

Hydrogen sulfide generation suppression by nitrate addition – application to solid waste landfill site

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Abstract: In Japan, three deaths were reported at an inert solid waste landfill site after exposure to hydrogen sulfide (H₂S) generated at the site. H₂S gas is produced when sulfate-reducing bacteria (SRB) convert sulfate derived from waste gypsum board under anaerobic conditions. The recommended countermeasure is to supply oxygen by installing gas venting pipes. However, a large cost is necessary for pipe installation and the pipes are not applicable to coastal landfill sites. On the other hand, H₂S generation suppression by nitrate (NO₃) addition has been reported in sewage systems. In this study, in order to develop a method to suppress H₂S generation by NO₃ addition at a landfill site, the extent of suppression was quantitatively estimated. When NO₃ reagent was added at the rate of 0.46 mmol-NO₃/(L·d) into a liquid from which H₂S gas was generated at the rate of 0.21 ± 0.05 mmol-H₂S/(L·d), H₂S generation rate was decreased to approximately 1/4 of the original rate. Although it was difficult to maintain the concentration of NO₃ because it tended to disappear rapidly, the frequent addition of NO₃ reagent to maintain the concentration of approximately 430 mg-NO₃/L enabled us to control H₂S concentration to below 1000 ppmv for one month and H₂S generation rate to below 1/20 of the original rate.

Keywords: Landfill Site, Waste Gypsum Board, Hydrogen Sulfide, Generation Suppression, Nitrate

1. Introduction

A strong offensive smell likened to rotten egg odor is recognizable when hydrogen sulfide (H₂S) gas is present in air at 20–30 ppmv (v: volume) [1]. Inhalation of H₂S gas at 750–1000 ppmv causes abrupt physical collapse [2] (hereafter, H₂S exceeding 1000 ppmv is called H₂S_{high}). H₂S gas is generated at landfill sites globally [3–8]. In 1999, three workers at an inert solid waste landfill site (a type of landfill site in Japan where waste plastic, rubber, metal, glass, ceramic, and construction and demolition debris (stones, bricks, and blocks) are acceptable, as well as waste gypsum board without protective paper sheet at the time of the accident. No gas venting pipes, liner for leachate, or water treatment facility was available.) died of exposure to 15000 ppmv H₂S gas. H₂S gas is produced at a landfill when sulfate-reducing bacteria (SRB) consume organic matter and convert sulfate under anaerobic conditions [3, 9, 10]. The source of sulfate in construction and demolition debris is predominantly gypsum (CaSO₄) [11]. No biodegradable

organic matter is acceptable at inert solid waste landfill sites in Japan. Therefore, only waste gypsum board from which protective paper sheet had been removed could be disposed at those landfill sites. Following the report that organic matter in waste gypsum board base material (gypsum part) showed potential for H₂S generation [12], the Ministry of the Environment in Japan issued a notice prohibiting the disposal of waste gypsum board at inert solid waste landfill sites in 2006 (Notice: Treatment of waste gypsum board from which protective paper sheet had been removed). Suggested measures for preventing H₂S generation or diffusion at a landfill site include the exclusion of anaerobic conditions (water exclusion and/or oxygen supply) [13], trapping of generated H₂S gas (conversion into iron sulfide using material containing iron [14, 15], and adsorption by activated carbon [16] or zeolite [17]). The Ministry of the Environment in Japan recommended the appropriate installation of cover soil (to prevent rainwater percolation and trap H₂S gas

generated by iron in soil) and the use of gas venting pipes (oxygen supply). However, in regard to the gas venting pipe, a large cost is necessary for installation and the identification of H_2S generation spots as the installation points. In addition, oxygen supply to the waste layer via gas venting pipes is impossible at coastal landfill sites because a major portion of the waste layer is found below the surface of inner leachate. Trapping by iron does not prevent H_2S generation *per se*. Therefore, it is necessary to develop a method to suppress H_2S generation.

In sewage systems, H_2S generation suppression by nitrate (NO_3) addition has been reported [18, 19]. This was attributed to the increase in oxidation-reduction potential (ORP) caused by the presence of NO_3 . As Gibbs free energies of oxidation of organic matter (CH_2O) with dissolved O_2 , NO_3 , and sulfate (SO_4) as the electron acceptor were -125 , -119 , and -25.4 kJ/mol [20], respectively, the reaction occurred in the order of respiration by aerobic bacteria > denitrification by denitrifying bacteria > SO_4 reduction by SRB (H_2S generation) [20]. Therefore, if dissolved O_2 or NO_3 exists, SO_4 reduction hardly occurs.

If the NO_3 addition method were applicable to the waste layer as well, that method would be a realistic H_2S generation suppression method at an actual landfill site. However, no quantitative investigation has been undertaken, such as the determination of the amount of NO_3 required to suppress H_2S generation. NO_3 reagent is used in sewage systems [19]. Compost made from waste organic matter also contains NO_3 [21, 22]. Therefore, if useless NO_3 in the market could be used at landfill sites, low cost and effective use of resources would be accomplished.

In this study, in order to develop a method for suppressing H_2S generation by NO_3 addition at a landfill site, the following parameters were quantitatively estimated.

- Concentration of NO_3
- NO_3 addition method (intermittent addition and concentration maintenance)
- NO_3 type (reagent and compost)

2. Materials and Methods

2.1. Outline of Experiments

In this study, H_2S gas was generated in an airtight bottle, and the effect of H_2S generation suppression by NO_3 addition was evaluated. Specifically, organic matter and SO_4 were introduced into the bottle, and an anaerobic condition was preserved. Then, the reproducibility of $\text{H}_{2\text{S}_{\text{high}}}$ generation was confirmed. H_2S in liquid and gas phases was removed by bubbling nitrogen (N_2) gas, NO_3 was added into the bottle, and the duration of H_2S generation suppression and the H_2S re-generation rate were measured. Sodium nitrate (NaNO_3) reagent and extract from sewage sludge compost were used as NO_3 sources. Two methods of NO_3 addition were adopted: intermittent addition and concentration maintenance (Fig. 1).

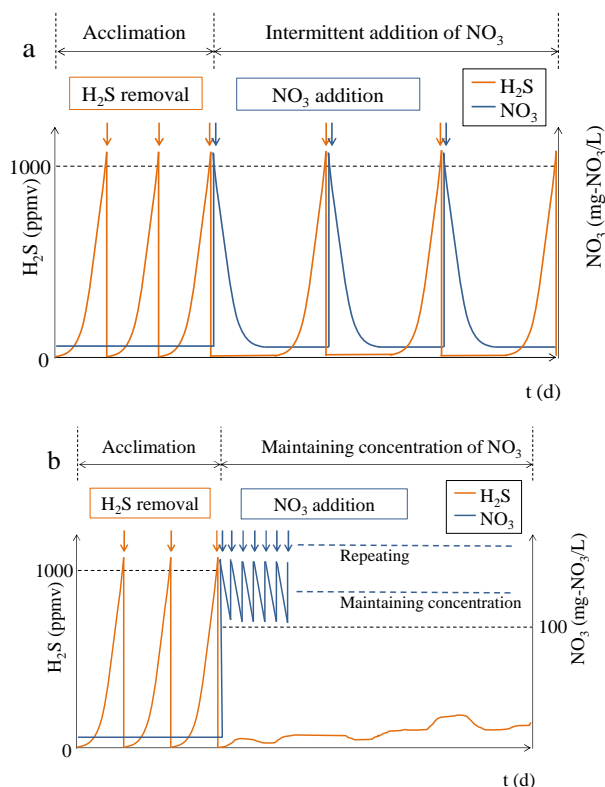


Figure 1. Outline of intermittent addition (a) and concentration maintenance (b) test.

2.2. Materials

Table 1. Details of samples.

Component	Product name, company	Amount used for a H_2S generation bottle
Organic matter	Dried Yeast Extract D-3, Wako Pure Chemical Industries	700~1000 mg-C/L
	Lactose Broth DAIGO, Wako Pure Chemical Industries (equivalently mixed, and added into pure water)	
Calcium Sulfate	Calcium Sulfate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), Japanese Food Additives, Wako Pure Chemical Industries	25 g
Nutrient	BOD Nutrient Buffer Pillows for 6 L sample, APHA Formulation, HACH COMPANY (1 pillow mixed with 500 mL pure water)	25 mL
Silica sand	TOYOURA KEISEKI KOGYO	25 g
Seeding	Black sediment in an ornamental pond (5 g mixed with 500 mL pure water)	25 mL
Nitrate (reagent)	Sodium Nitrate, 1st grade, Wako Pure Chemical Industries	Depending on the condition
Nitrate (compost)	Sewage sludge compost	

As only basic experiments were performed in this study, no actual sample, i.e., leachate from landfill site, was used.

Medium for microbial culture and calcium sulfate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) were used as organic matter and SO_4 sources,

respectively. NaNO_3 reagent and extract from sewage sludge compost were used as NO_3 sources. Hereafter, reagent NO_3 and NO_3 in extract from compost are represented by N_R and N_C , respectively. Sewage sludge compost and water were mixed at the weight ratio of 1:8 and stirred well, and the supernatant was collected as extract. The concentrations of NO_3 and total organic carbon (TOC) in the supernatant were approximately 3500 mg- NO_3 /L and 1900 mg-C/L, respectively. Details of the samples are shown in Table 1.

2.3. Experimental Setup

As shown in Fig. 2, an airtight bottle was made by connecting a gas washing bottle (polycarbonate and polyethylene) to a gas sampling bag (vinylidene fluoride) (called H_2S generation bottle hereafter). The gas sampling bag was filled with N_2 gas. Samples were introduced into the bottle in the amounts shown in Table 1, and pure water was added to make 350 mL. Total gas volume of head in the bottle and the gas sampling bag was approximately 700 mL.

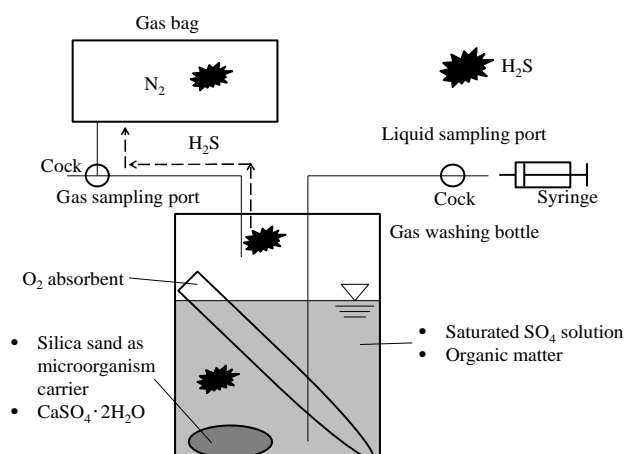


Figure 2. Schematic diagram of H_2S generation bottle.

2.4. Experimental Methods for Acclimation

The bottle conditions for $\text{H}_2\text{S}_{\text{high}}$ generation were as follows: maintenance of (1) appropriate environment for multiplication of SRB (anaerobic condition, temperature for mesophilic bacterial growth), (2) high concentration of organic matter, and (3) presence of SO_4 [12]. To maintain condition (1), oxygen absorbent (tube filled with sodium sulfite solution) was introduced into the H_2S generation bottle, and the bottle was set in an incubator (35 °C). To maintain condition (2), TOC in the liquid phase was measured two times a week, organic matter was injected, and

TOC after the injection was re-measured. Thus, TOC was maintained in an amount ranging from 700 to 1000 mg-C/L. To maintain condition (3), SO_4 was introduced into the bottle in an amount equivalent to 30 times that required for the saturation of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (approximately 1700 mg- SO_4 /L, 17.6 mmol- SO_4 /L [23]).

Nine H_2S generation bottles (RUNS 1–9) were prepared. H_2S in the liquid and gas phases was removed by bubbling N_2 gas when $\text{H}_2\text{S}_{\text{high}}$ was observed. $\text{H}_2\text{S}_{\text{high}}$ observation and H_2S removal were conducted three times as the acclimation stage, and then, the H_2S generation suppression test was performed.

2.5. Experimental Methods for H_2S Generation Suppression

In the H_2S generation suppression test, NO_3 was added into the H_2S generation bottle after acclimation was completed, and its effect on H_2S generation was evaluated. High (H) and low (L) concentrations of NO_3 were added, i.e., 7.0 mmol- NO_3 /L (430 mg- NO_3 /L) and 1.76 mmol- NO_3 /L (110 mg- NO_3 /L), respectively. Those concentrations are 0.4 and 0.1 times the saturation concentration of SO_4 (17.6 mmol- SO_4 /L).

Although ideally, the experiments should be conducted in individual bottles, because of limited space and budget, the same bottle (as it is, no changes of content) was used repeatedly for the experiments. After the intermittent addition test (I), the concentration maintenance test (M) was conducted in RUNS 1–6. Control experiments (no NO_3 addition) were performed in RUNS 7–9.

The procedures and the end conditions for each experiment are as follows. In the intermittent addition test, HN_R , LN_R or HN_C was added into the bottle and conditions (1)–(3) were maintained. When the H_2S generation suppression effect of NO_3 disappeared and $\text{H}_2\text{S}_{\text{high}}$ was observed, H_2S was removed by bubbling N_2 gas, and NO_3 was added again. The experiment was terminated after observing $\text{H}_2\text{S}_{\text{high}}$ six times. In the concentration maintenance test, conditions (1)–(3) were maintained, and HN_R or HN_C was added into the bottle to maintain NO_3 concentration from 100 to 430 mg- NO_3 /L. The experiment was terminated when H_2S concentration could be controlled to below 1000 ppmv for one month.

The experimental conditions and procedures are shown in Table 2. The abbreviations of the experiments are also listed in Table 2. For example, the abbreviation for high concentration (H), reagent NO_3 (N_R), and intermittent addition test (I) is $\text{HN}_\text{R}\text{I}$.

Table 2. Experimental conditions and procedures.

Experiment type	Abbreviation	Procedure	NO_3 addition (mg- NO_3 /L)	End condition	RUN No.
Acclimation		*— $\text{H}_2\text{S}_{\text{high}}$ generation— H_2S removal—back to *	0	3 times of $\text{H}_2\text{S}_{\text{high}}$ generation	1–9
High conc. nitrate (reagent) intermittent addition	$\text{HN}_\text{R}\text{I}$	*— $\text{H}_2\text{S}_{\text{high}}$ generation— H_2S removal— HN_R addition—back to *	430	6 times of $\text{H}_2\text{S}_{\text{high}}$ generation	1, 2, 3
Low conc. nitrate (reagent) intermittent addition	$\text{LN}_\text{R}\text{I}$	*— $\text{H}_2\text{S}_{\text{high}}$ generation— H_2S removal— LN_R addition—back to *	110	6 times of $\text{H}_2\text{S}_{\text{high}}$ generation	4, 5, 6

Experiment type	Abbreviation	Procedure	NO ₃ addition (mg-NO ₃ /L)	End condition	RUN No.
High conc. nitrate (compost) intermittent addition	HN _C I	*—H ₂ S _{high} generation—H ₂ S removal—HN _C addition—back to *	430	6 times of H ₂ S _{high} generation	4, 5, 6
High conc. nitrate (reagent) maintenance	HN _R M	*—< NO ₃ 100 mg-NO ₃ /L—HN _R addition—back to *	430	Maintaining <1000 ppmv H ₂ S for one month	1, 2, 3
High conc. nitrate (compost) maintenance	HN _C M	*—< NO ₃ 100 mg-NO ₃ /L—HN _C addition—back to *	430	Maintaining <1000 ppmv H ₂ S for one month	1, 2, 4
Control		*—H ₂ S _{high} generation—H ₂ S removal—back to *	0	—	7, 8, 9

2.6. Analytical Procedure

The objects of measurement were the gas and liquid phases in the H₂S generation bottle. Measurement items for the gas phase were H₂S concentration and gas volume. Measurement items for the liquid phase were pH, electric conductivity (EC), ORP, TOC, NO₃ concentration, and liquid volume.

In regard to the gas phase, H₂S was measured with a portable gas detector for worker safety (XS-2200, controlled potential electrolysis, NEW COSMOS ELECTRIC CO., LTD.). Gas volume in the gas bag was measured by reading the scale on a plastic syringe used to withdraw the gas from the bag. Then, the gas bag was newly filled with N₂ gas and reconnected to the bottle.

In regard to the liquid phase, HORIBA B-212 (pH), HORIBA B-173 (EC), HORIBA-D-55 and 9300-10D (ORP), Shimadzu TOC-V_{WS} (TOC), and HORIBA B-343 (NO₃) were used. Liquid volume was estimated by measuring the gross weight of the H₂S generation bottle. Liquid (10 mL) was sampled from the liquid sampling port (Fig. 2) with a plastic syringe. Liquid volume in the H₂S generation bottle was maintained by adding 10 mL solution containing organic matter or NO₃.

H₂S, gas volume, pH, EC, ORP, and NO₃ concentration were measured four times a week, and TOC was measured two times a week. In addition, TOC was re-measured immediately after adding organic matter.

3. Results

Changes in the concentrations of H₂S and NO₃, and the ORP in RUNS 1, 4, and 7 are shown in Fig. 3, 4, and 5, respectively. The results of RUNS 2, 3, 5, 6, 8, and 9 are omitted. The pH ranged from 5.9 to 8.8, was increased with

time, and was around 8 after 30 days. EC ranged from 3.2 to 11.9 mS/cm and was increased with time upon the addition of NO₃.

3.1. Intermittent Addition Test

ORP in HN_RI was increased upon the addition of NO₃. On the other hand, ORP in LN_RI and HN_CI did not show a constant increase. H₂S_{high} was generated when ORP was below −100 mV (standard hydrogen electrode). Regarding HN_RI and HN_CI, there were cases that NO₃ concentration was decreased to below the detection limit (50 mg-NO₃/L) on the next measurement day, i.e., after two days. Therefore, NO₃ might have disappeared in two days.

3.2. Concentration Maintenance Test

Because of the rapid disappearance of NO₃ in HN_RM, NO₃ reagent had to be added frequently. NO₃ concentration could not be maintained at an exact value because it was below the detection limit when NO₃ reagent was added. ORP was increased and exceeded 100 mV with the repeated addition of NO₃ reagent. H₂S_{high} was not generated for one month, i.e., the target time. Although NO₃ concentration was decreased slowly and NO₃ was added only two times in HN_CM, H₂S_{high} was not generated for one month. ORP was below −100 mV.

3.3. Control

ORP was below −100 mV after 10 days and H₂S_{high} was generated repeatedly in control (Fig. 5). ORP exhibited both increases and decreases, and no obvious tendency was observed. The regeneration of H₂S immediately after H₂S removal by bubbling N₂ gas was slower than that immediately before removal. NO₃ concentration was near the detection limit.

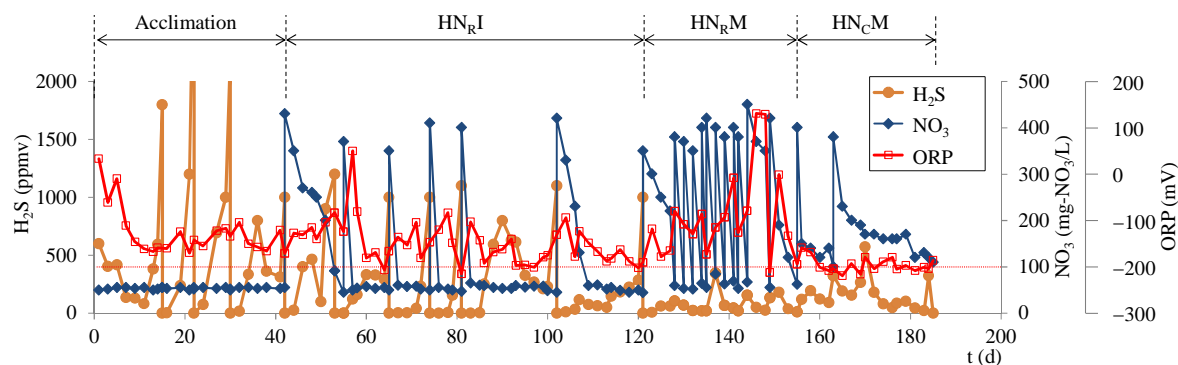


Figure 3. Concentration changes in RUN 1.

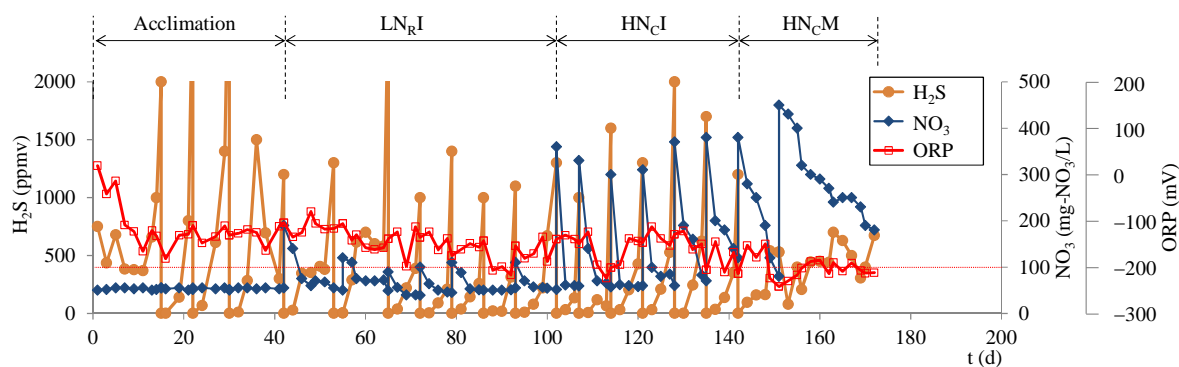


Figure 4. Concentration changes in RUN 4.

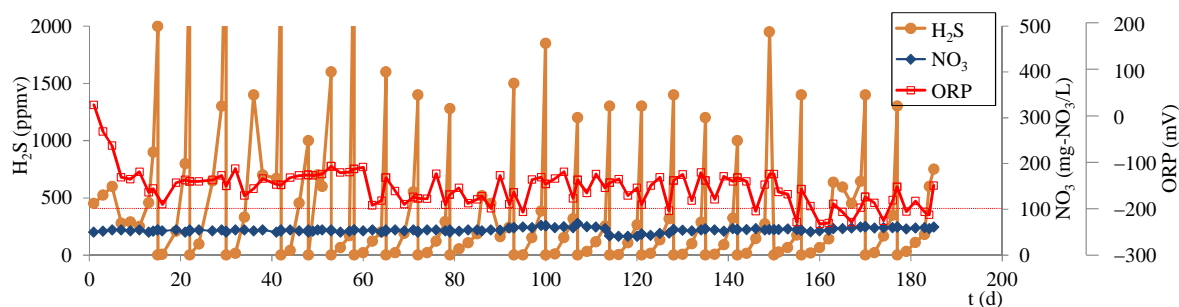


Figure 5. Concentration changes in RUN 7 (control).

4. Discussion

4.1. Quantitative Evaluation of H₂S Generation Suppression by NO₃ Addition

The effect of NO₃ addition on H₂S generation suppression was quantitatively estimated as follows. The number of days required for exceeding 1000 ppmv H₂S is shown in Fig. 6. In control, 7.6 ± 2.1 days (average \pm standard deviation) were required for exceeding 1000 ppmv H₂S. The required number of days in LN_RI and HN_CI was almost the same as that in control. On the other hand, in the case of HN_RI, 15.2 ± 8.2 days were required, i.e., twice that of control in spite of the large variance. H₂S_{high} was not generated in HN_RM and HN_CM for one month.

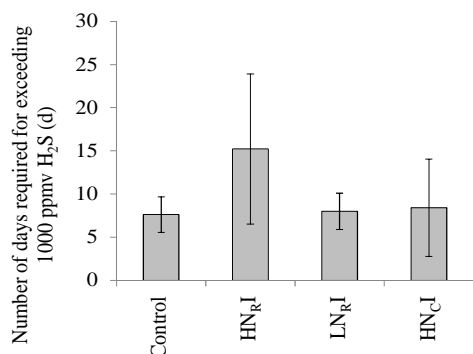


Figure 6. Number of days required for exceeding 1000 ppmv H₂S.

H₂S generation rates are shown in Fig. 7. Generation rate

was determined from the increase of H₂S concentration per unit time from the day of H₂S removal by bubbling N₂ gas to the day H₂S exceeded 1000 ppmv (However, two days were used for the concentration maintenance test). It is known that H₂S exists in not only the gas phase but also the liquid phase. Using the concentration in the gas phase, gas volume, liquid volume, and pH, concentration in the liquid phase was calculated according to Henry's law and an equilibrium relationship in the liquid phase [24]. As H₂S was generated from the liquid phase, the generation rate was converted into the rate per unit liquid volume (mmol-H₂S/(L·d)). The generation rate in control was 0.21 ± 0.05 mmol-H₂S/(L·d). The rates in LN_RI and HN_CI were the same as that in control. On the other hand, the rate in HN_RI was 0.051 ± 0.024 mmol-H₂S/(L·d), i.e., approximately 1/4 of control. In spite of the disappearance of NO₃, H₂S was not generated immediately, i.e., the generation was suppressed from 60 to 80 days. The reason for the lack of decrease of the generation rate in HN_CI is unknown. The rates in the concentration maintenance test (HN_RM and HN_CM) were below 1/20 of control. However, NO₃ concentration could not be maintained because of its rapid disappearance in HN_RM.

NO₃ addition rates are shown in Fig. 8. The rates in HN_RI, LN_RI, HN_CI, and HN_CM ranged from 0.20 to 0.71 mmol-NO₃/(L·d), whereas that in HN_RM was more than 3-fold, i.e., 2.2 mmol-NO₃/(L·d). This is because the experimental conditions required that NO₃ concentration be maintained at 100 to 430 mg-NO₃/L, i.e., it was not our purpose to determine the minimum concentration of NO₃ required to suppress H₂S_{high} generation. In addition, NO₃ disappeared rapidly. Therefore, it is possible to suppress

$\text{H}_2\text{S}_{\text{high}}$ generation even if the NO_3 addition rate is below 2.2 $\text{mmol-NO}_3/(\text{L}\cdot\text{d})$. For example, if HN_{RI} was regarded as having H_2S generation suppression effect because of the decreased H_2S generation rate, $\text{H}_2\text{S}_{\text{high}}$ generation was possibly suppressed by the above NO_3 addition rate of 0.46 $\text{mmol-NO}_3/(\text{L}\cdot\text{d})$ in HN_{RI} .

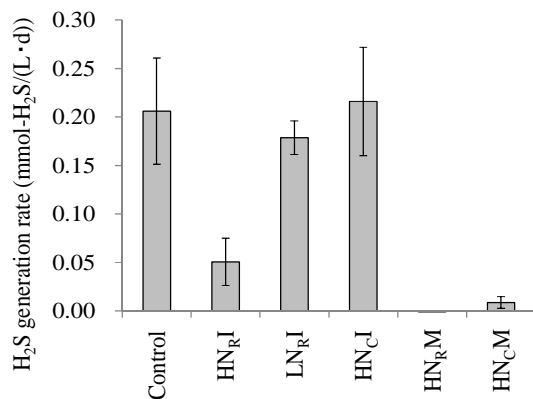


Figure 7. H_2S generation rates.

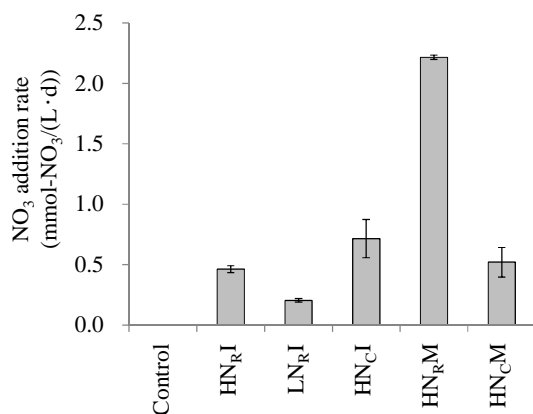


Figure 8. NO_3 addition rates.

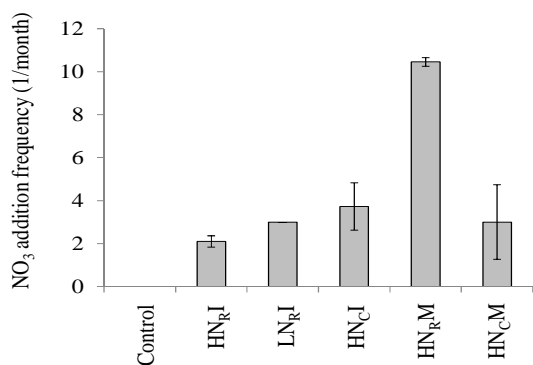


Figure 9. NO_3 addition frequencies.

NO_3 addition frequencies are shown in Fig. 9. The frequencies in HN_{RI} , LN_{RI} , HN_{CI} , and HN_{CM} ranged from 2.1 to 3.7 1/month, whereas that in HN_{RM} was more than twice those values, i.e., 10.5 1/month. The reason is the same as that for the NO_3 addition rate.

The following is a summary of the above results. By adding NO_3 reagent at the rate of 0.46 $\text{mmol-NO}_3/(\text{L}\cdot\text{d})$ into a liquid from which H_2S gas was generated at the rate of 0.21

± 0.05 $\text{mmol-H}_2\text{S}/(\text{L}\cdot\text{d})$, H_2S generation rate was decreased to approximately 1/4 of the original rate. Although it was difficult to maintain the concentration of NO_3 because of its rapid disappearance, by frequently adding NO_3 reagent to maintain a concentration of approximately 430 $\text{mg-NO}_3/\text{L}$, H_2S concentration could be controlled to below 1000 ppmv for one month, and H_2S generation rate was below 1/20 of the original rate.

4.2. Problems Encountered, Application of Results, and Future Issues

Regarding the addition of NO_3 derived from compost, it was assumed that the frequent addition of NO_3 in HN_{CM} was required because of the rapid disappearance of NO_3 , in the same manner as that in HN_{RM} . However, the results (Fig. 3) indicated that the decrease of NO_3 concentration was slow in HN_{CM} , and $\text{H}_2\text{S}_{\text{high}}$ generation was suppressed by the addition of NO_3 in small amounts. In addition, the disappearance of NO_3 proceeded more slowly in HN_{CM} than in HN_{CI} in spite of use of the same NO_3 derived from compost (Fig. 4). The internal environment might have been changed because some experiments were continuously conducted in the same bottle after another experiment was completed. Compost contains stable organic matter, such as humic substances [25]. A larger amount of stable organic matter was contained in compost made from sewage sludge than in compost made from food waste [26]. The hydrophobic domains in humic substances prevent microbial activity associated with water [27]. In this study, medium for microbial culture was added as organic matter to maintain TOC at 700–1000 mg-C/L . However, when extract from compost was used for NO_3 addition, humic substances contained in compost would be included as well. As humic substances are also organic matter, they would increase TOC in the bottle and consequently decrease the amount of organic matter derived from medium for microbial culture. Thus, the authors speculate that H_2S generation potential was decreased in HN_{CM} because TOC derived from humic substances, which are stable organic matter, was increased and consequently, the net amount of TOC available to SRB was decreased. This result led to two suggestions: the H_2S generation suppression effect in HN_{CM} was overestimated, and H_2S generation potential did not increase in spite of the increase of TOC due to humic substances contained in compost (availability of use at an actual landfill site).

The amount of NO_3 to be added to a waste layer of a landfill site where H_2S is generated can be estimated from the results obtained in this study. Future issues are as follows. In regard to the experiment using NO_3 derived from compost, additional experiments are required with control of the net amount of organic matter. Environmental loading, such as eutrophication by application of the NO_3 addition method at a pilot scale or an actual landfill site, and cost reduction by using useless NO_3 in the market should be evaluated. As NO_3 addition is an alternative method for supplying oxygen, its advantages should be evaluated quantitatively. The advantages include no consumption of large amounts of NO_3

in the aerobic zone and consumption of organic matter by denitrification. Specifically, the decomposition of organic matter has to rely on anaerobic decomposition because oxygen supply via gas venting pipes to the waste layer at a coastal landfill site, which has a large amount of retained inner leachate, is not feasible. If both H_2S generation suppression and the consumption of large amounts of organic matter by denitrification could be realized, stabilization of waste layer at landfill site would be accelerated.

5. Conclusion

In this study, in order to develop a method to suppress H_2S generation by NO_3 addition at a landfill site, the extent of suppression was quantitatively estimated. When NO_3 reagent was added at the rate of $0.46 \text{ mmol-NO}_3/(\text{L}\cdot\text{d})$ into a liquid from which H_2S gas was generated at the rate of $0.21 \pm 0.05 \text{ mmol-H}_2\text{S}/(\text{L}\cdot\text{d})$, H_2S generation rate was decreased to approximately 1/4 of the original rate. Although it was difficult to maintain the concentration of NO_3 because of its rapid disappearance, by frequently adding NO_3 to maintain a concentration of approximately $430 \text{ mg-NO}_3/\text{L}$, the concentration of H_2S would be controlled to below 1000 ppmv for one month and H_2S generation rate would be reduced to below 1/20 of the original rate. The disappearance of NO_3 derived from compost was slow and H_2S generation was suppressed by the addition of a small amount of NO_3 . Additional experiments are required as the net amount of organic matter for SRB could not be controlled.

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