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AMMONIA REMOVAL CHARACTERISTICS OF POROUS CONCRETE WITH ZEOLITE FOR ENHANCING SELF-PURIFICATION ABILITY IN RIVER SYSTEM

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ABSTRACT: Ammonia removal characteristics of porous concrete with zeolite are investigated. Ammonia is one of the common pollutants in river water, and it can cause several adverse impacts for human activities such as water supply and agricultural purposes. It has negative impacts on water ecosystem as well. Porous concrete with zeolite can adsorb ammonia by zeolite, and biological nitrification and its promotion can be expected because attached ammonia on the surface of the porous concrete can promote bacterial growth of nitrifying bacteria. It is expected that both chemical effects (ammonia adsorption) and biological effects (nitrification) can remove ammonia effectively from river waters when the porous concrete is applied in the river as an artificial riverbed for example. Experimental discussion is conducted in this study. It was shown that the specific surface area was increased by making porous concrete, and the ammonia removal rate can be increased twice as a result. It was made clear that chemical effect can be produced in the same level as biological effects as well. It is considered that both effects can increase the ammonia removal ability of porous concrete with zeolite effectively.

Keywords: Porous Concrete, Zeolite, Ammonia, Self-purification of river

1. INTRODUCTION

Concrete constructs are often used in rivers and small channels for river water control and water utilization. Ordinary concrete constructs have been used from the points of durability. On the other hand, environmental functions of rivers attract attention such as self-purification ability of river water, or forming diversified ecosystem surrounding rivers. River Act was revised in 1997 in Japan and environmental functions of rivers have been also focused as well as river water control (flood control) and water utilization (water supply). Therefore, preservation and utilization of the environmental functions of rivers started being paid more attention. There are concrete constructs incorporated in the river, and porous concrete start to be used instead of ordinary concrete because it has wide surface area and a lot of microorganisms can attach on the surface which result in enhancing purification ability of river water quality. Porous property promotes to catch a lot kinds of materials as well as microorganisms, which also helps purifying water quality, therefore purification by porous concrete is expected and porous concrete start to use for the purposes. If the purification is widely applied in the area where sewage system is undeveloped, pollutants loadings in the catchment area can be reduced. Setting porous concrete constructs are easy compared with that of construction of sewerage system. Therefore, several trials have been conducted, and function analysis of porous concrete from the view point of microbial attachment and purifying ability of water quality[1],[2]. It was reported that the microbial densities in porous concrete were 10^4 - 10^3 cells/cm^3 of nitrifying bacteria, 10^7 - 10^8 cells/cm^3 of aerobic heterotrophic bacteria, and 10^7 - 10^8 cells/gVSS (VSS: volatile suspended solids) of denitrifying bacteria, respectively[3].

On the other hand, improvements of purifying functions of porous concrete have been tried. One of the cases is using zeolite partly instead of the cement. Zeolite has cation exchange function, and concrete materials can have cation exchange capacity if zeolite is mixed into the concrete. Zeolite can adsorb NH_4^+-N especially, which is one of the major pollutants from human activity, therefore it is expected that porous concrete in which zeolite is mixed can have not only biological purification ability but also chemical purification ability, especially NH_4^+-N adsorbing ability. The application example for purification of river water quality using porous concrete structure with zeolite have been reported, however most of them is only a report of the application, and detail analyses for purification of water quality have not been conducted from the point of microbial attachment, their activity, and NH_4^+-N adsorption.

In this study, water purifying ability of porous concrete were investigated and evaluated through lab-scale experimental setup. Porous concrete can be a good attachment media for bacteria which can purify river water quality, and the addition of zeolite to concrete enhance adsorption of NH_4^+-N, which can also contribute water purification. It is also expected that adsorption of NH_4^+-N on the surface of the concrete media increase NH_4^+-N concentration which results in the concentrating microorganisms which use NH_4^+-N for their metabolism. Namely the concrete constructs can be a water purifying system if it can have certain amount of microorganisms and other functions such as adsorption of pollutants.

In this study, NH_4^+-N is focused as one of pollutants in river water and its removal efficiency using porous concrete with zeolite was investigated and evaluated. Several kind of porous concrete are targeted and effects of its characteristics such as shape and adding materials on the ability of purification of water body especially river water is investigated.

2. MATERIALS AND METHODS

Lab-scale experiments were conducted. Porous concrete pieces with zeolite were prepared and they were incorporated in to a vessel as simulated concrete constructions. Artificial wastewater was introduced into the vessel and the wastewater was purified by microorganisms attached on the surface of the concrete materials and/or concrete material itself by its adsorption function. The outline of the experimental setup is shown in Figure 1. The shape of each concrete piece is cuboid and its size is 4.9cm×4.9cm×3.2cm in each piece. In the vessel, three different foam types of concrete piece were incorporated. One is the ordinary concrete of which voidage is 0%, and the other two types of concrete pieces were porous concretes of which voidage is 10% and 20%, respectively. 5 concrete pieces of each type were used in a vessel, i.e. 15 concrete pieces were used in an experimental case. 5 experimental cases were set in which mixing ratio of zeolite in the concrete piece was different.

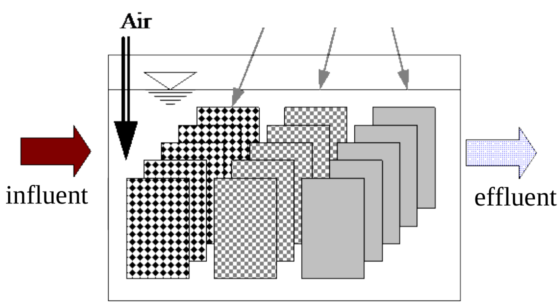

Fig.1 Schematic diagram of experimental apparatus

The mixing ratio of zeolite in the experimental Cases A was in a range of 0-50%, and effects of mixing ratio of zeolite on water purification were investigated.

The experimental condition was summarized in Table 1. These vessels were set in water bath controlled at 25 °C. Apparent volume ratio of concrete pieces to the total volume was 12.5%, and hydraulic retention time (HRT) was set to be 8hrs. Inorganic artificial wastewater was prepared which is simulated a polluted river water and the NH_4^+-N concentration was set to be 10 mg/L. The

Table 1 Experimental condition of concrete pieces used in Cases A

Case No.	Mixing ratio of Ca type zeolite (%)	Voidage 0%	Voidage 10%	Voidage 20%
Case A-1	0%	5	5	5
Case A-2	10%	5	5	5
Case A-3	20%	5	5	5
Case A-4	30%	5	5	5
Case A-5	50%	5	5	5

Table 2 Composition of artificial wastewater

Chemicals	Concentration	
NH_4Cl	10	mgN/L
$NaHCO_3$	107	$mgCaCO_3$/L
KH_2PO_4	0.1	mgP/L
$MgSO_4 \cdot 7H_2O$	1	mgMg/L

Table 3 Experimental condition of concrete pieces used in Cases B

Case No.	Zeolite type	Mixing ratio of zeolite (%)	Voidage 0%	Voidage 20%
Case B-1	Fe type	0%	2 pieces	2 pieces
		10%	2 pieces	2 pieces
		20%	2 pieces	2 pieces
		50%	2 pieces	2 pieces
Case B-2	Ca type	0%	2 pieces	2 pieces
		10%	2 pieces	2 pieces
		20%	2 pieces	2 pieces
		50%	2 pieces	2 pieces
Case B-3	Fe and Ca type	0%	2 pieces	2 pieces
		5+5%	2 pieces	2 pieces
		10+10%	2 pieces	2 pieces
		25+25%	2 pieces	2 pieces

Table 4 Composition of the concrete pieces (kg/m^3)

	without zeolite	with zeolite (20%)
Cement	322.1kg	234.6kg
zeolite	0.0 kg	46.9 kg
coarse aggregate	1535 kg	1535 kg
water	70.9 kg	75.1 kg

composition is shown in Table 2. Aeration was continued in order to maintain aerobic conditions, and aeration was carefully conducted as not to touch the concrete pieces directly for preventing unwanted agitation on the surface of the biofilm. The artificial wastewater was introduced continuously into the reactor, and growth of biofilm which contains nitrifying bacteria was investigated. The inside of the reactor was maintained under complete mixing condition by the aeration. The continuous reaction was conducted under the room temperature and after accomplishing complete nitrification in which NH_4^+-N was completely oxidized to NO_3^--N, nitrifying activities were measured by batch experiment. The concrete pieces with nitrifying biofilm were set in a glass vessel with the artificial wastewater, and nitrifying activity was calculated by measuring the nitrate or nitrite increasing rate in the vessel. After the measurement, the attached microorganisms were detached using both ultrasonic homogenizer and pipetting by which concrete piece would not be damaged, and the detached microorganisms were measured as VSS.

Experimental Cases B were set in order to investigate effects of zeolite type on microbial activity. In this study, three types of zeolite were prepared, i.e. Fe type, Ca type, and their mixed type(Fe + Ca type) were investigated.

Three reactors were prepared. They are operated continuously like Case A. In each reactor 8 kind of concrete pieces were incorporated. The types of the concrete pieces about the voidage are 0 and 20%, and in each porosity types, 4 types of the concrete pieces about the mixing ratio of zeolite were prepared, i.e. the value of the ratio was 0, 10, 20, and 50%. Two pieces in each type concrete pieces, totally 16 of concrete piece, were set in a reactor. Porous concretes with Fe type zeolite was set in a reactor and operated as Case B-1. Porous concretes with Ca type zeolite were used in Case B-2, and Porous concretes with both Fe type zeolite and Ca type zeolite were used in Case B-3. The detail of the experimental condition of concrete pieces used in Cases B is summarized in Table 3. Same as the experimental Cases A, concrete pieces were took from the reactors in order to measure nitrifying activity of each concrete piece after complete nitrification was attained at the reactor(56 days after the start-up). The nitrifying activity was measured by batch experiments and investigated ammonia removal characteristics in each concrete piece.

500mL of glass beakers were prepared, and the 400 mL of the artificial wastewater used in each Case was filled. Then a concrete piece was set in carefully in order to submerge it certainly. The liquid in each batch reactor was stirred by magnetic stirrer. Samplings were conducted 5 times, i.e. after 0.25, 1.25, 2.75, 4.75 and 7 hrs after the beginning the batch test. The amount of a liquid sample is determined as 10mL. In order to collect microorganisms attached on the surface of the concrete piece, the piece was submerged in to a beaker where 300mL of tap water was filled, and then ultrasonic irradiation was conducted twice for 3min. After ultrasonic irradiation, remaining microorganisms was detached from the concrete piece by pipetting.

Mixed liquor with the biofilm is stirred well and sampled for measurement of both SS(suspended solids) and number of nitrifying bacteria. Measurement methods of FISH was followed the Amann's procedure[4]. The collected sample was treated with 4% paraformaldehyde immediately and kept under the condition of 4°C for 2hrs. After that the sample was washed for three times with PBS(Phosphate buffered saline) buffer. The sample was preserved under the condition of -20°C after adding the buffer solution which is the mixed liquid of equivalent amount of both PBS and ethanol. Hybridization was conducted under 46°C for 2hrs. Direct counting methods of fluorescing sample was conducted with incident-light fluorescence microscope (OLYMPUS BX-FLA, OLYMPUS BX-50).

The zeolite mixed in the concrete piece is made from coal ash. The coal ash is treated under alkali condition with NaOH, and then Fe or Ca is added in order to change zeolite. CEC(cation exchange capacity) of the zeolite is 180-200 meq/100g, and its particle size is in a range of 5-100μm, whereas micro pore diameter is 5-100Å. The specific surface area is 100-500(m^2/g). The procedure to make porous concrete pieces is as follows; The coarse aggregate is set in a cement mixer, and water is added by bits. After mixing, powder materials(cement and zeolite) are added, and water start to be added in order to make the surface of the coarse aggregate covered by the paste which is the mixture of the powder materials and water. Then the mixed material is set into a steel form unit with using form vibrator. After 1 day waiting, hardening occurs and it is demolded. The composition of the concrete pieces (kg/m^3) is summarized in Table 4 and schematic diagram of porous concrete piece is shown in Picture 1.

For start-up of these experiments, cultivated sludge with culture media shown in Table 2 was added into each reactor in order to attach and grow nitrifying bacteria on the surface of the concrete piece quickly. The sludge itself was originally obtained from domestic wastewater treatment plant (WWTP).

3. RESULTS AND DISCUSSION

The results of experimental Case A-5 are shown in Figure 2 for example. Ammonium nitrogen was easily transformed to nitrite nitrogen, oxidation of nitrite nitrogen started and ammonia is quickly oxidized to nitrate nitrogen. And about 40 days after the start up, similar tendency were observed in the other cases. Apparent specific growth rates are calculated from the results of continuous treatment according to the following equation.

Apparent specific growth rate = [net specific growth rate: μ (1/day)] - [detachment/death rate: d (1/day)])

The results of apparent specific growth rates of AOB were 0.330 (1/day) in Case A-1, 0.232(1/day) in Case A-2, 0.297(1/day) in Case A-3, 0.332(1/day) in Case A-4, and 0.363(1/day) in Case A-5, respectively. On the other hand, these of NOB are 0.338(1/day) in Case A-1, 0.117(1/day) in Case A-2, 0.241(1/day) in Case A-3, 0.157(1/day) in Case A-4, 0.186(1/day) in Case A-5, respectively.

Apparent specific growth rates of nitrifying bacteria in the cases where concrete piece without zeolite were used were larger than these in the cases where the pieces with zeolite are used. The apparent specific growth rate also seems to become larger with the increase of zeolite mixing ratio in the cases the pieces with zeolite are used. However, the tendency was not so clear, and there were not big difference about nitrification occurrence among the Cases A. Generally, NO_2^--N accumulation is not observed if ammonia concentration is about 10 mg/L, however, NO_2^--N accumulation happened in all cases in this study. The reason is considered that free ammoniums which can be exist in high ratio under higher pH conditions. The activity of nitrite oxidation enzyme in NOB can be inhibited by FA[5], and the concentration from which the inhibition start is in a range of 1.0 - 10mgFA-N/L[6][7], in this experiment, initial pH value was in a range of 10 - 11.5, which can inhibit the nitrite oxidation enzyme in NOB. pK at 25°C is 9.25 and the ratio of FA is in a range of 85-99%, therefore partial nitrification was caused by higher pH at the surface of concrete pieces.

Relationship between zeolite containing ratio and biomass attached on the surface of the concrete pieces, or reduction rate of NH_4^+-N are shown in Figure 3. There are tendency that biomass on the surface of the concrete pieces increased with the increase of zeolite containing ratio. It is suggested that ion exchange capacity and adsorption capacity of NH_4^+-N can promote the phenomena because NH_4^+-N is one of the important substrates, and microorganisms themselves were concentrated on the positive charged surface by zeolite because microorganisms are charged negatively charged[8][9]. It was also suggested that pH increase can be suppressed when zeolite was used instead of cement which result in avoiding inhibition of activity of nitrifying bacteria caused by FA relatively.

Amount of microorganisms attaching to the porous concrete was higher than to the ordinary concrete. NH_4^+-N reduction rate per unit concrete piece was in a range of 0.39 - 0.81mgN/hr, and the value was on an increasing trend with an increase of voidage or mixing ratio of zeolite. The difference of the removal rates among them was not so greater than the difference of microbial amount. The thickness of biofilm became larger as attached amount of microorganism increased, and microbial activity inside the biofilm can decrease relatively, therefore, the difference of microbial activity was not so large than that of microbial amount.

Picture 1 Schematic diagram of porous concrete piece

NH_4^+-N removal characteristics by the concrete piece containing zeolite was shown in Figure 4. If the concrete piece cannot adsorb NH_4^+-N, the total concentration of NH_4^+-N and NOx-N should be 10 mgN/L during the batch experiment and the phenomena is drown as the solid line. However, the results of the experiment was different and the total concentration of NH_4^+-N and NOx-N was lower than 10 mgN/L in the initial stage, which shows that adsorption of NH_4^+-N have happened. On the other hand, biological nitrification progressed and the total concentration of nitrogenous compounds was beyond 10mgN/L, which means desorption of NH_4^+-N happens and adsorption ability was regenerated after 6 hrs from the staring the experiment. Finally, total concentration becomes 18 mg/L, and it is calculated that 3.2 mgN of NH_4^+-N was attached on the surface of the concrete pieces. The zeolite used in this study can adsorb NH_4^+-N and its adsorption isotherm is expressed as follows.

$$q = 4.7C^{0.18}$$

where, q: concentration in solid phase (mgN/gZ)
C: concentration in liquid phase (mgN/L)

The adsorbed amount of NH_4^+-N is calculable from the equation because the concrete piece is in the reactor operated continuously and NH_4^+-N is little existed because nitrification occurs before the batch experiment, therefore adsorbed amount of NH_4^+-N was also little, however, it is shown that attached NH_4^+-N on the surface of the concrete pieces can be nitrified and adsorbing capacity can be regenerated. It is suggested concrete pieces with zeolite can remove NH_4^+-N by both biological process and physicochemical process, which means multifunctional removal system can be accomplished.

Next, the results of Cases B are discussed. About for 40 days from the beginning, NO_2^--N was accumulated, which were similar as the Cases A. After that, nitrite oxidation started and NO_2^--N accumulation was resolved by 60 days from the beginning. There were not big difference among Case B-1, Case B-2, and Case B-3 about ammonia and nitrite oxidation, which means the type of zeolite have a small impact on these oxidation.

Relationship between mixing ratio of zeolite in the concrete pieces and ammonium removal rate obtained from the batch experiment were shown in Figure 5. And relationship between mixing ratio of zeolite in the concrete pieces and nitrate generation rate obtained from the batch experiment were shown in Figure 6, as well. Compete nitrification was accomplished and nitrite accumulation was

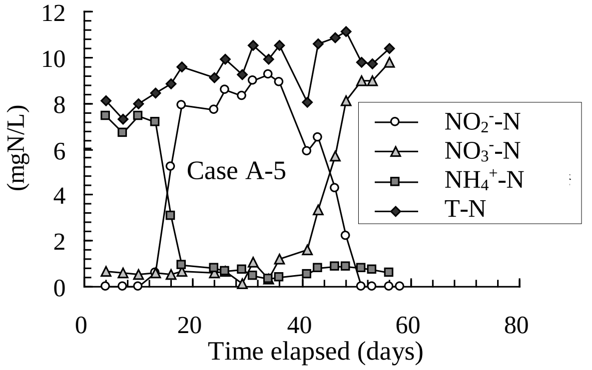

Fig. 2 Time course of nitrogenous compounds (Case A-5)

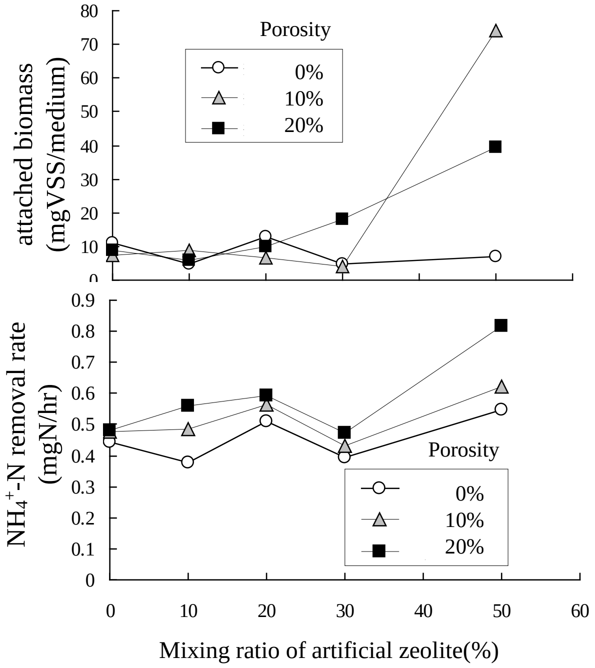

Fig. 3 Relationship between zeolite mixing ratio and attached biomass, or NH_4^+-N removal rate

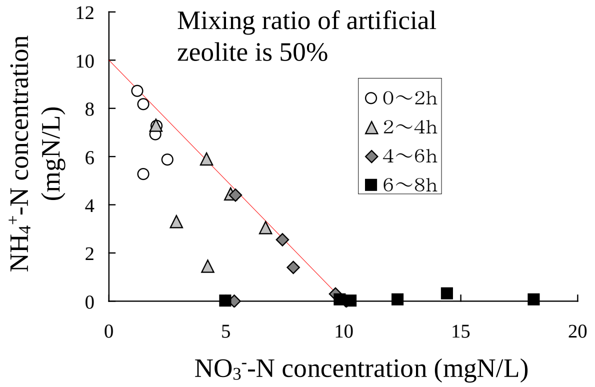

Fig. 4 NH_4^+-N removal characteristics disappeared when batch tests were conducted. Therefore nitrite accumulation was not observed during the batch experiment. NH_4^+-N removal rate of porous concrete was larger than that of ordinal concrete, and there were clear tendency that NH_4^+-N removal rate becomes larger with the increase of zeolite mixing rate clearly.

Because the removal rate increase according to the increase of specific surface area of the concrete pieces, NH_4^+-N removal rate of porous concrete can be increased due to the increase of surface area where NH_4^+-N can be concentrated with zeolite, which can result in enhancement of adsorption of NH_4^+-N to zeolite. It is understood that the ratio of NH_4^+-N adsorption to total NH_4^+-N removal rate can be the same level of that of biological oxidation. The adsorption of NH_4^+-N occurred quickly and in the first stage, therefore it can be expected that buffering function can acts effectively against the sudden increase of NH_4^+-N concentration in water phase. Clear difference about adsorption function was not observed between zeolite types.

On the other hand, nitrate generation rate was 0.4 -1.6mgN/(L・hr), and the rate of porous concrete was higher than that of normal concrete. There was a tendency that the nitrate generation rate increase d with the increase of zeolite ratio, however the correlation is not so clear than the case of ammonia removal rate, and the tendency was observes clearly in the cases of using ordinary concrete compared with the cases of porous concrete. The surface area of porous concrete is wider than the ordinary concrete and its helps forming of much amount of biofilms which can purify the river water quality. Moreover, shape of surface area of porous concrete can increase amount of microorganisms caught at porous, that results in the increase of amount of microorganisms as well. These phenomena result in the increase of self-purifying ability. As results, nitrate generation rate becomes twice compared with the ordinary concrete. There are also tendencies that nitrate generation rate of concrete piece with Fe type zeolite is larger than that with Ca type zeolite. In this study, tap water was used for preparing artificial wastewater, therefore Fe concentration was lower than Ca concentration in the wastewater. Fe is necessary for biological oxidation-reduction reaction because enzymes relating cytochromes uses electron transfer of Fe. It is suggested that nitrification can be promoted by the provision of Fe from zeolite[10]. The nitrifying activity attaching concrete piece with Fe type zeolite was 8.6 mgN/(gMLVSS・hr), which is almost in the same level as previously reported data(4.77 mgN/gTS・hr)[11]. It is shown that using concrete piece with Fe type zeolite can constitute biofilm which accomplish enough nitrification at least.

The density of bacteria attached on the concrete piece is shown in Figure 7. The number of the

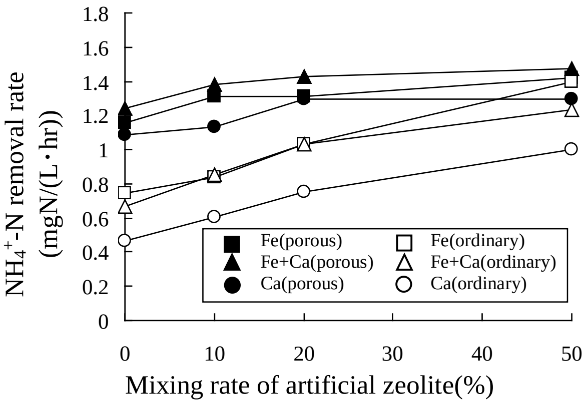

Fig. 5 Relationship between mixing rate of artificial zeolite and NH_4^+-N removal rate

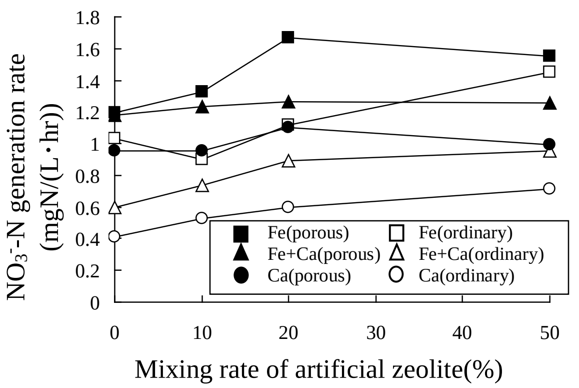

Fig. 6 Relationship between mixing rate of artificial zeolite and NO_3^--N generation rate

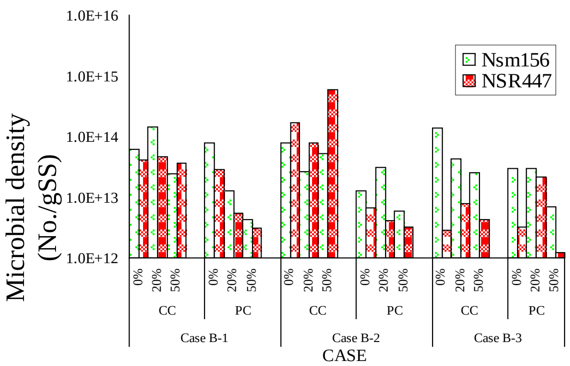

Fig. 7 Density of bacteria attached on the concrete pieces

bacteria on ordinary concrete in Case B-1 and B-2 was counted in a range from 10^{10} to 10^{11} cells by hybridization with probes of Nsm156 and NSR447, respectively, which suggests that the cell number of AOB such as *Nitrosomonas* spp., and NOB such as *Nitrospira* sp. were in the same level. In Case B-3, the number of cells hybridized with probe Nsm156 was around 10^{10}(N), whereas that with probe NSR447 was around 10^{9}(N). The results mean *Nitrosomonas* spp. exists in priority to *Nitrospira* sp. in the media. However, there were not strong relationships between nitrification activity and density or microbial number of nitrifying bacteria in this study. There was a tendency that the number of cells hybridized with probe Nsm156 decreased with the increase of mixing rate of artificial zeolite(%). It is suggested that biological activity per unit amount of the nitrifying bacteria was not same. By the way, the probe NSR447 is suitable for the detection of *Nitorospira* which is closely related to *Nitrospira moscoviensis* live in freshwater environment. On the other hand, we can't obtain clear image in using NIT3 which is a probe for the detection of *Nitrobacter*. It is also suggested that *Nitorospira* was dominant as NOB compared with *Nitrobacter*. in this research. Investigation of population dynamics of microorganisms on the surface of concrete media could be important research topics for next step.

4. CONCLUSIONS

In this study, ammonia removal characteristics of porous concrete with zeolite were investigated by conducting experiments. Some results obtained in this study were summarized as follows.

(1) Concrete pieces containing zeolite can attach microorganisms effectively and the attached amount of microorganisms can increase with the increase of zeolite amount in the concrete pieces. Porous shape can also increase the attaching amount of bacteria. It is suggested that the porous concrete with zeolite can attach microorganisms especially nitrifying bacteria. The concrete piece can adsorb ammonia and the adsorption ability can be regenerated by biologically. The applicability and feasibility to preserve water environment, especially river water quality are suggested.

(2) Nitrification activity of porous concrete with Fe type zeolite was greater than that with Ca type zeolite. Zeolite type would affect the nitrifying ability of concrete pieces.

(3) In this study, clear relationship between nitrifying activity and number of nitrifying bacteria were not observed. Population dynamics on porous concrete will be a challenge for the future.

5. ACKNOWLEDGEMENTS

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