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Nitrogen utilization and biomass yield in trickle bed air biofilters

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ABSTRACT

Nitrogen utilization and subsequent biomass yield were investigated in four independent lab-scale trickle bed air biofilters (TBABs) fed with different VOCs substrate. The VOCs considered were two aromatic (toluene, styrene) and two oxygenated (methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK)). Long-term observations of TBABs performances show that more nitrogen was required to sustain high VOC removal, but the one fed with a high loading of VOC utilized much more nitrogen for sustaining biomass yield. The ratio N_{consumption}/N_{growth} was an effective indicator in evaluating nitrogen utilization in the system. Substrate VOC availability in the system was significant in determining nitrogen utilization and biomass yield. VOC substrate availability in the TBAB system was effectively identified by using maximum practical concentrations in the biofilm. Biomass yield coefficient, which was driven from the regression analysis between CO₂ production rate and substrate consumption rate, was effective in evaluating the TBAB performance with respect to nitrogen utilization and VOC removal. Biomass yield coefficients (g biomass/g substrate, dry weight basis) were observed to be 0.668, 0.642, 0.737, and 0.939 for toluene, styrene, MEK, and MIBK, respectively.

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1. Introduction

Biofiltration has emerged as a reliable and cost-effective technology in treating volatile organic compounds (VOCs) emitted from organic or petroleum based solvents [1]. Trickle bed air biofilter (TBAB) facilitates more consistent operation than traditional biofilter due to better control of overall pressure drop, nutrient concentration, and pH [2–4], but it is conceptually a similar process.

TBAB has been successfully applied in our previous studies, specifically in treating individual VOC such as toluene [5]; styrene [6]; methyl ethyl ketone [7]; and methyl isobutyl ketone [8]. It was found that the overall performance of TBAB did not only depend on the fed substrate, but also the operational maintenance, including nutrient requirement. These observations are worthwhile to note for understanding the system performance and then design the system. The relationship between the removal of the targeted VOCs and their properties has been widely studied by others. For instance, Deshusses and Johnson [9] demonstrated that the elimination of VOCs in the biofilter can be correlated to Henry's law constant and octanol-water partition coefficient. Zhu et al. [10] also found that the VOC removal increased with a decrease in its Henry's law constant. Chan and Lai [11] discussed that the chemical struc-

ture of compound was the key factor in the microbial growth and biochemical reaction process.

Nitrogen is critical to sustain biomass growth in the biological treatment process [12,13]. When high VOC loading was applied, a typical biofilter might encounter a problem of nitrogen limitation [14]. Hence excess nitrogen level is required to achieve high contaminant elimination capacities in the biofilter; however, excess growth of biomass sometimes gave a negative effect on the biofilter performance, i.e., a decrease in contaminant removal efficiency due to clogging problem in the media [15,16]. For this regards, TBAB is more competitive than the typical biofilter [17].

Since nitrogen supply is one of the critical operational parameter in the application of TBAB for VOCs removal [12], the nitrogen cycle should be understood for proper process design and operation. The nitrogen cycle might not be identical when different substrates are treated in the TBAB. Detailed investigations will provide trends and correlations between the tested VOCs and nitrogen utilization in the TBAB.

This study was conducted to statistically investigate nitrogen utilization and subsequent biomass yield in four TBABs fed with different substrates. Investigations will be based on long-term experimental observations, which have been originally disseminated in previous studies [5–8]. The primary objective of the present work was to develop and validate such discussion, which helps understanding nitrogen utilization in the TBAB when different VOCs substrates are fed.

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Table 1
Nitrogen feeding condition and observation

	Operation condition		Observation		
	VOC load ^a (g/m ³ h)	Nitrate feed ^b (g N/day)	VOC removal (%, mean)	Nitrate utilization (g N/day)	COD/N ^c (g COD/g N)
Toluene	9.3 (0.7)	0.04 (50)	99	0.020 ± 0.003	92.6 ± 1.3
	18.8 (1.41)	0.08 (50)	99	0.043 ± 0.007	89.9 ± 17.6
	46.9 (3.52)	0.2 (50)	98	0.098 ± 0.014	92.9 ± 19.2
	75.1 (5.63)	0.3 (50)	95	0.139 ± 0.043	89.5 ± 33.9
	93.7 (7.03)	0.38 (50)	86	0.143 ± 0.041	118.9 ± 37.9
Styrene	8.7 (0.64)	0.04 (50)	99	0.019 ± 0.008	94.2 ± 23.7
	17.2 (1.27)	0.08 (50)	99	0.045 ± 0.008	75.6 ± 19.5
	25.8 (1.9)	0.2 (25)	99	0.128 ± 0.048	47.9 ± 20.1
	43.0 (3.17)	0.2 (50)	85	0.131 ± 0.026	58.5 ± 13.2
MEK	11.9 (0.7)	0.04 (50)	99	0.034 ± 0.008	58.1 ± 12.3
	24.1 (1.41)	0.09 (50)	99	0.075 ± 0.014	52.6 ± 13.3
	60.1 (3.52)	0.3 (30)	99	0.269 ± 0.231	26.0 ± 6.9
	95.6 (5.6)	1.2 (10)	99	1.168 ± 0.233	12.7 ± 5.4
	120.2 (7.04)	1.5 (10)	86	1.276 ± 0.231	12.3 ± 3.0
MIBK	16.7 (1.09)	0.06 (50)	99	0.043 ± 0.002	67.4 ± 2.5
	33.3 (2.17)	0.11 (50)	99	0.087 ± 0.009	67.7 ± 8.0
	49.9 (3.26)	0.9 (10)	99	0.894 ± 0.163	10.0 ± 2.3
	66.6 (4.34)	1.0 (10)	97	0.986 ± 0.157	11.6 ± 2.0
	83.3 (5.43)	0.6 (25)	89	0.586 ± 0.239	26.1 ± 17.7

 $^a\,$ COD loading rate of VOC (kg COD/m^3 day) is provided in parenthesis.

^b For the nitrogen source, nitrate was supplied in the biofilter. The values in parenthesis indicate the ratio of inlet COD/N (g inlet COD to g inlet nitrate) for a given VOC loading.

^c The ratio of substrate COD removal to nitrate utilization.

2. Materials and methods

2.1. Experimental biofilter system

Experimental work was performed within four independent lab-scale reactors for controlling single contaminants. The selected VOCs were two aromatic compounds, namely, toluene and styrene, and two oxygenated compounds, namely, methyl ethyl ketone (MEK) and methyl isobutyl ketone (MIBK). Liquid VOC was injected via syringe pump (Harvard Apparatus, Model NP 70-2208, Holliston, MA) in the air stream where it vaporized, and entered the biofilter. Each biofilter was constructed of seven cylindrical glass sections (Ace Glass Inc., Vineland, NJ) with an internal diameter 76 mm and a total length of 130 cm. Four gas sampling ports were evenly located axially along the media bed. The reactor was packed with pelletized diatomaceous earth biological support media (Celite[®] 6 mm R-635 Bio-Catalyst Carrier, Celite Corp., Lompoc, CA) to a depth of about 60 cm. The biofilters were operated at a constant temperature of 20°C and in a co-current gas and liquid downward flow mode. The air flow and nutrient flow to the biofilters was initially set up at the rate of 3.6 L/min (corresponding EBRT of 0.76 min) and 1.5 L/day, respectively. The feed buffered nutrient solution was only used in a pass-through-then-discard mode. The nutrient formulation for the biofilter contained the same amount of nutrient-nitrogen and phosphorus ratio for a given VOC loading (a COD-to-nitrogen ratio of 50:1 and a nitrogen-to-phosphorus ratio of 4:1, which were calculated based on nutrient requirement for biological growth). Nitrate (NO₃-N) was used as the sole source of nitrogen. 1 M NaHCO₃ was used as a pH buffer. The feed nutrient was sprayed as fine mist onto the top of the medium bed through a spray nozzle. As a biomass control strategy, periodic backwashing was conducted while the biofilter was off line by first using 18L of the buffered nutrient solution to induce full medium fluidization at about 50% bed expansion for 1 h per time every week, and finally the recycle was shut off and 18L of the buffered nutrient solution was passed through the column as a rinse. More detailed description of experimental setup and method can be found in our previous study [6-8].

2.2. Analytical methods

Gas-phase samples for VOC analysis were taken with gas-tight syringes. These VOC concentrations were measured by using a gas chromatograph (GC) equipped with a flame ionization detector (FID). A GC equipped with a thermal conductivity detector (TCD) was used for determining the CO₂ concentration in the effluent gas phase. Liquid-phase samples were analyzed for nitrate, total carbon (TC), inorganic carbon (IC), and volatile suspended solid (VSS) concentration. Nitrate was determined by using a Shimadzu UV mini 1240 UV–vis spectrophotometer at wavelength of 220 nm. TC and IC were determined by using a Shimadzu TOC 5000 analyzer. According to Standard Methods 2540G [18], the VSS concentrations in the effluent and backwashing solution were determined, their measurements were used to estimate nitrogen usage for biomass yield. It was assumed in the analysis of biomass yield that nitrogen makes up 12.4% of the dry biomass as Zhu et al. [10] proposed.

3. Results and discussion

3.1. Nitrogen utilization

Nitrogen feeding and utilization across the entire period of biofilter operation are summarized in Table 1. The net nitrogen utilization was calculated by subtracting the amounts for the nitrogen species in the effluent from the nitrogen species present in the nutrient feed. For all compounds, larger nitrogen utilization was apparently found when the biofilters had higher VOC loadings. In Table 1, biofilters treating oxygenated compounds (MEK and MIBK) were found to utilize much more nitrogen as compared to aromatic compounds (toluene and styrene). It should be noted that biodegradation of oxygenated compounds in the biofilter was effectively attained when excess nitrate was supplied to sustain the microbial activity [7,8], for which high value of inlet COD/N corresponded.

Fig. 1 shows the relation between nitrogen utilization and nitrogen usage for biomass yield at different substrate loading rates, speculating how much nitrogen was utilized for cell synthesis in this system as a function of substrate load. For this analysis, nitrogen utilization or consumption ($N_{consumption}$) was calculated by



Fig. 1. Ratio of net utilized nitrogen to estimated nitrogen usage for biomass yield with respect to substrate VOC loadings: The box plot provides the value of N_{consumption}/N_{growth} across the range of experimental periods, stretching from the lower hinge (defined as the 5th percentile) to the upper hinge (the 95th percentile). The solid vertical lines represent the critical loadings for VOCs. The critical loading can be defined as the maximum loading that can provide 99% removal.

the measurement of difference in liquid–nitrogen concentration in influent and effluent. Nitrogen usage for biomass yield (N_{growth}) was estimated by the measurement of biomass loss in the effluent and in the backwashing solution as Zhu et al. [10] proposed. Our hypotheses on this estimation include that nitrogen makes up the largest fraction of dry cell mass (about 12.4% for a bacterial cell formula of $C_5H_7O_2N$) [19], and biomass could not be accumulated within the biofilter bed because excess biomass has been periodically removed through biofilter backwashing, which was effectively applied in this study. A theoretical value of $N_{consumption}/N_{growth}$ is then equal to be 1 if only the cell synthesis was involved in the overall biological reaction for this system

It is obvious in Fig. 1 that the behaviors of nitrogen utilization in the system varied with substrate loads as well as substrate type. For hydrophobic compounds (toluene and styrene), the ratio of nitrogen utilization to nitrogen usage for biomass yield was relatively consistent regardless of substrate loading rates, and close to a ratio of 1. However, for hydrophilic compounds (MEK and MIBK) the ratio of nitrogen utilization to nitrogen usage increased with substrate loading rates, and was far beyond a ratio of 1. This indicates that nitrogen was more utilized for other means rather than cell synthesis in the system. For MEK and MIBK, which have dimensionless Henry's constant values of 0.00194 and 0.00062. respectively. Zhu et al. [10] discussed that O₂ limitation in the biofilm could be encountered when biofilters treat VOCs with substrate Henry's constant less than 0.03, causing accumulation of denitrifiers in the biofilm. It was therefore speculated from this study that denitrification would be attributed to larger nitrate utilization.

It is also observed from Fig. 1 that regardless of the critical loading (solid vertical line in Fig. 1), biodegradation of toluene and styrene generated a relatively similar behavior of $N_{consumption}/N_{growth}$. For MEK and MIBK, a rather higher ratio was found even below the critical loading. This observation elucidates that substrate availability affected nitrogen utilization and biomass yield in the system.

3.2. Maximum practical concentration for substrate in biofilm

Based on the maximum equilibrium concentration of O_2 in the biofilm at the air-biofilm interface, the maximum practical concentration for substrate VOCs in the biofilm at the interface can be calculated by the following equation, which was originally proposed by Williamson and McCarty [20].

$$S_{\text{substrate}} > \frac{v_{\text{substrate}} D_{\text{oxygen}} MW_{\text{substrate}}}{v_{\text{oxygen}} D_{\text{substrate}} MW_{\text{oxygen}}} S_{\text{oxygen}}$$
(1)

where $S_{\text{substrate}}$, S_{oxygen} is a concentration of substrate and oxygen at the biofilm surface, respectively. S_{substrate} represents the maximum practical concentration for substrate VOCs in the biofilm without oxygen limitation. $\upsilon_{substrate}$, υ_{oxygen} is a stoichiometric reaction coefficients for substrate and oxygen, respectively if substrate is completely mineralized to CO₂ and H₂O. MW_{substrate}, MWoxygen is a molecular weight of substrate and oxygen, respectively. D_{substrate}, D_{oxygen} is a diffusion coefficient within the biofilm for substrate and oxygen, respectively. The diffusion coefficients can be determined by the Wilke-Change equation [21]. The ratios of Doxygen/Dsubstrate were predicted to be 2.65, 2.63, 8.53, and 4.53 for toluene, styrene, MEK, and MIBK, respectively. Hence, the maximum practical concentrations in the biofilm without oxygen limitation were determined, and converted to gas concentrations of 513, 178, 19, and 2 ppm_v for toluene, styrene, MEK, and MIBK, respectively. The converted-COD loading rates at the given experimental condition were 7.21, 2.25, 0.26, and $0.05 \text{ kgCOD/m}^3 \text{day}$ for toluene, styrene, MEK, and MIBK, respectively. This estimation well corresponded to our previous speculation that VOC with low Henry's constant could encounter oxygen limitation in the biofiltration and then caused denitrifier colonies develop in the biofilm. Biofiltration treating VOC at levels more than the maximum concentration in the biofilm would be more frequently exposed to a problem of denitrifier development, and, therefore, causing an increase of nitrogen requirement for sustaining the biological reaction.

3.3. CO₂ generation and biomass yield

Since the TBAB system is basically aerobic biological process, the analysis of CO_2 production can be used as a good estimate of biological activity. Table 2 summarized CO_2 productions in TBAB systems. CO_2 productions were obtained from theoretical estimation and experimental observation, assuming that primary biological interaction was aerobic VOC oxidation. It is interesting to note in Table 2 that the difference between observed values and theoretical values were obvious, and a relative large difference was found for oxygenated VOCs (MEK and MIBK). This analysis indicated that most of the consumed substrates were used as the primary electron donor

Table 2

 CO_2 production observed in the TBAB systems in comparison to theoretical CO_2 production.

	A Theoretical CO ₂ production ^a (mole CO ₂ /mole VOC _{consumed})	<i>B</i> Observed CO ₂ production ^b (mole CO ₂ /mole VOC _{consumed})	B/A (%
Toluene	7	4.67	66.7
Styrene	8	6.33	79.1
MEK	4	2.03	50.8
MIBK	6	2.65	44.2

^a Aerobic oxidation of VOC studied to CO₂ and water: Toluene, C₇H₈ +9O₂ \rightarrow 7CO₂ +4H₂O; Styrene, C₈H₈ +1OO₂ \rightarrow 8CO₂ +4H₂O; MEK, C₄H₈O + 11/2O₂ \rightarrow 4CO₂ + 4H₂O; MIBK, C₆H₁₂O + 17/2O₂ \rightarrow 6CO₂ + 6H₂O.

^b The slope of the regression line of Fig. 2.

for biomass growth (cell synthesis), and the remaining were oxidized to inorganic (mostly, CO_2 for energy generation) and organic end products, which is obvious in any biological system [19]. On the other hand, CO_2 can be evolved from substrate degradation and endogenous respiration. For this regard, CO_2 production rate can be expressed as a carbon mass balance, which was originally suggested by [22]:

$$R_{\rm CO_2} = R_{\rm substrate} - R_{\rm biomass} + R_{\rm ER} \tag{2}$$

where R_{CO_2} is the CO₂ production rate (mole C/day). $R_{substrate}$ is the substrate consumption rate (mole substrate as C/day). $R_{biomass}$ is the biomass production rate (mole biomass as C/day, $C_5H_7O_2N$ as biomass formula). R_{ER} is the endogenous respiration rate (mole C/day). This mass balance can be rearranged by using biomass yield coefficient (*Y*, mole biomass as C/mole substrate as C/day):

$$R_{\rm CO_2} = (1 - Y)R_{\rm substrate} + R_{\rm ER} \tag{3}$$

Fig. 2 shows the daily CO₂ production rate as a function of the substrates consumed. The plotted data were presented on a carbon-mole basis. The regression line of Fig. 2 represents a term of (1 - Y) in Eq. (3). The y-intercept means the endogenous respiration rate. Therefore, the observed biomass yield coefficients (g biomass/g substrate, dry weight basis) were 0.668, 0.642, 0.737, and 0.939 for toluene, styrene, MEK, and MIBK, respectively, if a molecular formula for biomass C₅H₇O₂N is considered. These

coefficients appeared to be relatively high in TBAB systems with oxygenated VOCs. This could support our previous argument that biodegradation of oxygenated VOCs utilized much larger nitrogen for cell growth as compared to aromatic VOCs. Considering the previous determination in Section 3.2, biomass accumulation over the biofilm was persistent throughout the period of operation of the TBAB. This observation explains the argument that more frequent and longer duration of backwashing was unavoidable in order to attain consistently high removal efficiencies for the two oxygenated VOCs. Since TBABs operation included periodic backwashing as a biomass control strategy, operational problems related to excess biomass retention were not encountered.

It is also worthwhile to note that Fig. 2 has excellent linear correlation between CO_2 production rate and substrate consumption rate. This reveals that the biological activity in a TBAB system has operated at pseudo-steady state conditions throughout the substrate inlet loadings applied. However, this is valid only when substrate loading was not beyond the critical loading.

4. Conclusions

TBAB performance linked with nutrient usage strongly depended on substrate VOC availability. In this study, in order to elucidate the relation between nutrient usage and TBAB performance, nitrogen utilization and biomass yield were investigated by employing statistical analyses based on long-term experimental observations for four different substrate VOCs. Conclusions that can be drawn from this study include the following:

- (1) The ratio of net nitrogen utilization to nitrogen usage for biomass yield (N_{consumption}/N_{growth}) was effective in understanding the overall TBAB performance. When substrate loadings increased, nitrogen utilization tended to enhance energy production and denitrification, rather than biomass yield as cell synthesis. The observation was apparent in the biodegradation of oxygenated VOCs such as MEK and MIBK.
- (2) Maximum practical concentration for substrate in biofilm, which is based on maximum equilibrium concentration of oxygen in the biofilm at the air-biofilm interface, was effectively used to understand both nitrogen utilization and biomass



Fig. 2. CO₂ productions as a function of substrate consumed: CO₂ production rate and substrate consumption rate were calculated on a carbon-mole basis.

yield. This was a useful indicator to rapidly sketch the nitrogen requirement for a biological reaction in TBAB system.

(3) Biomass yield coefficient can be driven by using the regression analysis between CO_2 production rate and substrate consumption rate. This coefficient was effective in understanding the TBAB performance.

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