

Effects of pollutant concentration ratio on the simultaneous removal of NH₃, H₂S and toluene gases using rock wool-compost biofilter

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Abstract

The biological treatment of a tri-component mixed waste gas system in BRC1 and BRC2 biofilters packed with rock wool-compost media was studied. The model gases were NH₃, H₂S and toluene. The gases were fed initially at about 50–55 ppm each. H₂S was found to have the shortest start-up while toluene had the longest. Under two different NH₃:H₂S:toluene concentration ratios of 250:120:55 and 120:220:55 (in ppm) for BRC1 and BRC2, the removal efficiencies of NH₃, H₂S and toluene were found to be affected by their respective loading rate. On the other hand, toluene removal was observed to be inhibited at H₂S concentration of 220 ppm as well. Almost complete removal of NH₃ and H₂S was achieved when loading rate was applied up to 16.14 g-NH₃/(m³ bed h) and 36.09 g-H₂S/(m³ bed h), respectively. The maximum elimination capacity for NH₃ was determined to be 23.67 g-NH₃/(m³ bed h) at 78.6% removal efficiency and for H₂S, 38.50 g-H₂S/(m³ bed h) at 68.1% removal efficiency. The maximum toluene elimination capacity was 30.75 g-toluene/(m³ bed h) at 87.9% removal efficiency when the concentration of NH₃:H₂S:toluene was 250:120:55 in BRC1, and was 16.60 g-toluene/(m³ bed h) at 45.5% removal efficiency when the concentration of NH₃:H₂S:toluene was 120:220:55 in BRC2. The pressure drops along both columns were low and the ratio of bed compactions over biofilter height was observed to be less than 0.02.

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1. Introduction

Ammonia (NH₃), hydrogen sulfide (H₂S) and volatile organic compounds (VOCs) are dominant air pollutants released simultaneously from livestock farming, wastewater and night soil treatment, composting and food processing plants, and various industrial processes plants including petrochemical refining, fuel treatment, and pharmaceuticals [1–3]. Biofilter has been known as an effective waste gas control technology for these compounds which are associated with olfactory nuisance and health risks. It has significant economical and operational advantages over other physical and chemical methods such as incinera-

tion, wet scrubbing and adsorption [4,5]. The biofilter process has appropriate applicability for large gas volumes of compounds at low cost of maintenance, and produces harmless by-products.

Many researchers have investigated the treatment of odorous compounds or VOCs as single pollutant in a biofilter [6–12]. However, in real systems, such as publicly owned treatment works (POTWs), emissions usually contain more than one odorous gases or VOCs including ammonia, hydrogen sulfide, benzene, toluene, chloroform and so on [13]. The concentrations of these compounds vary in proportion to each other. Consequently, treatment of a mixed gas system has been one of the major challenges in biofilter. Binary system of the odorous compounds NH₃ and H₂S have been studied in many researches [1,14–16]. Chung et al. observed an inhibitory behavior for the NH₃ and H₂S system during range of high gas concentrations [15,16]. Malhautier et al. reported a negative effect of high

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Table 1
Mineral medium composition for cultivation of each microbial strain

NH ₃ -oxidizing (AMM strain)	Sulfur-oxidizing (<i>Pseudomonas</i> sp. SUL4)	Toluene-degrading (<i>Pseudomonas</i> sp. TAS4B)
Na ₂ HPO ₄ : 1.0 g/L	KH ₂ PO ₄ : 2.0 g/L	KH ₂ PO ₄ : 5.0 g/L
CH ₃ COONa: 1.0 g/L	K ₂ HPO ₄ : 2.0 g/L	K ₂ HPO ₄ : 4.5 g/L
KH ₂ PO ₄ : 0.3 g/L	NH ₄ Cl: 0.4 g/L	(NH ₄) ₂ SO ₄ : 2.0 g/L
NH ₄ Cl: 26.8 g/L	MgCl ₂ ·6H ₂ O: 0.2 g/L	Mg SO ₄ ·7H ₂ O: 0.34 g/L
Yeast extract: 5.0 g/L	FeCl ₃ ·6H ₂ O: 0.02 g/L	Trace elements: 200 μL/L
Deionized water: fill to 1 L	Na ₂ S ₂ O ₃ ·5H ₂ O: 8.0 g/L	Deionized water: fill to 1 L
	Yeast extract: 5.0 g/L	300 ppm toluene ^a
	Deionized water: fill to 1 L	

^a To be added after autoclaving (in liquid form).

amount of H₂S on the activity of nitrifying bacteria [14] while some studies have been focused on VOC systems like BTEX and paint solvent mixtures [17–20]. There are a few studies on mixed system of odorous gases and VOCs. Cox and Deshusses reported that co-treatment of H₂S and toluene can be obtained in a single-stage biotrickling filter in both acidic and neutral pH range without conflicting pH optima between H₂S-degrading bacteria and toluene-degrading bacteria [13]. In a study on the removal of a VOC, NH₃ gas can play a role of nutrient for microorganisms in removing VOC, resulting in improvement of VOC removal efficiency [21].

In this work, the simultaneous removal of model gases NH₃, H₂S and toluene was studied to understand interactions among these three gas components in terms of removal efficiency and elimination capacity at different concentration ratios of each component. The packing material used for concurrent removal of the mixed gas was rock wool-compost media. This composite media was developed by incorporating rock wool with compost in firm ball shape. Rock wool is a fibrous material usually used in hydroponics due to good water holding and buffering capacity, high porosity, large surface area and high chemical persistence [22]. On the other hand, compost is commonly used as organic

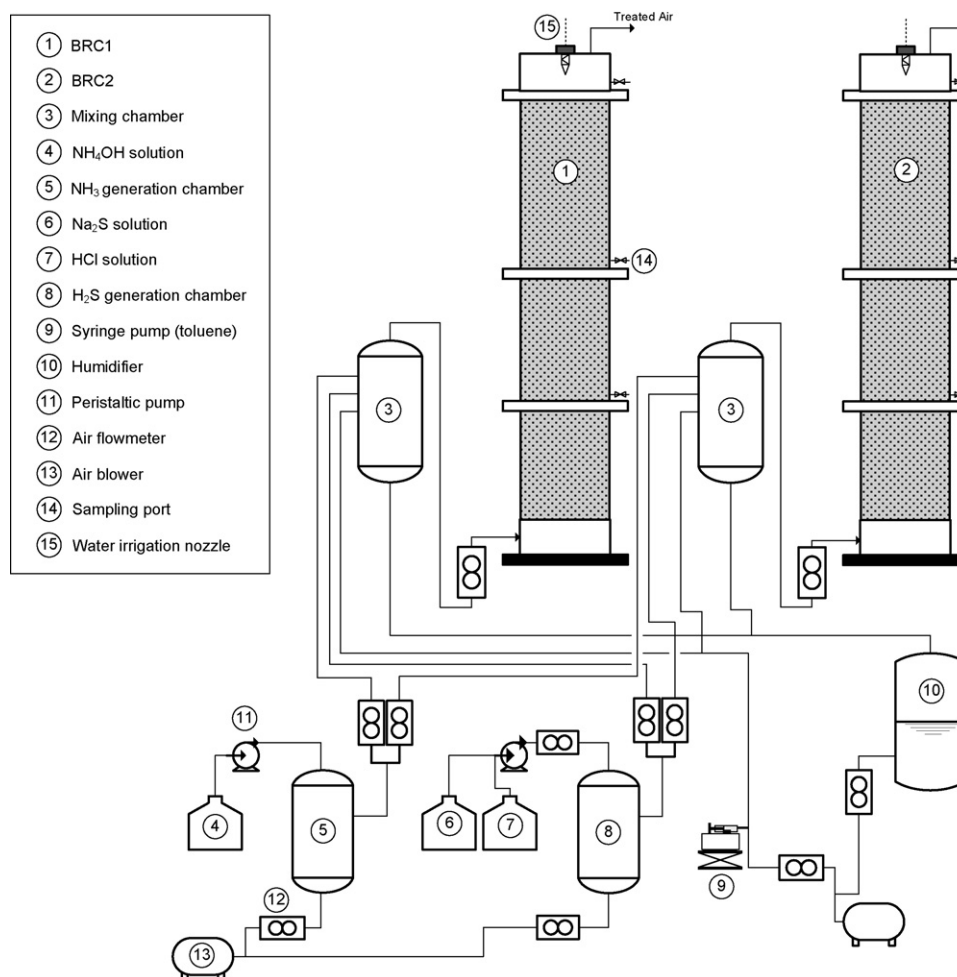


Fig. 1. Schematic diagram of the mixed gas experiment.

packing material because of its diverse microbial population and inherent nutrients. Therefore, the composite media of rock wool-compost having advantages of both organic and inorganic media would offer great potential for high microbial population with reduction of compacting tendency and pressure drop in the biofilter bed.

2. Materials and methods

2.1. Biofilter media and inoculation

The media was prepared by mixing rock wool and compost at 70:30 weight ratios. Binding solution of 20% polyvinyl alcohol (PVA) was initially prepared. To a 130 g rock wool-compost mixture, 30 mL of PVA solution and a solution of 7.5 g bentonite in 15 mL water were added. Granulated activated carbon at 2.5% (w/w) was also added to increase adsorption capacity. After thorough mixing, the resulting mass was passed through an extruder and molded into spherical shapes with 0.8–1.0 cm diameter. After which, the media were placed in a drying oven at 60 °C for 4 h. The media's properties were determined as: (i) bulk density, 0.53 g/mL, (ii) true density, 2.44 g/mL, (iii) porosity, 78.5%, and (iv) water holding capacity, 0.72 g H₂O/g medium. Initial moisture content and packing density were determined to be 40% and 820 kg/m³, respectively. The biofilter media were seeded with specific strains grown in mineral medium as shown in Table 1. The strains were previously isolated from activated sludge taken from the Y municipal wastewater treatment plant (Gyeonggi-do, Korea). The strains used were *AMM*, *Pseudomonas* sp. SUL4 and *Pseudomonas* sp. TAS4B strains for degradation of NH₃, H₂S and toluene, respectively.

2.2. Experimental setup and analytical methods

The schematic diagram of the experiment is shown in Fig. 1. The two biofilters, 10 cm in diameter and 54 cm in height, were designated as BRC1 (biofilter with rock wool-compost media) and BRC2. Ammonia solution (NH₄OH), delivered at a controlled rate by a peristaltic pump, was bubbled up with air to produce NH₃ gas which was directed to the mixing chambers. H₂S, on the other hand, was generated from the reaction of disodium sulfide (Na₂S) and hydrochloric acid (HCl) solutions. Toluene (Sigma–Aldrich, 99.8% HPLC Grade) was introduced by a syringe pump (Model 200 KD Scientific, USA) into a T-type stainless connector where a low flow air stream was flowing and directed towards the mixing chambers. The biofilters were operated in an upflow mode with the inlet port located at the bottom-side of the columns. Table 2 lists the operating parameters for the biofilters and Table 3 shows the pollutant inlet concentrations for BRC1 and BRC2 during the experimental run. The biofilters were operated at a constant empty bed retention time (EBRT) of 25 s.

Three sampling ports were positioned 16, 38 and 54 cm from the bottom of the biofilters. NH₃ concentration was analyzed by impingement method of the gas stream onto a 0.05 N H₂SO₄ solution, from which the NH₃ concentration was deter-

Table 2
Operating parameters for the biofilter setup

Bed volume (L)	4.24
Air flow rate (L/min)	10
Empty bed residence time (EBRT) (s)	25
Inlet gas relative humidity (%)	85–95
Bed temperature (°C)	26–28
Water spray (mL/day)	500; one-time frequency within 1.5 min

mined using an NH₃ selective ion probe (Orion, USA). H₂S concentration was determined using an automatic gas analyzer (Multi-RAE PLUS PGM-50, USA). Higher concentrations of NH₃ and H₂S were determined using gas detection tubes (Gastec, Tokyo, Japan) with 0–500 ppm measurable range. About 500 µL of gas sample was taken from each port and analyzed for toluene using a gas chromatograph equipped with a flame-ionization detector (HP 6890 Series GC-FID System). The GC carrier gas was nitrogen and the operational conditions were: inlet temperature, 200 °C; initial oven temperature, 80 °C; final oven temperature, 150 °C; oven ramp, 10 °C/min; oven post-run temperature, 200 °C; detector temperature, 250 °C. Toluene retention time was at 4.97 min.

Leachate taken daily from the bottom of the biofilters was analyzed for pH (Thermo Orion, USA). Nitrate/nitrite and sulfate concentrations of the leachate were determined by spectrophotometer (Bran+Lubbe Automatic Analyzer 3) and ion chromatograph (Waters), respectively. Biofilter media samples taken at different column height at the end of the experiment were also analyzed for pH, microbial count and pressure drop. Pressure drops at different column heights were determined using a digital manometer (Dwyer Series 477, USA).

3. Results and discussion

3.1. Ammonia, hydrogen sulfide and toluene removal efficiencies at various pollutant ratios

Start-up and steady-state performances of BRC1 and BRC2 were evaluated in terms of removal efficiency. Removal efficiency (RE) is the fraction of the pollutant removed by the biofilter. It is expressed as percentage and can be calculated

Table 3
Pollutant concentration for the biofilters

Load (ppm)	Time (day)	BRC1	BRC2
NH ₃	0–8	50 ± 3	49 ± 5
	9–18	120 ± 14	121 ± 16
	19–50	250 ± 24	118 ± 12
H ₂ S	0–8	49 ± 3	50 ± 4
	9–18	120 ± 7	120 ± 8
	19–50	119 ± 13	220 ± 21
Toluene	0–13	55 ± 8	55 ± 6
	14–24	100 ± 10	100 ± 4
	25–50	55 ± 6	55 ± 4

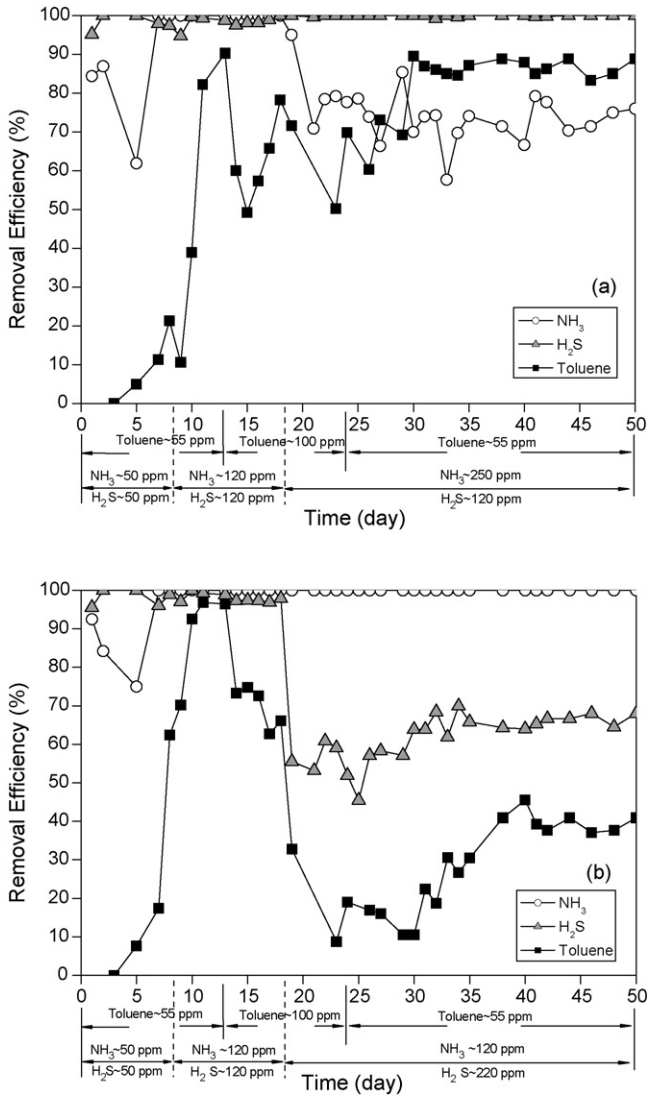


Fig. 2. Pollutant removal efficiencies of (a) BRC1 and (b) BRC2.

by Eq. (1) where C_{Gi} = inlet concentration (ppm, g/m^3) and C_{Go} = outlet concentration (ppm, g/m^3) [4]:

$$RE = \left(\frac{C_{Gi} - C_{Go}}{C_{Gi}} \right) \times 100 \quad (1)$$

During the initial or start-up phase, BRC1 and BRC2 were fed with simulated waste gas containing almost same concentrations of NH_3 , H_2S and toluene at around 50–55 ppm, corresponding to loading rates of about 5.4 $g-NH_3/(m^3 h)$, 12.0 $g-H_2S/(m^3 h)$ and 31.9 $g-toluene/(m^3 h)$, respectively. The removal efficiency of each gas pollutant in the BRC1 and BRC 2 was represented in Fig. 2. The sulfate concentration, pH and nitrate/nitrite concentrations of leachate that percolated from BRC1 and BRC2 were plotted in Fig. 3. In both biofilters, complete removal of H_2S was observed from day 2 (Fig. 2). It was ascribed to the physical adsorption of H_2S gas onto the media because insignificant amount of oxidized sulfate from H_2S was measured in leachate of the biofilter as shown in Fig. 3(a). Initial NH_3 removal efficiency of more than 85% obtained from both biofilters decreased

by about 20% during the first five days of operation (Fig. 2). A high pH of 8–8.8 maintained for the first 5 days of operation could explain lower ammonia removal compared to H_2S for the same duration, since most of ammonia gas exist in the form of ammonium ion when pH level is less than neutral (Fig. 3(b)). A complete NH_3 removal was achieved in both biofilters after day 5 (Fig. 2) when pH decreased with production of high sulfate concentration caused by biodegradation of H_2S (Fig. 3(a) and (b)). On the other hand, gradual increase in toluene removal was observed after day 5. It also has been reported in toluene biofiltration that the acclimation period required for toluene

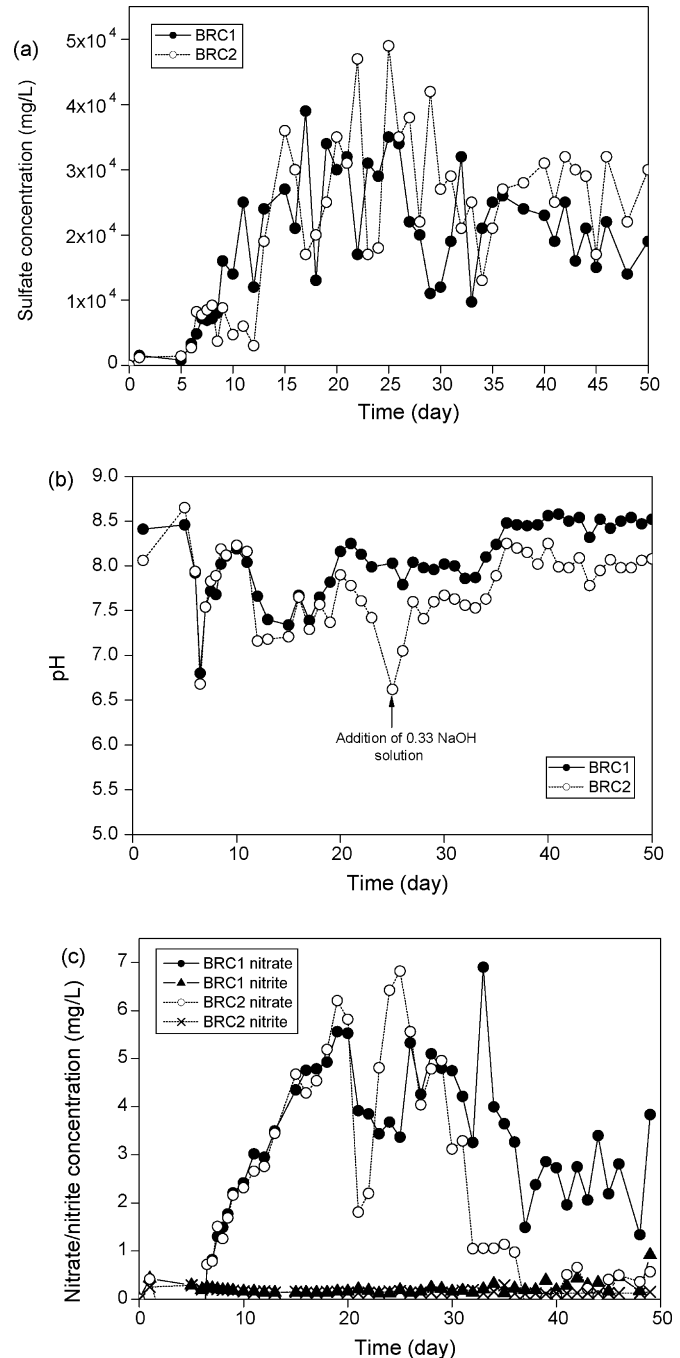


Fig. 3. Sulfate concentration (a), pH (b) and nitrate/nitrite concentrations (c) of BRC1 and BRC2 leachates.

degradation was about 7–20 days at inlet loading range of about 6–34 g/(m³ h) [7,13,23].

From days 9 to 18, the concentrations of NH₃ and H₂S in the inlet stream were increased to about 120 ppm. The removal efficiencies of NH₃ and H₂S were ~100% and 97–100%, respectively. Toluene removal efficiency continuously increased and achieved more than 90%. It indicates that toluene-degrading bacteria TAS4B required more than 1 week to be acclimated as stated by other researchers, and the activity of TAS4B was not inhibited by the co-existence of NH₃ and H₂S compounds at the concentration of 120 ppm. An inlet toluene concentration of 100 ppm applied on day 13 showed immediate decline in toluene removal efficiency in both biofilters. Although the removal efficiency of toluene gradually increased from days 15 to 18 in BRC1, it was apparent that the activity of TAS4B was inhibited by a high inlet toluene concentration itself or relatively insufficient EBRT of 25 s provided for removing inlet toluene concentration applied to the system. The EBRT for toluene biofiltration in many studies was applied in the range of 0.8–5 min [7,23–25]. The removal of toluene decreased with increasing toluene load due to insufficient contact time between the gas and the biofilm [23]. On the other hand, both odorous components NH₃ and H₂S were removed above 98%. This implies that the performance of simultaneous removal of NH₃ and H₂S was not negatively affected by the increase in toluene loading up to 100 ppm.

On day 19, the inlet concentrations of NH₃ in BRC1 and of H₂S in BRC2 were increased. The NH₃:H₂S:toluene ratio of the inlet gas to BRC1 was 250:120:100 (in ppm) from days 19 to 24. The high NH₃ concentration did not affect the 100% H₂S removal efficiency as reported by Malhautier et al. [14] and Chung et al. [26]. However, the NH₃ removal efficiency decreased, which might be attributed to the high inlet NH₃ concentration itself. In BRC2, an abrupt drop in the removal efficiency of both H₂S and toluene was observed as inlet H₂S concentration was increased to 220 ppm (NH₃:H₂S:toluene = 120:220:100). The activity of the toluene-degrading strains may have been inhibited by the acidification of the system due to higher production of sulfuric acid as consequence of high H₂S oxidation. This was evident from the high sulfate concentration and lower pH in leachate of the BRC2 compared to the BRC1 (Fig. 3(a) and (b)). On the contrary, the NH₃ removal efficiency in BRC2 was maintained at almost 100% irrespective of high inlet H₂S concentration.

In order to determine the effect of high inlet load of NH₃ or H₂S on toluene removal, inlet toluene concentration was decreased to 55 ppm at which toluene removal efficiency was not limited by its load for the first 13 days of operation. The ratio of NH₃:H₂S:toluene (in ppm) was 250:120:55 in BRC1 and 120:220:55 in BRC2 from days 25 to 50. The toluene removal efficiency in BRC1 gradually increased and was stable to about 90% which was similar to the removal efficiency obtained at the ratio of NH₃:H₂S:toluene of 120:120:55 after the TAS4B acclimated (days 10–14). Therefore, the high inlet NH₃ applied in this study did not adversely affect the toluene removal. The excess NH₃ (aqueous) would rather be utilized

by the toluene-degrading microorganisms as nutrient source. According to a study by Morales [21], injecting gaseous NH₃ to a biofilter enhanced toluene removal performance. Song [20], on the other hand, used nitrate solutions in their studies on the influence of nitrogen supply on the removal of paint VOC mixtures. In BRC2, in order to counter the reduction of pH due to the oxidation of a high inlet H₂S concentration, base (0.33N NaOH solution) was added with the irrigation water on day 26. The toluene removal efficiency gradually increased but to only about 40% (day 50) even with the inlet toluene concentration decreased to 55 ppm and adjustment of pH to above 7.5. This removal efficiency was significantly lower than that achieved at the ratio of NH₃:H₂S:toluene of 120:120:55 (days 10–14). Therefore, toluene removal performance was limited at inlet toluene concentration of about 100 ppm due to mass transfer limitation and inlet H₂S concentration of 220 ppm as well.

Fig. 3(c) shows the concentrations of nitrate/nitrite in leachate from the biofilter. Unlike sulfate concentration, a significantly low concentrations of nitrate/nitrite were measured, implying that there was little nitrification. Pinnette et al. reported that biological ammonia removal in many reported research of biofiltration may be disguised by absorption in moisture present in the biofilter and adsorption onto the surface of the organic material. After which, it would be washed out during water irrigation, resulting in significant ammonia in the leachate removed from the biofilter [27]. Cho et al. reported that in the presence of H₂S, NH₃ was removed to (NH₄)₂SO₄ by chemical neutralization with SO₄²⁻ oxidized from H₂S [28,29]. A poor nitrification in the present study might be attributed to inhibitory effect of sulfide on the growth of nitrifying bacteria, reducing the efficiency of NH₃ biooxidation [14,30–33]. It was reported that even low concentration of sulfide (0.5 mg/L) had a considerable negative effect on nitrification activity [30]. In the study by Joye and Hollibaugh, nitrification reduced by 50% and 100% with addition of 60 and 100 μM of hydrogen sulfide, respectively. Chung et al. reported that NH₃ removal was not affected by the coexistence of H₂S provided gas retention time was sufficient. They suggested the retention time of at least 65 s to obtain greater than 95% biological ammonia removal efficiency in the presence of H₂S at 6.25 g-S/(m³ h). The critical loading of NH₃ for its 100% removal and the maximal loading corresponding to about 50% removal was 4.2 g-N/(m³ h) and 16.2 g-N/(m³ h) [33], which was lower than those applied in the present study. Hence, a relatively short EBRT of 25 s could be another possible cause for insignificant nitrification.

3.2. Pollutant concentration and pressure drop at different bed heights

Fig. 4 shows the pollutant concentration profile at different biofilter heights obtained on day 50 of the experimental run. The three sampling ports located at 16, 38 and 54 cm from the bottom of the biofilters are represented in terms of H/H_0 as 0.3, 0.7 and 1.0, respectively. H₂S in the BRC1 was completely removed at H/H_0 of 0.7, indicating that the biofilter may be

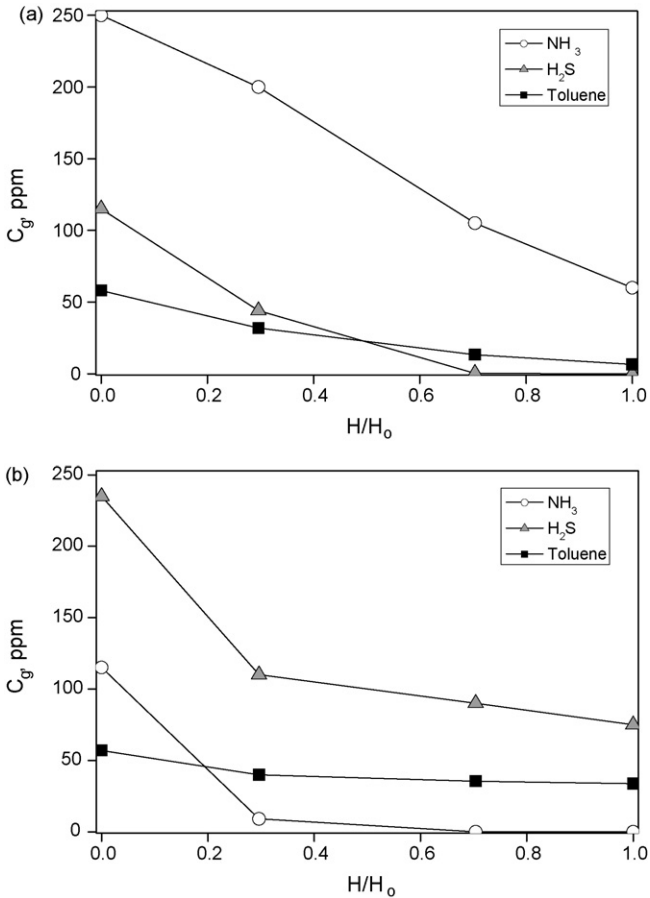


Fig. 4. Pollutant concentration profile of (a) BRC1 and (b) BRC2 at different column heights on day 50.

able to treat higher loading of H_2S than the applied. However, it should be noted that there may have a negative effect on toluene removal as shown in Fig. 2. The NH_3 was gradually removed over the column, indicating that inlet NH_3 at a high loading was adsorbed over the column height, while sulfur-oxidizers and toluene-degraders were mainly active in the bottom and middle sections of the BRC1 biofilter. In BRC2, most of the NH_3 were removed at H/H_0 of 0.3. H_2S and toluene were also removed mostly from the bottom of the column, indicating that the activity of these compounds degrading bacteria might occur primarily at the bottom section of the BRC2 biofilter. The summed final outlet concentration of NH_3 , H_2S and toluene was apparently lower at the end of the experiment when inlet mixed gases ratio of $NH_3:H_2S:toluene$ was 250:120:55 (BRC1) than 120:220:55 (BRC2).

Media samples taken at different column heights of the biofilters on day 50 were analyzed for moisture content and pH. Moisture contents of the samples were determined to be between 40% and 45% (data not shown) which was suitable for microbial activity. Fig. 5 shows the pH of samples at different H/H_0 . Media pH of about 7.25 was measured in BRC1 until H/H_0 of 0.7 or correspondingly 38 cm from the bottom. At this height, H_2S was completely removed as previously shown in Fig. 4. Hence, above this point, no sulfate may be produced and pH increased. A large portion of H_2S was removed in BRC2 at H/H_0 of 0.3 or

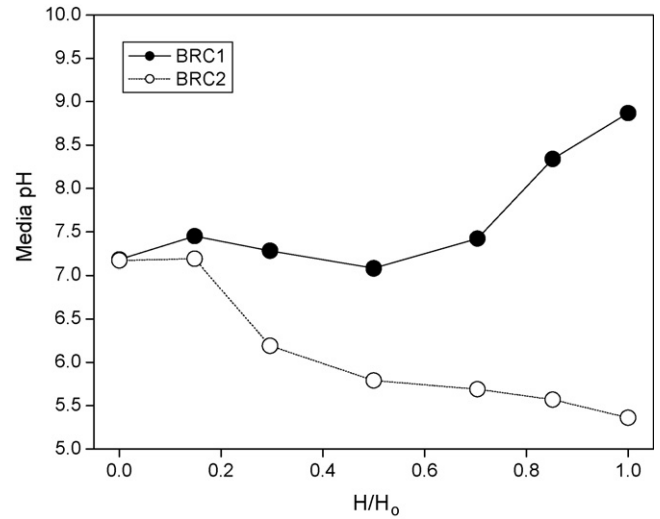


Fig. 5. pH of biofilter media samples taken at different bed heights on day 50.

at 16 cm from the bottom, resulting in a high sulfate production and decrease in pH to around 6.

Fig. 6 shows the pressure drops along the BRC1 and BRC2 measured at different bed heights. Pressure drops of 3.7 and

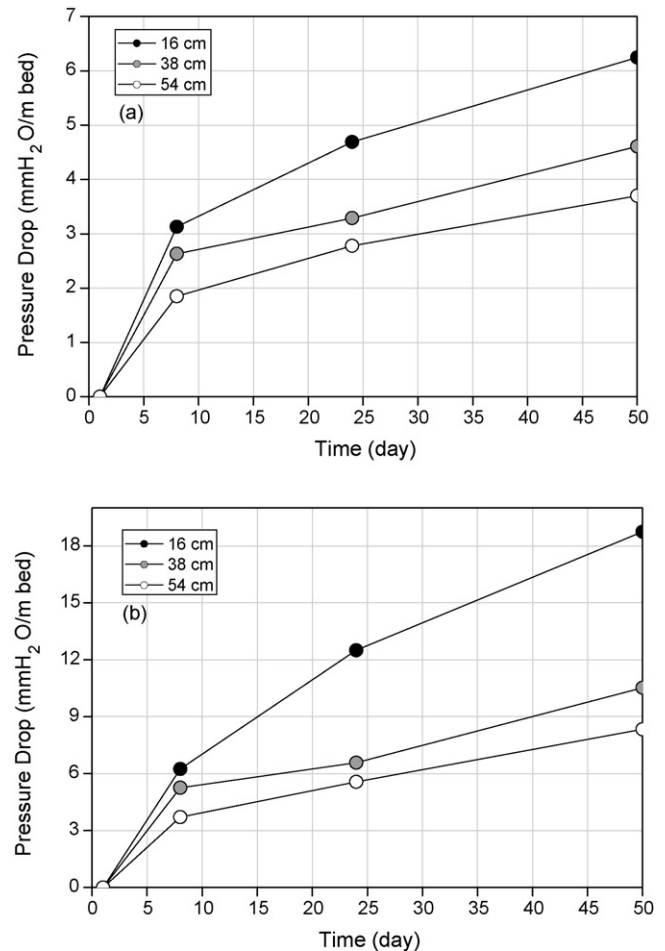


Fig. 6. Pressure drop profiles of BRC1 (a) and BRC2 (b) determined at different bed heights.

8.3 mm H₂O/m of bed were obtained at the top of BRC1 and BRC2, respectively. The higher pressure drop in the BRC2 resulted from thicker sulfur accumulation that was visually observed on the surface of the BRC2 rock wool-compost media, suggesting incomplete H₂S metabolism of the *Pseudomonas* sp. SUL4 strain at higher inlet H₂S concentration. Duan et al. also reported observation of white deposits on the pellet activated carbon media that was proportional to the rate of H₂S loading. Both biomass and elemental sulfur are most likely to cause fouling and blocking of the media bed, which may result in the less efficient biofilter performance as result of mass transfer rate limitation [11]. The accumulated residual sulfur itself may also have contributed to suppressing the activity of the toluene-degrading strains causing poor toluene removal rate in BRC2, as reported by Chung [16]. Nevertheless, the pressure drops obtained were comparable with the 8 and 11 mm H₂O/m for biofilters packed with 18 and 24 pores/cm carbon foams, respectively [5]. The obtained values were lower than the reported 10–41 and 5–30 mm H₂O/m bed pressure drops for compost and granulated sludge-packed biofilters, respectively [34]. The spherical shape of the rock wool-compost media used in this study might have allowed good air flow and distribution along the filter bed, showing that the bed heights of BRC1 and BRC2 compacted to only less than 1 cm as measured on day 50.

3.3. Elimination capacities

Elimination capacity refers to the mass of pollutant degraded per unit volume of the filter material per unit time, defined in Eq. (2) where C_{Gi} = inlet concentration (ppm, g/m³); C_{Go} = outlet concentration (ppm, g/m³); V_f = filter bed volume (m³); Q = air flow rate (m³/h) [4]:

$$EC = \left(\frac{C_{Gi} - C_{Go}}{V_f} \right) \times Q \quad (2)$$

The elimination capacities for NH₃, H₂S and toluene of BRC1 and BRC2 were plotted against their respective pollutant mass loadings in Fig. 7. The straight line indicates 100% elimination of the target pollutant. In the case of toluene elimination curves, the data points were collected from days 25 to 50 since wide fluctuation in the toluene removal was observed for the first 25 days due to the requirement of about 10 days acclimation period and high loading of toluene applied from days 13 to 24. As noted, the removal of NH₃ and H₂S in the tri-component mixed gas was affected by their respective loading rate. The maximum elimination capacities for NH₃ and H₂S were determined to be 23.67 g-NH₃/(m³ h) and 38.50 g-H₂S/(m³ h), respectively. The maximum EC values are relatively higher than those obtained from other NH₃-H₂S binary studies [14–16], albeit this study includes toluene as a major component of the inlet waste gas. The highest toluene elimination capacity was 30.75 g-toluene/(m³ bed h) when the concentration of NH₃:H₂S:toluene was 250:120:55 in BRC1, and was 16.60 g-toluene/(m³ bed h) when the concentration of NH₃:H₂S:toluene was 120:220:55 in BRC2.

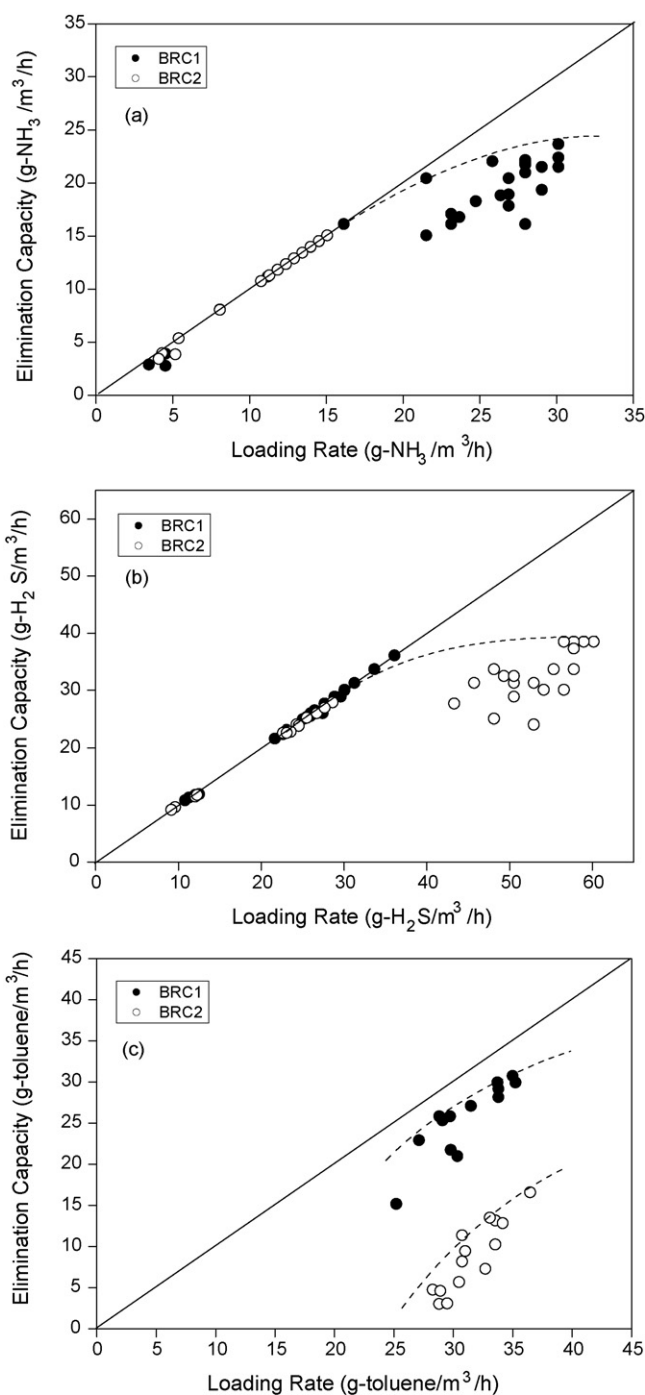


Fig. 7. Elimination capacity curves for NH₃ (a), H₂S (b) and toluene (c) obtained for BRC1 and BRC2.

4. Conclusion

The biofiltration of a mixed gas system containing NH₃, H₂S, and toluene was investigated in biofilters packed with rock wool-compost media. The rock wool-compost media performed well as a new packing material in the treatment of mixed waste gas. H₂S removal showed the shortest start-up while toluene had the longest. NH₃ removal efficiency was not affected by H₂S loading rate and vice versa, but rather by their respective loading rates. However, most of NH₃ removal in this study

was not accomplished by ammonia oxidation but by physico-chemical reaction, which might be attributed to the presence of H₂S. Toluene removal was also limited by its high loading of 100 ppm. In addition, the high loading of H₂S (220 ppm) has negative effect on toluene removal. From experiment with different ratios of tri-component gases, it was determined that the resulting pressure drop from BRC1 and BRC2 were relatively low at 3.7 and 8.3 mmH₂O/m of bed, respectively. No significant bed compaction was noted. Further study in molecular aspects is required for better understanding of the interaction of these gaseous compounds.

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