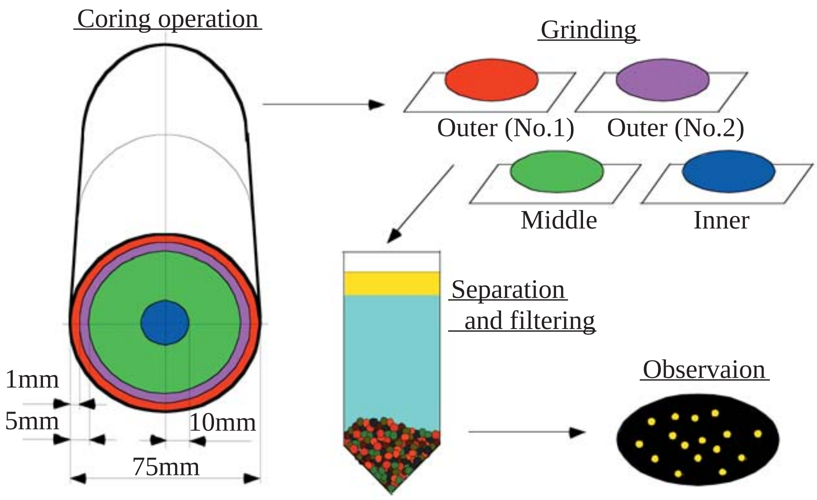

Figure 9: Analysis procedure of the microspheres

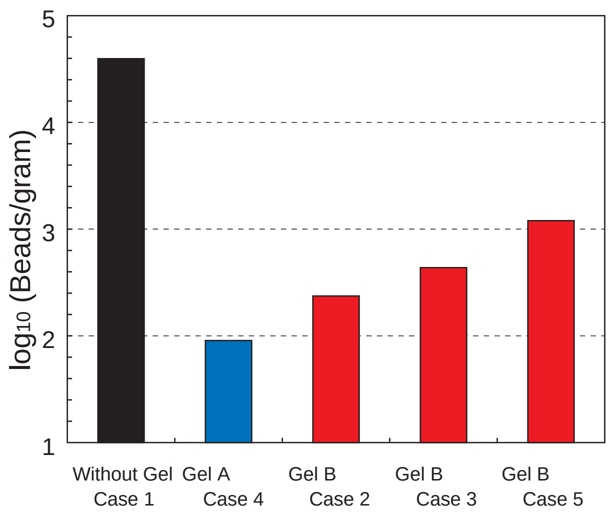

Figure 10: Analyzed result of core contamination (Case 1 : 4.0×10^4, Case 2 : 2.4×10^2, Case 3 : 4.4×10^2, Case 4 : 90, Case 5 : 1.2×10^3)

in accordance with chemical characteristics of the gels. Therefore, adoption of anti-contamination gel with suitable chemical characteristics is extremely important to reduce the core contamination as much as possible, in addition to the satisfactory coating on the core surface.

6. Conclusions

The coring system, which can coat the surface of core while drilling was developed to reduce the biological contamination as much as possible, and the fundamental functions and the coating effect were confirmed by on-land drilling test. By analysis of the recovered cores, it was confirmed that biological contamination could be reduced considerably in combination with the anti-contamination gel has suitable characteristic.

However, the final performance of this anti-contamination coring system such as mechanical functions and the reduction effect of contamination should be confirmed by the actual drilling test in the sea, since the coring operation in the formation has developing fracture, and is also required in the scientific ocean drilling. Although the type of drill bit and the shape of core catcher adopted in this drilling test has not been seriously considered, an appropriate choice and the optimization of flow points of drilling fluid on bit are also needed.

Further improvement is of importance in order to reduce the microbiological contamination and to improve a recovery rate. Even if the chemical characteristics of gel is different from that used in this drilling test, the coating effect of this core barrel does not so differ as long as the gel has suitable hydrodynamic characteristic. Therefore, this coring system can be utilized for the other scientific researches such as geo-chemistry and geology or industrial fields by using the gel has different chemical characteristic. The development of anti-contamination gel has suitable chemical/physical characteristics for various analyses is needed to realize the wide utilization of this coring system, in addition to the mechanical improvement of the coring system.

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