Effective Vertical Solute Transport in Soils by Artificial Macropore System

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Abstract: Solute transport is an important factor governing soil environmental processes, such as effective fertilizer application or dispersion areas of remediation chemicals for contaminated soils. Macropores are ubiquitous in soils. In unsaturated conditions, they enhance air intrusion into soils, thus reducing the chances of clogging and surface ponding. However, their structure is hard to maintain, and they tend to collapse during long-term infiltration. In this experiment, macropore fillings were introduced into the pores to maintain their structure. Solute transport experiments were conducted for four soils with no macropores, empty macropores, macropores with paper towel fillings, and macropores with glass fiber fillings. The macropores with fillings worked as water pathways that conducted solutions to the deeper profile without saturation at the surface, thus avoiding clogging. When bio-remediation experiments were conducted using these four soil columns, soil columns with glass fiber fillings maintained 0.6–0.8 of the saturated water content, which was found to enhance biological activity. The resultant bio-remediation was best for glass fiber fillings because artificial macropores with fibrous fillings maintained a macropore structure, which gave a stable infiltration rate for 30 days. **DOI: 10.1061/(ASCE)HZ.2153-5515.0000192.** © *2014 American Society of Civil Engineers.*

Author keywords: Macropore; Infiltration; Bio-remediation; Oil-contamination; Clogging.

Introduction

In recent years, various regulations (e.g., Soil Contamination Countermeasures Act, Ministry of the Environment, Government of Japan 2002; CERCLA, USEPA, United States 1980) have been promulgated to prevent soil contamination by environmentally toxic chemicals. However, oil contaminants, apart from benzene, have hardly been regulated because they are everywhere and are basically nontoxic. Because of their amount and frequency, contamination by these contaminants needs to be resolved with environmentally sound techniques (Balba et al. 1998). Oil is lighter than water; therefore, remediation techniques in the vadose zone (unsaturated zone) are crucial (USEPA 1995).

In general, extraction operations by pumping or vacuum application are most effectively conducted when the concentrations of contaminants are relatively high (USEPA 1988; Baker and Bierschenk 1995). However, at lower concentrations residue contaminants tend to be present in soil micropores, which make remediation processes difficult. Bio-remediation techniques have been developed recently that conduct air and nutrients to the contaminated soil profile to stimulate the local microbial population (bio-stimulation), or cultivate specific microbes for specific contaminants (bio-augmentation) (Vidali 2001). In spite of these efforts to enhance biological activity, bio-remediation processes are sometimes challenging because soil heterogeneity may cause bypass flow (Beven and Germann 1982), which can seriously affect the efficacy of the chemical application.

Moreover, when biological activities are enhanced, biological colonies can clog the soil pores, forming a less permeable layer (Baveye et al. 1998). Seki and Miyazaki (2001) conducted water infiltration experiments and found that biological colonies were created in the column, which reduced the hydraulic conductivity. Also, infiltration itself sometimes causes dispersion of small soil particles with resultant clogging (Baveye et al. 1998). In these conditions, solutions will not be effectively delivered to the soil profile, leading to potential failure of the bio-remediation process.

In a previous study, the authors reported successful enhancement of biological activity by controlling nutrient dispersion and distributing nutrients to the soil micropores where contaminants usually remain (Mori and Higashi 2009). Bypass flow was successfully prevented in structured soils, where macropores deliver oxygen to the contaminated area. As long as macropore flow is prevented, macropores are important structures for aerobic bio-remediation (Mori et al. 2013). Consequently, the authors recommended using artificial macropores to effectively conduct the bio-remediation. An issue for this technique is that it is difficult for an artificial macropore system to maintain its structure for long periods, and it sometimes collapses during remediation experiments (Mori et al. 2013). If the macropore structure is effectively maintained and infiltration during the bio-remediation process can be continued for long periods, this approach is potentially beneficial for various fields, such as bio-remediation of contaminated soils, leaching of soils with salt accumulation, or effective fertilizer application in agriculture.

In this study, the introduction of fibrous material into artificial macropores is investigated, with the intention of having the fibrous material reinforce the macropore structure, and thus maintain macropore function even if the empty macropores collapse. The duration of infiltration without clogging, uniformity of distribution of the water and solute distribution, unsaturated infiltration without ponding for enhancing biological activity, and bio-remediation efficiency were measured. The objectives of this study were to evaluate the artificial macropores in soils by conducting a solute

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transport experiment and a bio-remediation experiment with and without fibrous materials in the macropores.

Materials and Methods

Soil Column Preparation

Vertical solute transport experiments were performed to investigate the solute transport process enhanced by artificial macropores with and without fibrous fillings. A volcanic ash soil (Kuroboku soil) was used for the experiment. Two macropore fillings were introduced to reinforce the macropore structure and support infiltration. One was glass fiber and the other was paper towel material. Brown et al. (1986) found that glass fiber wicking had a high capillary rise of 54 cm and a saturated hydraulic conductivity of 10^{-2} cm s^{-1}. They also found that glass fiber did not adsorb inorganic ions or organic chemicals. Paper towels have been developed for cleanliness purposes, such as wound dressings, sanitary protection, and incontinence products (Dutkiewicz 2003). They absorb water faster than traditional toweling (Shepherd and Xiao 1999). The two materials also have the advantage that glass fiber is made from a naturally occurring compound (SiO_2), and paper towels from natural cellulose fibers (Shepherd and Xiao 1999). Their physical properties are presented in Table 1. The macropore fillings have higher porosity and higher water conductivity than the soil used in the study.

Solute transport experiments were conducted using a double-layered soil column (Fig. 1). Each column was separated into upper and lower parts (diameter of 5 cm, height of 12 cm) to observe the solute distribution through the column. At the top of the upper column, a 3-cm overhead space was established to observe surface ponding. Twenty-seven-cm-long fibrous materials (diameter of 0.5 cm, one made of glass fiber and the other of paper towels) were inserted vertically through the double-layered column. At the beginning it was not clear whether the fibrous material could conduct water to the lower profile. Therefore, a 3-cm space was set up between the upper and lower column, so that conduction through the fibers could be confirmed. Then the soils were packed separately into the upper and lower columns with a bulk density of 0.95 g cm^{-3}. Thus, the physical length of the total column was 30 cm.

To compare macropore structures, empty artificial macropores were made by inserting seven stainless steel needles (diameter of 0.1 cm) into the columns, one at the center and the other six arranged in a concentric pattern 1 cm away from the center. The resulting hollow macropore structure resembled that of rice roots (Mori et al. 1999a, b, 2001). These macropores were empty without any fillings. This structure had previously shown successful solute transport at a flux rate of 10^{-5} cm s^{-1} (Mori and Higashi 2009). The upper and lower columns were connected to each other with an empty tube (diameter of 0.5 cm). Saturated tube flow or film flow along the tube walls were expected to occur without resistance, thus conducting water and solutes to the lower column.

Table 1. Physical Properties of Soil and Fibrous Materials

Materials	Kuroboku soil (Andisol)	Glass fiber	Paper towel
Density (Mg m^{-3})	2.59	2.54	1.54
Porosity (m^3 m^{-3})	0.635	0.800	0.799
K_s (cm s^{-1})	2.40×10^{-4}	1.57×10^{-3}	3.05×10^{-3}
Bulk density (Mg m^{-3})	0.95	0.51	0.31

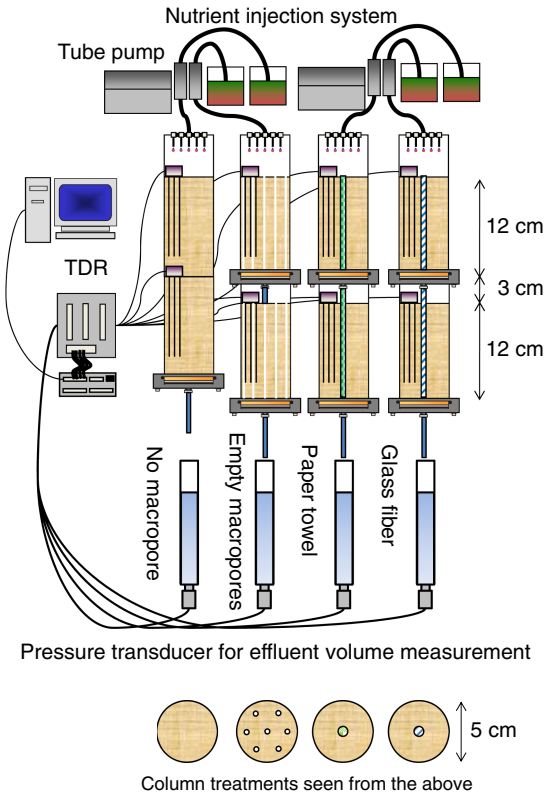

Fig. 1. Experimental setup for the bioremediation experiments

Soil columns with no macropores were also prepared for comparison. Air-dried soil samples (447.5 g) were repacked to a height of 24 cm to achieve a bulk density of 0.95 g cm^{-3}. The upper and lower columns were connected directly for this no-macropore column. The soil surface was scratched to allow smooth connection.

TDR soil water sensors (TDR100, Campbell Scientific, Logan, Utah) were used to monitor the water content and electrical conductivity. Three-needle type TDR probes (length of 7 cm, SK-TDR 1006-18T, Sankeirika, Tokyo, Japan) were installed vertically into the upper and lower parts of the column to evaluate the water and solute distribution between the columns. First, deionized water was introduced to the column for 48 h at a flux rate of 10^{-4} cm s^{-1}, followed by being drained for 24 h under gravitational drainage.

Solute Transport Experiments

Solute transport experiments were conducted for four soil columns: (1) with no macropores, (2) empty macropores, (3) macropores with paper towel fillings, and (4) macropores with glass fiber fillings.

First, to evaluate how the solute transport process was affected by structural differences, solute transport experiments without contaminants were conducted. Nutrients with N:P:K ratios = 5:10:5 (Aid#2, Sumika-Takeda Garden Products, Tokyo), as shown in Table 2, were applied at an injection rate of 10^{-4} cm s^{-1}, which corresponded to the saturated hydraulic conductivity of this soil. This infiltration rate is higher than that successfully used by Mori and Higashi (2009), so there is a greater potential risk of clogging. The experiments were conducted for 48 h. A peristaltic tube pump (AP-2250, PSM050DA, ADVANTEC, Tokyo) was used to maintain a constant flux rate.

Table 2. Constituents of Injected Nutrients

Anions	Concentrations (mg/L)	Cations	Concentrations (mg/L)
NO_3^-	99.43	NH_4^+	134.28
PO_4^{3-}	344.25	K^+	117.56
SO_4^{2-}	0.61	Mg^{2+}	0.81
Cl^-	4.21	Ca^{2+}	0.77
—	—	Na^+	3.50

As described in the previous section on soil column preparation, TDR soil water sensors (TDR100, Campbell Scientific) were installed in the upper and lower part of each column. The water content (θ) and bulk EC (EC_b) were measured and recorded with a data logger (CR10X, Campbell Scientific). This was then transformed into soil solution EC_w by using the equation of Rhoades et al. (1976)

$$EC_b = \theta\tau(\theta)EC_w + EC_s \quad (1a)$$

$$\tau(\theta) = a\theta + b \quad (1b)$$

which defines the contribution of θ, a water content–dependent tortuosity term, $\tau(\theta)$, and the solid surface conductivity (EC_s) to the bulk soil electrical conductivity. Rearranging Eq. (1) yields

$$EC_b = aEC_w\theta^2 + bEC_w\theta + EC_s \quad (2)$$

The calibration consists of fitting Eq. (2) to measured EC_b and θ data for known values of EC_w, yielding values for the a and b coefficients, and the soil surface conductivity, EC_s. All of the experiments were conducted at a controlled temperature of 25°C. Calibration using Eq. (2) provides a way to estimate the soil solution conductivity, EC_w, from measurements of EC_b and θ. After obtaining a, b, and EC_s, the soil solution EC (EC_w) was calculated from

$$EC_w = \frac{EC_b - EC_s}{a\theta^2 + b\theta} \quad (3)$$

The measured EC_b values were transformed into EC_w to eliminate the θ effect and extract the solution EC at each part. A preliminary experiment before bio-remediation was conducted, yielding values of $a = 0.375$, $b = 0.124$, and $EC_s = 4.47$ mS m^{-1}, respectively.

Bio-Remediation Experiment

Soil samples containing 5,000 mg kg^{-1} of a cutting oil contaminant were prepared to perform bio-remediation experiments for the columns with different macropore structures. The cutting oil was mixed with the volcanic ash soils, and repacked to give a bulk density of 0.95 g cm^{-3}. Soil column preparation and water intrusion before the bio-remediation experiments were conducted as described in the previous section. The same nutrients were injected for 30 days at a flow rate of 10^{-4} cm s^{-1}. The nutrients were intended to stimulate the soil microbial biomass and activity, thus enhancing decomposition of the cutting oil. Effluent volumes were measured automatically by four pressure transducers (HTV-001, HI-TECHS, Tokyo, Japan) (Fig. 1).

Biological activity was measured for the soils after the experiments using the fluorescein hydrolysis activity method (Schnurer and Rosswall 1982). Fluorescein diacetate (FDA) hydrolysis is a nonspecific reaction that represents esterase activity, involving proteases, esterases, and lipases. These enzymes are usually found in the biodegradation processes of organic material. Microbial degradation activity was determined by measuring FDA hydrolysis following the methodology described by Schnurer and Rosswall (1982). If bacteria are successfully activated following effective nutrient injection, their numbers will increase and so will the carboxyl esterase. Fluorescein diacetate is hydrolyzed into fluorescein by carboxyl esterase. The resultant fluorescein emits fluorescence, which was measured by spectrophotometry at 490 nm.

After the experiments, soil samples in the soil columns were separated into their upper and lower parts. Then each part was further cut into three parts: upper, middle, and lower. Several grams of soil samples were used for the FDA method.

Finally, the efficiency of bio-remediation was confirmed by extracting the residual cutting oil using the normal-hexane extracting method (after Japan Industrial Standard method JIS K010224 and Central Environmental Council 2006). The remediation efficiency was calculated by dividing the remediated oil mass by the original mass of the contaminant, as shown in Eq. (4)

$$\text{Remediation efficiency} = \frac{\text{Contaminated oil weight} - \text{Residue oil weight}}{\text{Contaminated oil weight}} \quad (4)$$

Results and Discussion

Vertical Water/Solute Transport

Fig. 2(a) shows the vertical infiltration process. The soil water content in the four columns increased in the order of upper and lower column, and water reached each position at almost similar times, regardless of the column treatment. However, after water reached the lower columns, the water content in the upper columns of the macroporous structure did not increase, resulting in a difference in water content between the upper and lower columns. The higher water contents in the lower column and lower water contents in the upper column could result from the macropore function that introduced excess water to the lower column.

Filling the artificial macropores with fibrous fillings successfully worked as water paths that conducted water and solutes to the lower columns. Fig. 2(b) shows EC values in the soil solution derived from Eq. (3). The introduced deionized water leached out the solution from the soil column, and the resultant EC values were slightly higher for the lower column (index L in Fig. 2) and slightly lower for the upper column (index U in Fig. 2), respectively.

After 24 h, there was still nonuniform distribution of solutes in the four columns. At the beginning of the graphs in Fig. 3, the upper columns showed EC values of 50–100 mS m^{-1}, whereas the lower columns had values of 150–250 mS m^{-1}. The authors began nutrient injection with this uneven distribution. Fig. 3 shows changes in EC values in the soil solution when nutrients (141.0 mS m^{-1}) were introduced into the columns. It took 4, 5, 15–20, and 20–25 h for the glass fiber–filled column, paper towel–filled column, empty macropores column, and no-macropore column, respectively, to reach an even distribution of approximately 150 mS m^{-1}.

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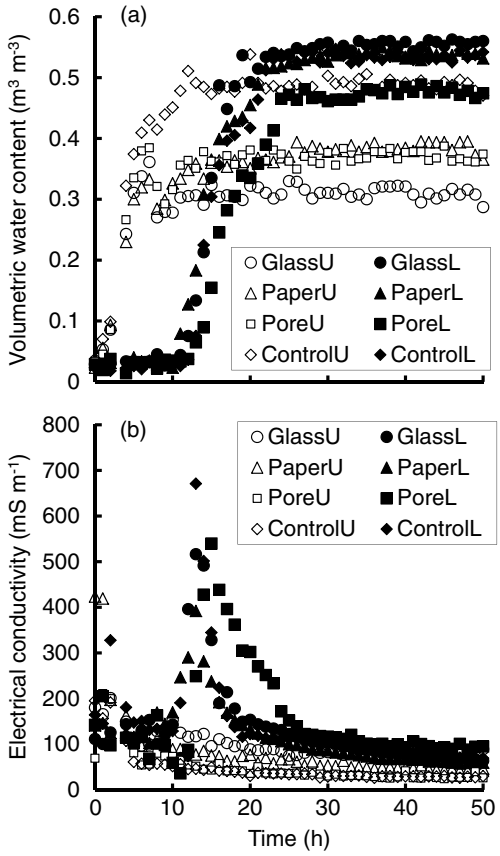

Fig. 2. (a) Water content and (b) EC distribution during infiltration of deionized water (U and L denote the upper and lower column, respectively)

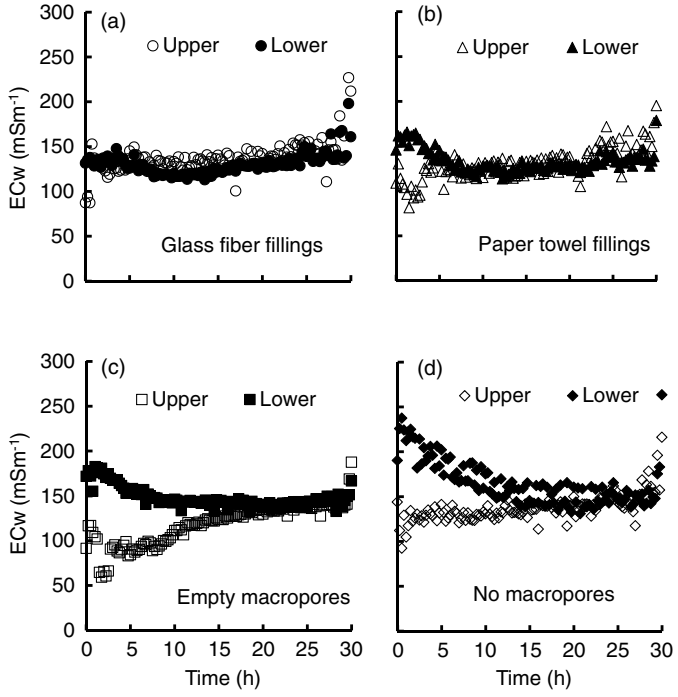

Fig. 3. Solution infiltration for nonuniformly distributed solutes in soil columns

According to the deionized water infiltration experiment, the fact that the water content in the lower column was higher than for the columns without macropores suggests that the macropore structure enhanced water intrusion into the lower column. As shown in Fig. 3, for solute transport, the fibrous fillings enhanced solute transport, and therefore obtained an even solute distribution within several hours, whereas it took more than 10 h for the empty macropores column and no-macropore column to reach this state. Empty macropores would work as water paths for solute transport for a short time, but might collapse during the infiltration process.

Fig. 4 shows the volumetric water content after the experiment in the upper, middle, and lower sections of each of the upper and lower columns. In general, maximum biological activity would be observed at a water content of 0.6–0.8 of saturation (Nishio 1989). The saturated water content was 0.635 $m^3\ m^{-3}$ in this experiment with volcanic ash soils, so the optimum water content for biological activity should be 0.38–0.51 $m^3\ m^{-3}$ (0.635×0.6 and 0.8). Two dashed lines are drawn at 0.38 and 0.51 $m^3\ m^{-3}$ in the graph, respectively. Fig. 4 shows that three soil columns (with the exception of the empty macropores column) had appropriate water contents within this range.

Bio-Remediation Processes for Oil Contaminated Soils

Infiltration Process

Fig. 5 shows effluent volumes for the four soil columns during the bio-remediation experiment. The infiltration process itself causes dispersion of small soil particles, which may result in clogging. In addition, because bio-remediation enhances biological activity, the enhanced biological activity increases the risk of bio-clogging, which might cause infiltration difficulties.

Surface ponding was observed for the no-macropore and empty macropore soil columns at day 5; therefore, the infiltration rate was lowered until day 10 in anticipation of eliminating surface ponding. However, because surface ponding did not completely disappear for these two columns, watering continued intermittently only when soil surface was free from ponding. Watering at the original rate continued for the columns containing macropores with glass

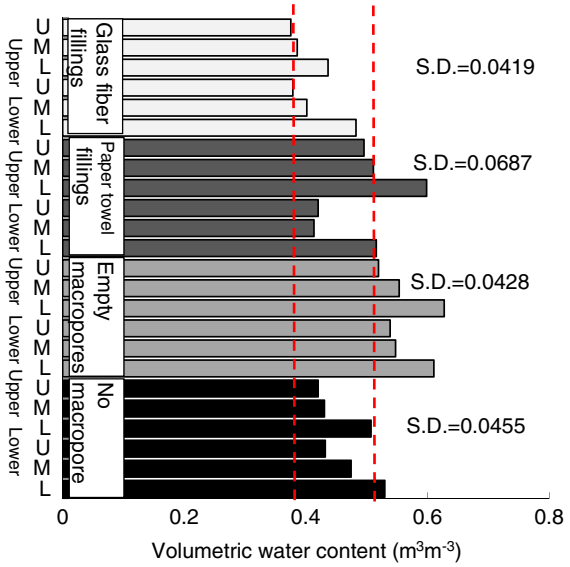

Fig. 4. Water content distribution in the soil columns after solute transport experiment (dashed line denotes 60 and 80% of saturation; U, M, and L denote upper, middle, and lower part of each column, respectively)

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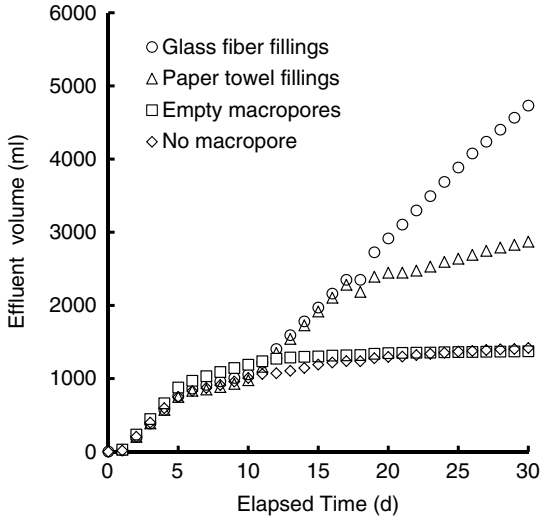

Fig. 5. Observed effluent volume during the bio-remediation experiment

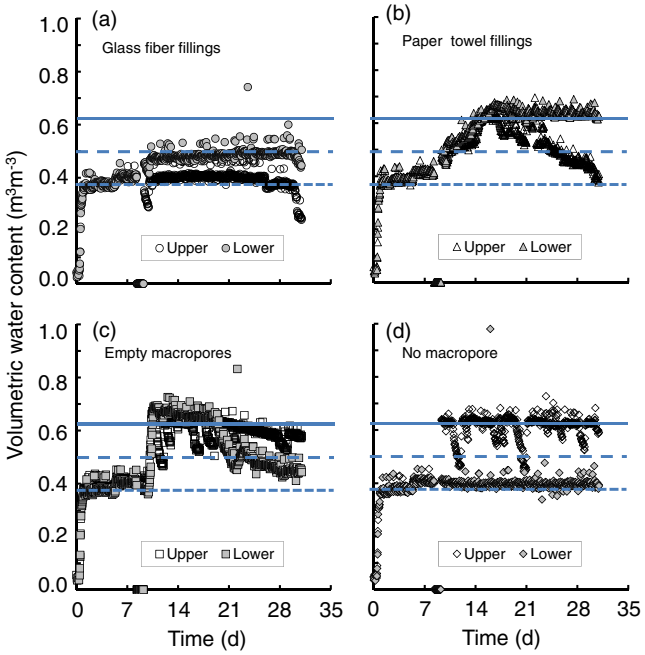

Fig. 6. Water content distribution during bio-remediation experiment [(a) glass fiber fillings; (b) paper towel fillings; (c) empty macropores; (d) no macropore; dashed line denotes 60 and 80% of saturation; solid line denotes saturation]

fiber fillings and paper towel fillings. At day 17, the column with paper towel fillings showed surface ponding. The paper towel material tends to swell as it absorbs water (Akinli-Kogak 2001; Masoodi and Pillai 2010), which causes a narrowing of the water path. Moreover, biological colony could be formed on the paper towel itself because it is an organic material (Gendron et al. 2012). Therefore, only the column with glass fiber fillings achieved the original watering rate.

Water contents during the bio-remediation experiments are shown in Fig. 6. The water content was approximately 0.40 $m^3 m^{-3}$ when surface ponding was observed at days 7–10. This means that the soil columns themselves were not saturated, but partial clogging apparently caused surface ponding for the no-macropore and empty macropore columns. Subsequently, when the flux rate returned to its original rate at day 10, the water content increased to saturation. The water content was characteristically higher in the upper columns and lower in the lower columns for the no-macropore and empty macropore columns. Water contents seemed to exceed saturation for some columns, probably because of the swelling of the soil.

The water content for the column with paper towel fillings gradually increased to reach saturation at day 17 [Fig. 6(b)], which corresponded well with observations and effluent volumes in Fig. 5. After that, the volumetric water content for the column with paper towel fillings was saturated, especially in the lower column. The column with glass fiber filling was the only one that achieved continuous infiltration throughout the experimental period. This was observed from both the water content sensor and effluent volume measurement. It was obvious for the columns with paper towel filling and glass fiber filling that the water content was lower in the upper column and higher in the lower column. This is explained by the characteristics of the artificial macropores with fiber materials, as the fibrous materials enhanced water infiltration to the lower profile when the soil water content was near saturation.

In a previous study (Mori and Higashi 2009), the authors applied a suction of 3 kPa at the bottom of the soil column to maintain unsaturated conditions. In this study, fiber materials worked actively as conductive materials for flow by inducing excess water to flow downward, hence keeping the soil column in an unsaturated state. This technique would be beneficial for field application where suction control is not available. Moreover, the optimum water content (0.6–0.8 of saturation) for biological activity was maintained for the column with glass fiber filling.

Solute Transport Process

Trends in electrical conductivity are shown in Figs. 7(a–d). In general, the graphs can be divided into two groups. One group includes the no-macropore [Fig. 7(d)] and empty macropore [Fig. 7(c)] columns, which showed maximum conductivities of 100–150 mS m^{-1} with increasing time. The other group includes the columns in which the macropores contain paper towel fillings [Fig. 7(b)] and glass fiber fillings [Fig. 7(a)]. These columns showed a trend of increasing EC values with time toward 300 mS m^{-1}. Then, ion concentration in the effluents was measured by ion chromatography, which showed a highly increasing trend in nitrate concentrations for the columns with paper towel fillings and glass fiber fillings (Fig. 8). In this condition, nitrifying bacteria apparently generated NO_3^- through aerobic metabolism. Taking into consideration that glass fiber fillings and paper towel fillings maintained the original infiltration rate, whereas the other two conditions did not, higher NO_3^- concentrations (Fig. 8) caused higher solution EC values (Fig. 7). The generated NO_3^- showed that aerobic conditions were achieved in this bio-stimulation for the columns with glass fiber fillings and paper towel fillings.

Biological Activity in the Soils

Biological activity was measured by the FDA hydrolysis method for the upper, middle, and lower sections of each of the upper and lower columns (Fig. 9). As expected from the previous experimental results, biological activity increased in the order of no-macropores, empty macropores, glass fiber filling, and paper towel filling. The standard deviation (s.d.) was calculated for the six different positions as an index of the uniformity of biological activity, for which a smaller s.d. would show uniformity of remediation through the column. Standard deviation values became larger in the order of paper towel, glass fiber fillings, no-macropores, and empty macropores. In addition, especially for the empty macropores and

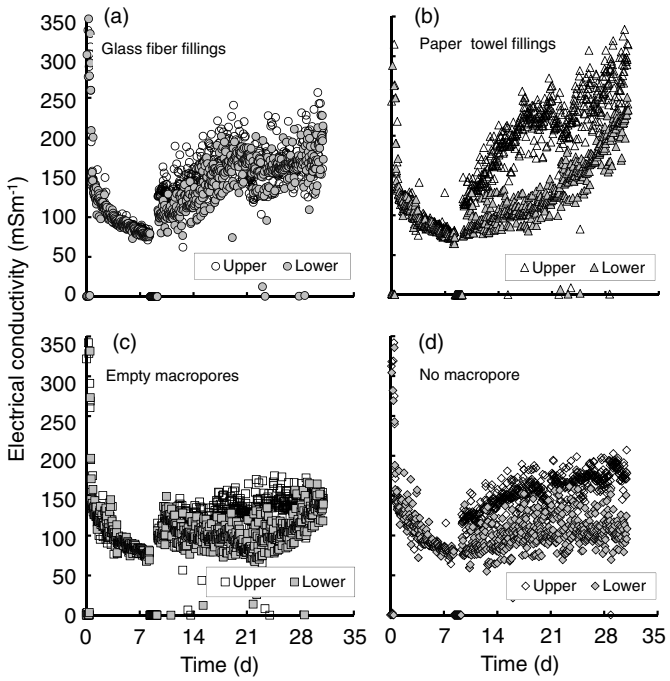

Fig. 7. Electrical conductivity distribution during bio-remediation experiment: (a) glass fiber fillings; (b) paper towel fillings; (c) empty macropores; (d) no macropore

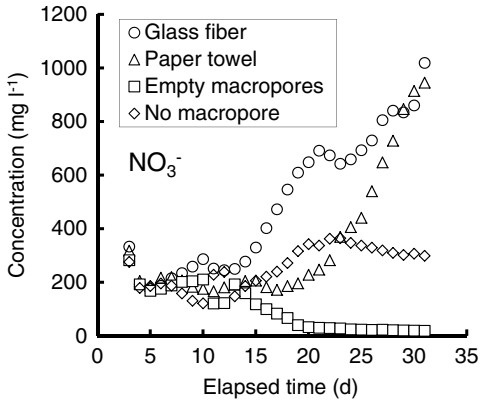

Fig. 8. Nitrate concentrations in effluent during bio-remediation experiment

no-macropore columns, there were trends in biological activity, with low biological activity in the upper part of the columns. It is likely that the upper part of the column was easily clogged by small clay particles or local biological colonies. In these conditions, the presence of fiber material fillings in the macropores could improve the water pathway when the soil columns are approaching saturated conditions. Fibrous materials have a capillary force (Beuther et al. 2010) that starts working in unsaturated conditions, whereas empty macropores work only in saturated conditions, namely the ponding/surface water flow condition. Thus, artificial macropores with fiber fillings have an advantage over empty macropores because of their reinforced structure and wider range of infiltration characteristics.

Remediation Efficiency

Finally, the remediation efficiency is evaluated in Fig. 10. This efficiency could not truly be compared because the nutrient infiltration volumes were different for the four columns. However, taking into consideration that bio-remediation in situ always

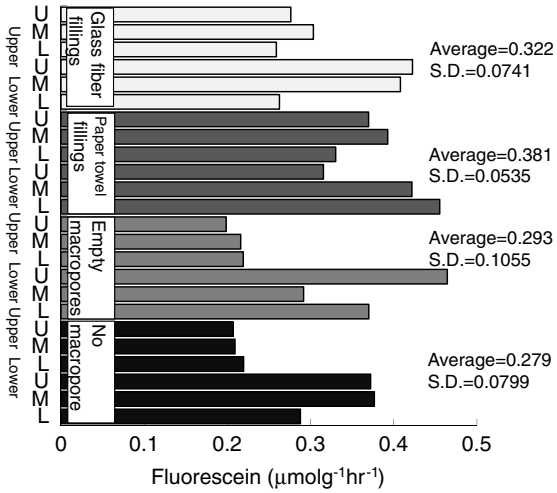

Fig. 9. Biological activity in soil after bioremediation experiment (U, M, and L denote upper, middle, and lower part of each column, respectively)

involves infiltration problems such as clogging, evaluation is worthwhile, even allowing for these differences in infiltration volume. The bio-remediation efficiency was greater in the order of glass fiber fillings, paper towel, empty macropores, and no-macropore columns. The standard deviation (s.d.) was calculated for the six different sampled positions. The resultant s.d. values show that uniformity was larger for the soil columns with macropores. The difference in uniformity and remediation efficiency would be expected to become larger when bio-remediation was continued for more than 1 month.

In the course of the experiments, the authors have understood the function of macropores with fibrous materials to be as follows. First, when nutrients are injected into the contaminated soils, microbes would be gradually stimulated by the nutrients and air, and thus biological activity would increase. As biological activity increases, remediation would proceed while bio-clogging gradually occurs. During that process the water content would increase.

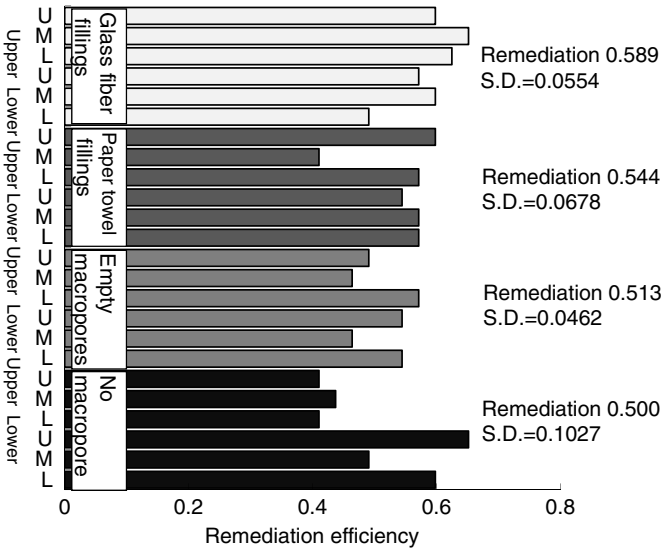

Fig. 10. Remediation efficiency after bioremediation experiment (U, M, and L denote upper, middle, and lower part of each column, respectively)

If macropores are not present, water would pond easily, leading to failure of the bio-remediation. If there are empty macropores, water ponding would be avoided for a while, but the macropores would gradually collapse as water infiltrates into the soil. If there are macropores containing fibrous materials, the fibrous material would induce excess water to flow from the surface into the soil body, maintaining unsaturated conditions in the soil column. Bio-remediation would therefore continue with nutrient infiltration along with fresh air.

Conclusions

Artificial macropores with fibrous fillings were used to effectively transport nutrients to the deeper soil profile. Solute transport and bio-remediation experiments were conducted for the soil columns with and without macropores and macropore fillings to examine effective solute transport into the soil profile. The following results were obtained.

1. Macropore fillings with fiber materials reduced the chance of saturation of the soil during the experiment, allowing excess water to be conducted deeper into the soil profile. Nutrients were delivered quickly and evenly through the profile in a short time;
2. The bio-remediation experiment was successfully conducted for 30 days using artificial macropores with glass fiber fillings. Paper towel fillings showed fair infiltration for 2 weeks. However, they gradually clogged, probably because of their swelling properties or the formation of a bio-colony on the paper itself;
3. Macropores with fibrous fillings showed a higher EC trend than empty macropores and columns with no macropores. An increase in nitrate concentrations showed that aerobic decomposition had been achieved; and
4. The bio-remediation efficiency, along with biological activity, was high for the fibrous fillings. The small standard deviation showed that biological activity and remediation were occurring uniformly through the columns. The remediation efficiency was highest for the glass fiber fillings.

When combined with the results from the solute transport and remediation of oil contaminated soils, glass fiber fillings showed the best results with high infiltration ability without causing ponding at the soil surface and enhanced biological activity at a saturated water content of 0.6–0.8.

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