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Performance of composite-ceramic and organic-clay carriers in biofilters treating hydrogen sulfide gas and toluene vapor

Ok Hyun Park† and Jee Eun Han*

Department of Environmental Engineering, Pusan National University, Busan 609-735, Korea
*Institute of Public Health & Environment, 18-4, Sinheung-dong, Jung-gu, Incheon 400-101, Korea
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Abstract–In order to find promising materials for bio-carriers, five kinds of spherical beads of 1 cm diameter were manufactured using natural soil including organic clay and inorganic clay such as diatomite, zeolite, bentonite, and germanium, which are cheap and favorable for pot-planting. Their performance was comparatively investigated according to material properties, microbial attachment and growth, and the biofiltration effect of H_2S and VOC vapor when applied to biofilters. A composite-ceramic carrier made of 1 : 1 mixture of diatomite and bentonite clay showed the best performance among these carriers, according to the maximum removal capacity of these gases. Particularly, bentonite clay appears to be a good material for bio-carriers. The organic-clay carrier can be used as an alternative to peat and compost, being readily dried with a high flow rate particularly in the absence of a water supply.

Key words: Bio-carrier Performance, Biofiltration, Composite-ceramic, Removal Capacity, Specific Surface Area

INTRODUCTION

Since biofiltration is an innovative and reliable new technology for removing hydrogen sulfide gas and toluene vapor, it has been extensively used during the last two decades and has become recognized in Korea as a representative innovative technology for removing odors and gaseous pollutants. The most important priorities in the development of carrier-packed-bed biofilters are (i) development of carriers supplying good inhabitable places for microbes; (ii) identification of bacteria capable of removing corresponding pollutants; and (iii) development of treatment technology employing microbes attached to bio-carriers. Peat and compost have been extensively used as bio-carriers by many investigators because they are easy to acquire, cheap in cost, and superior in gas removal performance [1,2]. However, they cause a reduction of effective bed volume as well as an increase of power requirement because decomposed materials clog bed voids and, as a result, increase the pressure-drop [3]. Since microbes are grown on the packing material, a proper selection of the material is important to maintain a high biofiltration performance being affected by environmental factors such as nutrient, moisture content, pH, and temperature [4]. Hydrogen sulfide gas was selected as a representative odor gas, and toluene vapor was also selected as a representative organic vapor because it is a widely used solvent, but its biodegradability is relatively low [5].

In this study, five kinds of carriers were manufactured using natural materials including organic clay and inorganic clay such as diatomite, zeolite, bentonite, and germanium, which are cheap and favorable for the growth of plants. Evaluation of those bio-carriers was conducted through comparative investigations of microbial attachment and growth, and the biofiltration performance of hydrogen sulfide and toluene vapor by using five sets of lab-scale biofilters in which each bio-carrier of different composition was packed, respectively. Although associated microbes and mechanisms for H_2S removal are different from those for toluene vapor removal, biofiltration performance of each biocarrier for H_2S and toluene vapor was put together in this paper so that readers can have a more comprehensive understanding of useful biocarriers. Particularly, the value of the organic-clay carrier was examined according to its usefulness as a substitute for compost and peat.

†To whom correspondence should be addressed.
E-mail: ohpark@pusan.ac.kr

MATERIALS AND METHODS

1. Manufacture of Bio-carriers

Four kinds of porous ceramic bio-carriers were manufactured by mixing inorganic clays such as diatomite, zeolite, bentonite, and germanium in certain ratios as shown in Table 1, forming them in spherical shapes of 1 cm diameter after adding water and chaff to provide pores and a rough surface, drying at 100-105 °C, and then calcining at 800 °C. Organic-clay carriers of identical size and shape were produced by shaping and then drying at 100-105 °C for one week so that organic matter was not burnt down to examine their relative favorableness for microbial survival under conditions without nutrient-solution supply and to maintain microbial attachability at a high level through preserving basic radicals.

2. Experimental Methods

The experimental apparatus consisted of a gas inflow component and a biofiltering reactor (Fig. 1), and an H_2S-air mixture and toluene-air mixture were separately supplied to the tops of biofilters made of acryl in cylindrical structures of 10 cm diameter and 80 cm height, in which various bio-carriers were packed to a height of up to 60 cm, respectively. The first to fifth biofilters were packed with the first to fifth carriers, respectively (Table 1).

Excess sludge, that is, mixed culture from a waste water treatment plant, was supplied through the upper part of each reactor after acclimation to H_2S gas, and H_2S-removing microbes were attached to the surface of the bio-carriers. A water-jacket was provided by

Table 1. Physical properties and compositions of various carriers

Property \ Carrier label	1st	2nd	3rd	4th	5th
Porosity (%)	34.11	31.0	35.6	26.7	38.8
Bulk density (g/cm^3)	1.57	1.56	1.46	1.65	1.50
Apparent density (g/cm^3)	2.34	2.15	1.77	2.19	2.45
Water absorption (%)	19	19.9	23.5	16.2	25.9
Specific surface area (m^2/g)	12.97	8.662	1.037	7.124	6.113
Compressive strength (kg$_f$/cm^2)	55.84	47.47	58.94	6.113	11.39

Composition; 1st=D (50%)+B (50%), 2nd=D (50%)+Z (50%), 3rd=D (30%)+Z (30%)+B (40%), 4th=D (40%)+Z (30%), G (30%), 5th=O.C. (100%) where, D=diatomite, B=bentonite, Z=zeolite, G=germanium, O.C.=organic clay

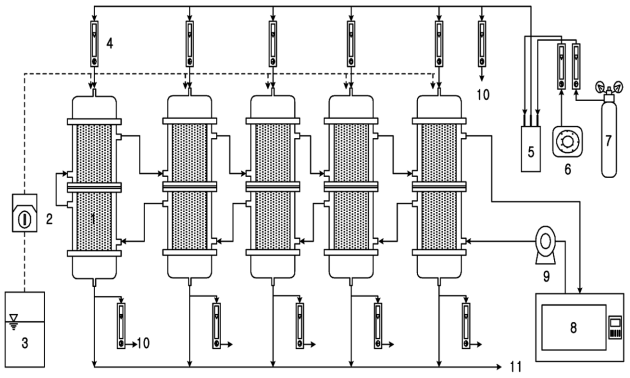

Fig. 1. A schematic diagram of a lab-scale biofilter.

1. Biofilter
2. Peristaltic pump
3. Water reservoir
4. Air-flow meter
5. Mixing chamber
6. Air pump
7. Gas cylinder
8. Water bath
9. Water pump
10. Gas sampling port
11. Gas & water outlet

installing another cylindrical structure of 12 cm diameter outside of the bio-reactors, and the reactor temperature was maintained within a range of 25-30 °C by circulating hot water from a water bath.

Filter-bed moisture was maintained at 40-60%, as per Wani et al.'s report [6], by supplying nutrient solution necessary to the growth of microbes with a peristaltic pump; however, the bio-filter was operated without supplying nutrient solution in cases where a bio-carrier relatively more favorable to microbial survival had to be found. The initial operating conditions of the biofilters are as shown in Table 2.

Experimental apparatus for toluene biofiltration was constructed as in Fig. 1 except for the gas generating component, which consisted of a mass flow controller (MFC), a check-valve for stopping reverse gas flow, and an impinger filled with crude toluene liquid of 99.9% concentration. The concentration of toluene vapor inflowing into the bioreactors was controlled by diluting the concentrated toluene vapor generated at the impinger. Sludge from a secondary settling tank of a waste water treatment plant in a car manufacturing plant, which had been exposed to toluene vapor, was sampled and aerated for 2 days and then settled for about 4 hours to concentrate it. After bio-carriers were dipped into the concentrated sludge, they were packed up to a height of 55 cm in biofilters. Microbes were attached to the surface of the bio-carriers by inflowing the sludge into the top of each biofilter with a peristaltic pump, and grown by injecting toluene vapor of 50-60 µl (toluene)/*l* (air). At the initial stage of microbial growth, an inorganic nutrient solution or water of 250 ml was supplied 1-2 times per day to the biofilters in addition to toluene vapor. The compositions of nutrient solution were K_2HPO_4 (4.3 g/*l*), KH_2PO_4 (3.5 g/*l*), KNO_3 (18.1 g/*l*), $FeSO_4$ (0.001 g/*l*), $MgSO_4 \cdot 7H_2O$ (0.5 g/*l*), and $CaCl_2 \cdot 2H_2O$ (0.02 g/*l*). Biomass was determined in terms of volatile suspended solid (VSS) being the weight difference between before and after combustion at 550 °C of total SS, which is the weight difference of biomass between before and after drying of samples, at 105 °C for 90 min, detached from carriers and then filtered by using a GF/C filter and vacuum pump. Air flow rate in biofilters was sometimes indirectly expressed in terms of empty bed residence time (EBRT), meaning the empty bed filter volume divided by air flow rate. In this study, the biofilters were operated for 115 days under the following conditions: 2-4 *l* (air)/min flowrate, 1-2 min residence time, and a 50-400 µl (toluene)/*l* (air) toluene concentration range.

Concentrations of hydrogen sulfide gas in samples taken from the inlet and the outlet of the biofilters were determined according

Table 2. Initial operating conditions of biofilters for hydrogen sulfide removal

Operating condition \ Biofilter label	1st	2nd	3rd	4th	5th
Initial pH	7.1	7.0	6.8	6.9	5.9
Dry weight of carriers (kg)	4.02	3.88	4.18	4.40	4.84
Packed bed volume (*l*)	4.71	4.71	4.71	4.71	4.71
Packed bed height (cm)	60	60	60	60	60
Flow rate (*l*/min)	4.0	4.0	4.0	4.0	4.0
Sulfur loading (g (S)/m^3 (reactor)/hr)	3.2-131.6	3.4-116.0	3.3-131.6	3.5-131.6	3.9-132.7

to the methylene blue method [7] employing a UV-spectrometer (UV-1600, Shimadzu, Japan), after absorption of the sample gas in sorption tubes filled with a zinc amine complex solution. Concentrations of toluene vapor samples were measured employing gas chromatography (Perkin-Elmer Co., Autosystem XL) equipped with an FID (flame ionization detector).

3. Nonbiological Gas Removal

As a blank experiment to investigate the removal performance of hydrogen sulfide gas by other mechanisms beside biodegradation in biofilters, the concentrations of H_2S gas sampled from the inlet and the outlet of the biofilters were measured 4 times per day at 3-hour intervals under the conditions of a 4 *l* (air)/min flow rate and a 390-520 μl (H_2S)/*l*(air) inlet concentration range, in situations where all operating conditions except microbial attachment to the carrier surface was identical to those of the biodegradation experiments. The mean values of four removal-efficiency data were taken as representatives for each biofilter in which the corresponding carriers were packed, respectively, and then nutrient solution or water of 250 ml was supplied 1-2 times per day. The experimental results showed that the removal efficiencies of hydrogen sulfide gas due to sorption in various packed columns significantly varied from trial to trial, and average values for each reactor ranged from 8 to 27% under the above-mentioned conditions. This range suggests that the physical sorption mechanisms do not work so effectively compared with biofiltration, for which Park and Jeung [8] reported 70 to 100% H_2S removal under the operating conditions of 100-1,500 μl (H_2S)/*l* (air) concentration, a 0.17 Kg (H_2S)/g (MLSS)/sec load, and 7 pH. According to Devinny et al. [9], the gas adsorptivity (defined loosely as absorptivity plus adsorptivity) of bio-carriers is a function of porosity and gas concentration, and allows the stable operation of biofilters through adsorption and desorption.

Since the H_2S removal by absorption in water film of packed bed keeps nearly constant level after about 5 hrs of packed bed operation [10], this abiotic removal of H_2S may be supposed to continuously contribute to whole removals in biofilters. The average abiotic removals for first to fifth carriers were 27%, 19%, 17%, 8%, and 21%, respectively.

To check whether toluene vapor is significantly removed by other mechanisms beside biological degradation, a preliminary test of removal was conducted under identical conditions with biofilters except biological condition. Each reactor packed with each carrier was sterilized by using steam at conditions of 121 °C and 2.5 Kg_f/cm^2 for 25 min, and then cooled at room temperature. Concentrations of toluene vapor sampled from the inlet and outlet of each reactor were measured while nutrient solution was supplied to each reactor with a peristaltic pump at a rate of 1 ml/min before adding sludge. Absorption of toluene vapor in a wet bed reactor packed with various biocarrier turned out to be negligible probably due to the hydrophobic property of toluene vapor [11].

RESULTS AND DISCUSSION

1. Characterization of Biocarriers

The principal chemical constituents of the five carriers investigated with an X-ray luminescence spectrometer were Si, Al, and Fe. The distributions of pore sizes for each carrier were determined according to the mercury infiltration method employing an automatic pore analyzer. The pore size of the first carrier ranged from less than 1 μm to 10 μm, whereas the second and fourth carriers had fine pores of less than 1 μm and relatively coarse ones ranging from 10 to 100 μm, respectively.

Compression strength is a required property for bio-carriers used in packed-bed reactors, and its measurements for the first to fourth carriers were 55.84, 47.47, 58.94, and 64.50 Kg_f/cm^2, respectively, more than double the values for general porous materials (10-25 Kg_f/cm^2). The characterizations of the manufactured ceramic carriers are shown in Table 1. The density and porosity data are relatively large and small, respectively, compared with other commercial ceramics. The specific surface area for the first carrier is significantly large compared with that of the other carriers, and the fifth (organic clay) carrier is superior to the others in water absorption.

2. Performance Comparison of each Carrier in Biofiltration of Hydrogen Sulfide Gas

Though biofiltration is economical compared with other treatment technologies for waste gases of high volume and low concentrations [6], gas removal capacity per unit biomass or unit bed volume should be determined by investigating the variation of treatment performance with gas load in order to appropriately design biofilters.

In this study, the variation of H_2S removal efficiency with inlet concentration was investigated for various bio-carriers under conditions of 45-1,800 μl (H_2S)/*l* (air) concentration, being increased in stepwise fashion, 27 °C reactor temperature, and 4 *l*/min flow rate.

Though all of the biofilters packed with various bio-carriers showed removal efficiencies higher than 99% for H_2S concentrations less than 500 μl (H_2S)/*l* (air), only the first and fifth biofilters revealed 95-97% efficiencies, even at high concentrations up to 1,300 μl (H_2S)/*l* (air).

The relationship between H_2S removal capacity and gas load for biofilters packed with various carriers, determined for the above conditions of flow rate and inflow concentration range, are displayed in Fig. 2. The maximum removal capacities for the first to fifth carriers were 117.0, 62.81, 68.04, 62.67, and 97.19 g (S)/m^3

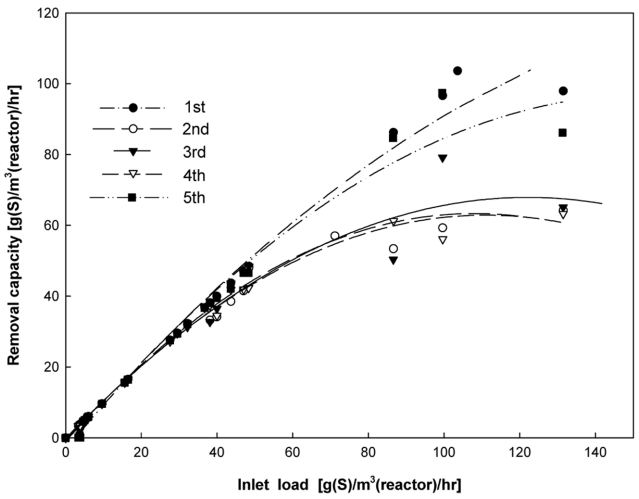

Fig. 2. Relationships between the removal capacity and the load of hydrogen sulfide gas in biofilters packed with various carriers.

(reactor)/hr, respectively. This appears to suggest that the first and fifth carriers are favorable to the attachment and growth of microbes. Considering that the maximum biofiltration rate for compost as an organic carrier is 1.36 g (S)/kg (compost)/day [12], the organic-clay carrier appears to have significantly large biofilterability, while increased pressure drop does not occur with operation time. The significant properties of the first ceramic carrier made of a 1 : 1 mixture of diatomite and bentonite are a relatively large porosity and an excellent specific surface area as well as excellent compression strength, whereas those of the organic-clay carrier are an excellent porosity and a relatively large absorptivity of water, as expressed in Table 1.

Scanning electron photomicrographs of microorganisms growing on the surface of the carriers were taken for the sample carriers sampled from the mid-height position of each biofilter at the terminating point of experiments on the variation of H_2S gas removal with inflow gas load. Each photomicrograph for each biofilter displayed similar microbial species. Short rod-shaped microorganisms similar to purple sulfur bacteria [13,14] formed biofilms. The mass of microorganisms attached to the carriers in each biofilter was calculated based on the measurements for sample carriers from each biofilter after an operating duration of 115 days. The concentration of microorganisms attached to the organic-clay carrier was 4,946.7 mg (VSS)/*l*, which was the largest value among the five kinds of carriers. This seems to indicate the appropriateness of organic-clay as a bio-carrier material partly because organic elements in organic clay are available for microbes. Among the other four ceramic carriers, the first one showed the highest mass value, 4,126.7 mg (VSS)/*l*, probably due to having the largest specific surface area favorable to the attachment of microbes, which leads to the largest removal capacity of H_2S gas. The biomass values for second, third and fourth carriers were 1,506.7, 3,780.0 and 3,266.7 mg (VSS)/*l*, respectively. The reason the removal capacity of H_2S gas for the organic-clay carrier is relatively high next to that for the first ceramic carrier, despite the fact that the microbial concentration for the former was higher than that for the latter, is probably that most sulfur oxidizing microbes take CO_2 as a carbon source and their capacity for sulfur oxidization is inhibited by organic matter [14].

Control of drain water pH has been known as an important operation parameter in biofilters because the final product, SO_4^{-2} of the H_2S oxidation process, acts to retard the biodegradation of H_2S gas [12]. Measurements of drain water pH were conducted during a biodegradation experiment, for which a small amount of water was supplied to biofilters to control the moisture within the filter bed as an essential environmental condition for biofilms. Sulfate formation process was not observed because it was required to compare the resultant performance for each biocarrier in order to distinguish better media.

The drain water pH maintained itself at about 2.5 under a low gas load condition (flow rate: 4 *l*/min; inflow H_2S concentration: 50-200 μl (H_2S)/*l* (air)) at the initial operation stage, but gradually decreased to about 1.3 under a high gas load condition (flow rate: 4 *l*/min; inflow H_2S concentration: 200-600 μl (H_2S)/*l* (air)) as operation time lapsed. However, there was no decrease in H_2S removal efficiency due to such a decrease of pH in this study. This may be attributable to the fact that sulfur-oxidizing bacteria can generally endure an acidic water environment to a pH of 1 [15].

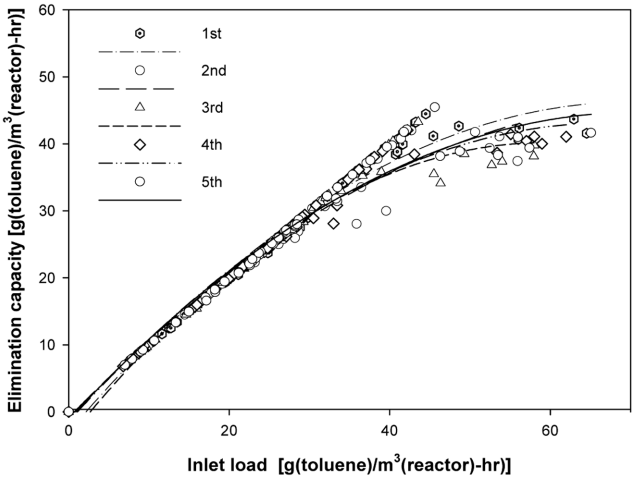

Fig. 3. Relationships between the removal capacities and the loads of toluene vapor in biofilters packed with various carriers.

3. Comparison of Biofiltration Performance of Toluene Vapor between each Biofilter Packed with Various Carriers

Measurements of toluene vapor biofiltration varying with operation time at flow rate of 2 *l*/min show that the performance of toluene vapor removal for the first and fifth biofilter was the best.

The performance improved after 30 days of operation, but the experimental results showed that biofiltration could be inhibited by an abrupt increase of vapor load even after 100 days of highest-level microbial activity. The reason appears to be that the vapor load exceeded the maximum removal capacity per unit biomass.

The maximum toluene vapor removal capacity per unit bed volume or biomass is a design criterion indicating the biofilter performance, and that capacity has been reported to be influenced by biocarrier and operating conditions [12]. The variations of toluene vapor removal capacity with inflow vapor load for each carrier were investigated, as shown in Fig. 3.

All of the biofilters filled with each carrier demonstrated nearly a 100% removal efficiency for vapor load up to about 30 g (toluene)/m^3 (reactor)/hr, and the maximum allowable load for each biocarrier was found to be about 40 g (toluene)/m^3 (reactor)/hr, based on biofilter design. This fact suggests that each biocarrier manufactured by using natural soil including organic clays and inorganic clays, which are favorable for pot-planting, are suitable for biofiltration of organic vapor although the contributions of each chemical component are not distinguished. The significant scatter of gas removal capacity data near the maximum allowable load appears to be attributed to variations in biofilter operating conditions such as temperature, bed moisture, and gas flow rate. The maximum removal capacity of the biofilter packed with organic-clay carrier was found to be about 42.5 g (toluene)/m^3 (reactor)/hr, which is a significantly large value compared with that for the compost carrier, 11.6-20.0 g (toluene)/m^3 (reactor)/hr [1]. Therefore, organic clay carrier appears to have value as a substitute for compost and peat, which have some disadvantages.

In order to see the effect of the flow rate on the toluene removal efficiency in the biofilters, a flow rate of 2 *l* (air)/min (EBRT: 2.16 min) was maintained for 152 days of operation at a constant concentration (300 μl (toluene)/*l* (air)), and experiments on the varia-

tion of the removal efficiency with the flow rate were conducted for the stepwise increase of the flow rate up to 4 *l* (air)/min (EBRT: 1.08 min) for 4 days. The toluene vapor removal efficiencies of the biofilter packed with various carriers increased with EBRT; however, the variation pattern was not linear.

The toluene vapor removal efficiencies of the five biofilters with different carriers increased with the empty bed residence time (EBRT), and these results are identical with those of the previous reports [16, 17]. The toluene vapor removal efficiency appears to be directly proportional to the water absorptivity, which is related to resistance to bed dryness occurring when the flow rate increases. The decreased magnitude of toluene vapor removal efficiency for the organic-clay carrier due to the EBRT shortening was the smallest among those for the five carriers, and this appeared to be also due to the fact that organic-clay carrier has the largest water absorptivity, resisting against bed dryness due to the flow rate increase (Table 1).

It is necessary to specially supply nutrient solution to biofilters in order to maintain the biofiltration performance at a high level, because the nutrient obtainable from pollutant gases in a gas purification process is not sufficient for microbes even though nutrient elements from organic carriers such as compost are more or less available to them. In order to observe the effect of nutrient supply on toluene vapor removal, measurements of the toluene vapor removal capacity of the first and fifth carriers were carried out for cases with and without nutrient solution addition after 40 days of operation with water supply only. The results are plotted in Fig. 4, in which the regression curves for the nutrient solution supply were drawn with reference to Park et al. [11].

The maximum toluene vapor removal capacity for the first biofilter when the nutrient solution was supplied was 42.5 g (toluene)/m^3 (reactor)/hr, corresponding to about 3 times that for without nutrient supply. This result closely approximates that of Sorial et al. [1], who reported experiments on biological removal of toluene vapor using beads made of peat and styrofoam as bio-carriers. Significantly, the toluene vapor removal capacity for the organic-clay carrier when only water was supplied was about double that for the first ceramic carrier when the vapor load was about 40 g (toluene)/m^3 (reactor)/hr. The reasons might be that the microbial density on the surface of the organic-clay carrier with basic radicals favorable to their attachment [18] was relatively high, and that organic elements necessary to microbial growth can be replenished from the organic-clay carriers themselves.

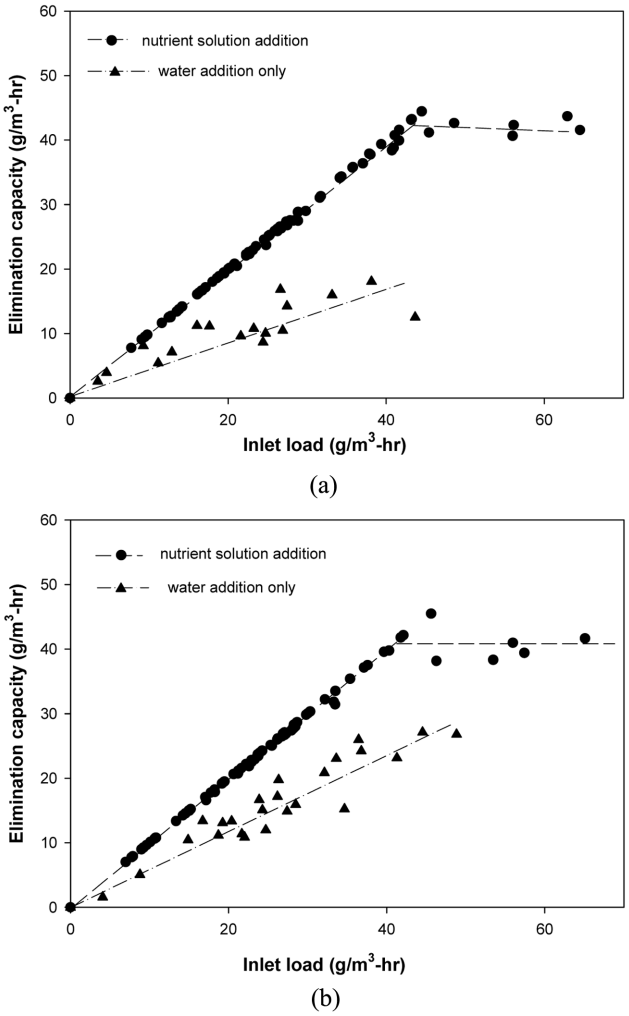

Fig. 4. Comparisons between the toluene vapor removal capacity of biofilters for nutrient solution addition and that for water supplying only: (a) for 1st carrier; (b) for 5th carrier.

4. Overall Evaluation of Carriers' Performance

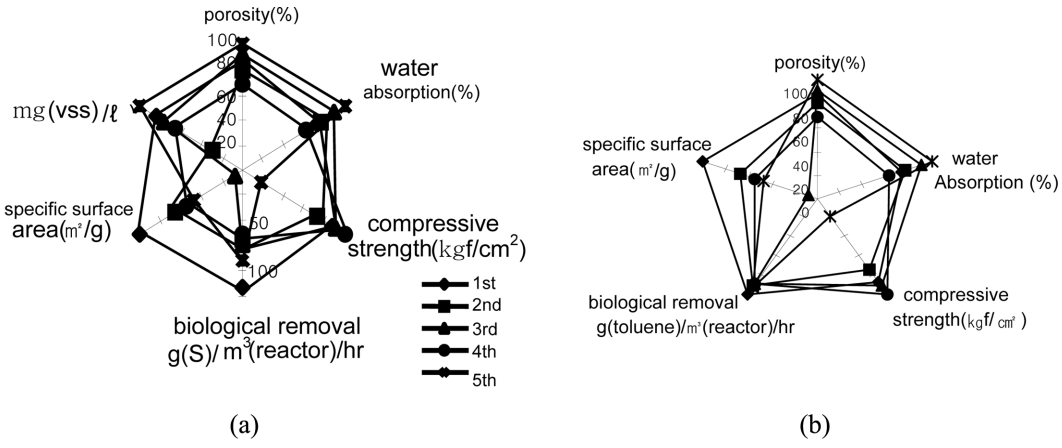

Fig. 5. Overall appraisal for various carriers based on material properties and biofiltration: (a) for hydrogen sulfide (b) for toluene vapor.

The physical/chemical properties and biofiltration performances of each carrier are overall expressed in radar graphs (Fig. 5) in the light of Hirai et al. [16]. Each property axis was graduated according to the relative ratio (%) of each carrier's value to the maximum value among the various carriers, and Fig. 5 shows that the first ceramic, polygons having the largest area, had the best performances. Although the organic-clay carrier has relatively smaller values of compressive strength and specific surface, its biological removal capacity for H_2S gas and toluene vapor was relatively good compared with other composite-ceramic carriers, probably because the organic-clay carrier serves nutrient and water [19].

CONCLUSIONS

Evaluations of the physical properties of organic-clay carriers and ceramic ones composed of natural mineral-clays, which have been commonly used as artificial culture soil for pot-planting, were carried out. Investigations of gas removal performance as well as microbial attachment and growth for those bio-carriers were also conducted. Based on these evaluations and investigations, the following conclusions were drawn:

1. Whole carriers made of natural mineral and organic-clay manifested excellent mechanical strength due to the principal component of SiO_2, and showed gas adsorptivity resulting from their fine-pore composition.

2. As the result of an investigation of the gas removal performances for five biofilters, in which various ceramic and organic-clay carriers of 1 cm diameter were packed, the ceramic carrier composed of a 1 : 1 mixture of diatomite and bentonite was found to have the maximum removal capacity of 117.0 g (S)/m^3 (reactor)/hr and 42.5 g (toluene)/m^3 (reactor)/hr, which corresponded to the best capacity among those for the five carriers. The significant properties of this carrier were an excellent specific surface area and a relatively large porosity.

3. Another ceramic carrier manufactured with a mixture of 30% (by wt.) diatomite, 30% zeolite, and 40% bentonite also showed a relatively better biofiltration performance than the composite ceramic carriers without the bentonite component. This fact appears to manifest the fact that bentonite is a valuable raw material in producing bio-carriers.

4. The biofilter packed with the organic-clay carrier showed high removability of H_2S gas and toluene vapor compared with a ceramic carrier composed of a 1 : 1 mixture of diatomite and bentonite. The organic-clay carrier was found to have maximum toluene removal capacity two times more than compost and peat, and demonstrated relatively better removability for H_2S gas and toluene vapor compared with the inorganic ceramic carrier when nutrient solution was not supplied and when, as a result, the biofilter bed was drying with a high flow rate. Thus, the organic-clay carrier appears to have value as a bio-carrier supplementing the disadvantages of compost and peat readily drying with high flow rate and biologically breaking into pieces that clog bed voids.

5. Various biocarriers made of organic clay or inorganic clay, favorable for pot-planting, appear to be suitable for biofiltration of toluene vapor.

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