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Effect of Unexpected Foaming Incident on Nitrogen Removal in a Vertical Subsurface Wetland

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수직지하 흐름형 습지에서 거품발생이 질소제거에 미치는 영향

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Abstract
A lab-scale vertical flow subsurface (VFS) wetland composed of three parallel columns with polypropylene synthetic fiber as main substrate was operated. Piggery stormwater diluted from swine excreta was fed to the wetland on the basis of three different hydraulic regimes or hydraulic retention time (HRT) of 2, 4, and 8 days with daily internal recirculation. Then, monitoring of common water quality parameters was carried out. Unexpectedly, an increase of effluent COD concentration accompanying the appearance of foams was observed during a distinct period in the wetland with HRT 2, 4, and 8 days, successively. Subsequently, a series of experiments was conducted to investigate the origin of the foams. Foams and the increase of COD concentration were found to be induced by the release of organic matter from the synthetic polypropylene fiber which was fed with piggery stormwater. Meanwhile, nitrogen removal was found to be enhanced during a period which overlapped the distinct foaming period signifying that foaming played two important functions in biological nitrogen removal. Foams which form rapidly and then burst easily could hold up and then release oxygen for nitrification. Foams which contain organic surfactants could serve as carbon sources for denitrification as well. Hence, nitrogen removal was enhanced during the foaming stage. After that, COD concentration decreased slowly to a level prior to the foaming stage, and nitrogen removal efficiency declined as well.

Key words : Denitrifcation; Nitrification; Nitrogen Removal; Piggery stormwater; Subsurface wetland; Polypropylene Fiber

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요 약
합성섬유를 주 여재로 충진한 실험실 규모의 수직 지하흐름형 습지를 제작하여 인공축산단지 강우유출수 처리시험을 수행하였다. 3개의 습지에 대하여 수리학적 체류시간은 각각 2, 4, 8일 로 운전하였으며 매일 1회씩 내부순환을 실시하였다. 운전기간 동안에 매일 일반적인 수질인자에 대하여 모니터링을 실시하였으며 갑자기 HRT에 따라 순차적으로 거품이 발생되면서 처리수의 COD가 증가하는 현상이 관찰되었다. 따라서 연구의 방향을 거품의 발생원인을 분석하는데 초점을 맞추게 되었으며 거품의 출처 및 유기물질 농도의 증가는 습지에 충진한 폴리프로필렌 합성섬유가 분해되면서 방출된 것으로 분석되었다. 거품의 발생 및 유기물질의 농도증가는 질소제거의 효율증가와 밀접한 관련이 있었으며 2가지의 중요한 역할을 수행한 것으로 밝혀졌다. 먼저 거품은 습지내부에서 질산화에 필요한 산소를 억류하면서(hold-up) 질산화를 촉진시키며 거품에 내재된 유기물질은 탈질에 필요한 탄소원으로 작용한 것으로 판단된다. 따라서 거품발생시기와 질소제거효율이 증가한 시기가 일치한 것으로 보인다. 이후에 거품문제가 사라지면서 질소제거 수준은 서서히 감소하여 이전의 거품이 없던 시기로 회귀하였다.

핵심용어 : 탈질, 질산화, 질소제거, 축산단지 강우 유출수, 폴리프로필렌 섬유

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1. Introduction

Piggery wastewater, which is commonly composed of urine, residual excrement, forage residue, and rinse water, has induced vital concerns in agricultural areas in many countries (Ho and Ho, 2012; Sánchez et al., 2005). If piggery wastewater is washed away with stormwater runoff from livestock farms and enters natural and constructed water systems including rivers, streams, reservoirs, and cultivated lands, this piggery stormwater will cause serious water quality impairment of the surface and underground water. Piggery stormwater contains high concentrations of nutrients such as nitrogen, phosphorus, and organic matter (Rico, 2012), which induce eutrophication concerns (Kumar et al., 2010), and toxic substances such as nitrate in water which were reported to be able to cause serious diseases (Alvarez et al., 2006).

Piggery stormwater, as one of the important non-point sources of pollutions (Karcı and Balcıoğlu, 2009), has been extensively treated by constructed wetlands over several decades (Montalvo et al., 2005; Harrington and Scholzet, 2010). In vertical subsurface flow (VSF) wetlands (Gálvez et al., 2003) coupled with water distribution, recirculation, and collecting systems, water flows from the surface to the bottom vertically through different layers of filtration substrates to purify polluted water. The treatment capacity of VSF wetlands highly depended on the main substrate employed.

Synthetic polypropylene fiber is widely employed in water treatment because of its good mechanical properties, strong chemical resistance, high thermal stability, high porosity, large specific surface area, and low density (Baker, 2004). It is a recalcitrant substance, but it can be degraded by auto-oxidation (Hamid, 2000), thermal degradation (Bernstein et al., 2007), and supercritical water degradation technology (Aymonier et al., 2001). In addition, it works as a support substrate for biofilm growth. Foaming has been reported to block oxygen transfer when stable foams are produced, especially in a sludge treatment process (Jenkins et al., 2003). On the other hand, foams are usually created by the rapid stirring and aerating to supply sufficient quantities of oxygen to aerobic fermentations (Davis et al., 2001).

Originally, the objective of this study was to evaluate the performance of the main substrate synthetic polypropylene fiber and the vertical subsurface flow wetland in treating piggery stormwater. Unexpectedly, accidental increases of COD concentration accompanying the appearance of foams and enhanced nitrogen removal were found to occur simultaneously, which led to a hypothesis that there should be a cause-and-affect relation between the release of organic matter and the facilitated nitrogen removal. Hence, the effect of the increase of COD concentration (with respect to foaming and release of organic matter) on nitrogen removal was investigated.

2. Materials and Methods

2.1 Wetland Operation

A lab-scale VSF wetland composed of 3 parallel columns filled with polypropylene synthetic fiber was operated from February 8 to December 6 in 2012. The columns were made of black light-proof acryl with a height of 1 m and a diameter of 0.1 m (Fig. 1).

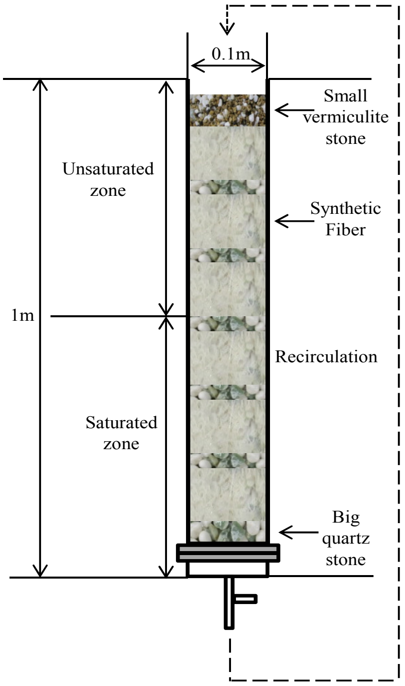

Fig. 1. Schematic diagram of the vertical column wetland

The synthetic polypropylene fiber (Fig. 2 (a)) employed in this study was made into tight donuts (Fig. 2 (b)). Then, they were put into a column wetland layer by layer with big quartz stones as a partition between each layer. Additionally, there was a layer with small vermiculite stones above the fiber layer. The total length of the fiber in each wetland column was 20 m, which supplied the same surface area of 30 m^2 regardless of the number of fiber donuts. Each donut has a similar length. The properties of the fiber are listed in Table 1.

Table 1. General properties of the synthetic fiber

Items	Properties
Diameter	45~150mm
Specific surface area	1.5m^2/m
Porosity	95% - 96%

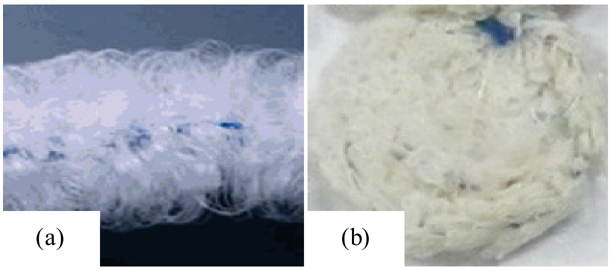

Fig. 2. Fiber Types: (a) fiber type from the manufacturer; (b) fiber donuts

The wetland was fed with the same 2.1 L of diluted piggery stormwater. Sampling was carried out every 2, 4, and 8 days, simulating different hydraulic regimes or hydraulic retention time (HRT) of 2, 4, and 8 days with daily recirculation. The instant hydrologic loading rate was 240 m/day. Common water quality parameters were measured and analyzed. Temperature, turbidity, pH, electric conductivity (EC), and dissolved oxygen (DO) were measured in situ immediately after the water samples were taken out from the effluent. Temperature, pH, and EC were measured by YSI 556MPS Incorporated (www.YSI.com). Turbidity and DO were measured by a HACH 2100H Turbidimeter and YSI 5000 Dissolved Oxygen Meter, respectively.

Other water quality parameters such as total suspended solids (TSS), total nitrogen (TN), total phosphorus (TP), ammonia (NH_4^+-N), nitrate (NO_3^--N), and chemical oxygen demand (COD) were analyzed according to the standard methods for the Examination of Water and Wastewater, 19th Edition (APHA et al., 1995), after they were stored in a refrigerator. Table 2 shows the characteristics of the water quality of the influent and effluent of the wetland. In addition, the bivariate relationship between water parameters was examined using the Pearson correlation coefficient (*r*) by SPSS (version 19.0). 10 months of data were used in the correlation analysis unless otherwise specified.

2.2 Foaming Experiments

The polypropylene fiber was separated into four parts including blue, green, white, and transparent parts, and then was boiled in different types of water for 2 and 4 hours. The influent COD concentration of the piggery stormwater was about 95 mg/L, and the mass of the separated fiber in each flask was 3 g.

The water volume was 200 mL, and the heating temperature was set to 150 °C. The liquid in the flasks could not spill out after evaporation since a reflux condensation system was connected to the heating system. After being boiled, the foaming was compared and the COD concentration of the boiled liquids was analyzed.

Foams are difficult to separate from the liquid completely as they disappear quickly. Repeated separation with a separatory funnel was executed continuously. The liquid was oscillated violently to form foams, which were separated rapidly. Then, the liquid left was separated again in the same way continuously. After most of the foams were separated and turned liquid again, the parameters were analyzed. A microscope with 100 times of magnification was used to search for microorganisms in the samples. The liquid was stained first and then investigated.

Table 2. Characteristics of the water quality of the influent and effluent

Parameters	Unit	Influent	Effluent		
			HRT 2days	HRT 4days	HRT 8days
Temperature	° C	8.0~29.4	14.2~29.5	14.2~29.7	16.5~28.5
pH	-	5.89~7.59	5.86~7.23	5.89~7.30	5.88~7.50
EC	μs/cm	290~434	287~389	301~472	323~642
Alkalinity	mg/L	73~145	57~122	61~127	69~141
Turbidity	mg/L	13.30~39.30	0.08~8.24	0.10~8.28	0.10~7.40
TSS	mg/L	19~77	0~12	0~13	0~15
$TCOD_{Cr}$	mg/L	52~168	11~340	5~484	6~434
$SCOD_{Cr}$	mg/L	10~105	0~288	0~457	3~429
TN	mg/L	10.46~24.94	4.03~20.64	2.97~27.62	2.72~42.81
TKN	mg/L	7.26~23.99	1.53~17.78	1.09~24.94	0.37~39.99
DTN	mg/L	9.20~21.92	3.50~18.14	2.64~17.97	1.97~40.81
NH_4^+-N	mg/L	5.16~14.89	0.06~8.22	0~6.79	0~31.16
NO_3^--N	mg/L	0.06~4.50	0.69~9.62	0.25~12.86	0.16~12.86
TP	mg/L	0.45~2.86	0.10~2.81	0.16~2.25	0.13~2.16
DTP	mg/L	0.32~2.33	0.06~2.58	0~2.14	0.06~2.11

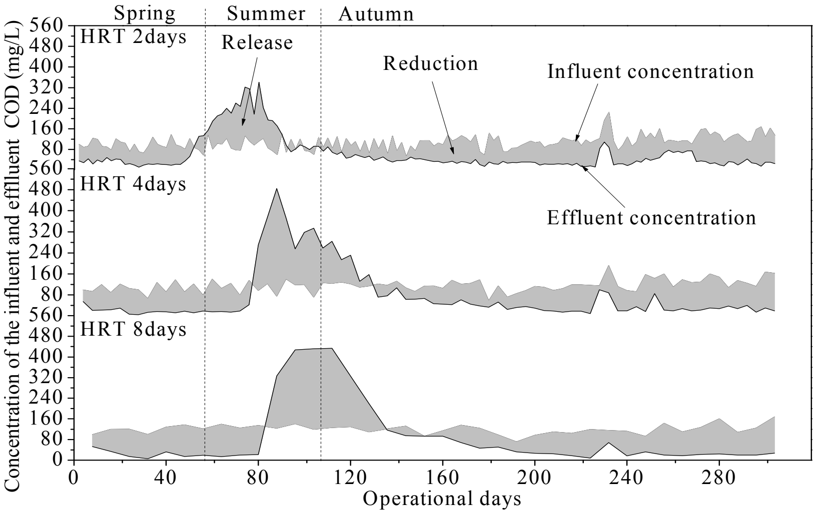

Fig. 3. Variation of the COD concentration with respect to operational days with different HRT: the black line represents the effluent COD concentration and the area filled with grey shadow is indicative of the release or reduction of organic matter

3. Results and Discussion

3.1 Overall organic matters removal

Generally, the effluent COD concentration was extremely low, except that an unexpected increase of COD concentration occurred during a distinct period, regardless of different hydraulic regimes (Fig. 3). Particularly, foams appeared and then disappeared accompanying the increase and decrease of the COD concentration.

3.2 Overall changes of the nitrogen species

As depicted in Fig. 4, the removal efficiency of total nitrogen (TN) increased gradually, remained high during a distinct period, and then decreased finally regardless of HRT. This distinct period overlapped the period when the COD concentration increased accompanying the appearance of foams. Therefore, one thing to be noted is that foaming or the increase of COD concentration must be strongly related to the nitrogen removal.

As illustrated in Fig. 5, the reduction efficiency of NH_4^+-N and TKN gradually increased during the initial acclimatization period. Then, high efficiency was attained and maintained with small fluctuations regardless of HRT. Therefore, it should be noted that the release of organic matter must be associated with the denitrification.

3.3 Changes of the nitrogen species in different phases

Based on the discovery of the distinct period, the operation is divided into three phases. Phase 1, Phase 2, and Phase 3 represent the periods before, during, and after the distinct period, respectively (Fig. 6-9). Pronounced enhancement of the reduction of TKN during Phase 2 than that during Phase 1 was found, whereas a slight decrease was observed during Phase 3 regardless of HRT (Fig. 6). Fig. 7 clearly illustrates that more ammonium was removed during Phase 2 than during Phase 1 under all the three different operation modes. During Phase 3, less ammonium was removed than during Phase 2 with 2 days of HRT, while more or similar amounts were removed with 4 and 8 days of HRT.

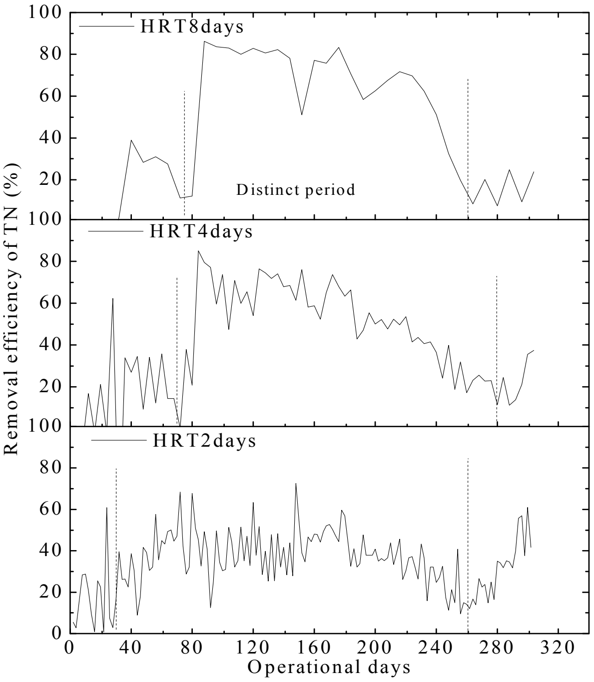

Fig. 4 Removal of TN with respect to operational time

In terms of the accumulation of nitrate, as shown in Fig. 8, similar values during different phases are shown with 2 days of HRT, while a significant decrease during Phase 2 is

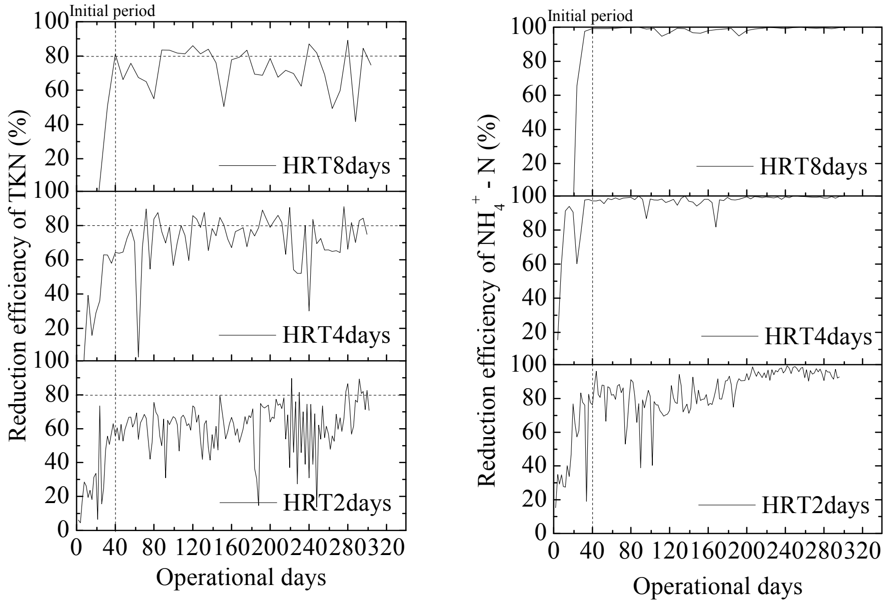

Fig. 5 Reduction of NH_4^+-N and TKN over operational time

illustrated with 4 and 8 days of HRT. Thus, a significant enhancement of denitrification occurred during Phase 2 than during the other phases. Regarding the reduction of TN shown in Fig. 9, significant promotion of denitrification was also observed during Phase 2 than during the other phases.

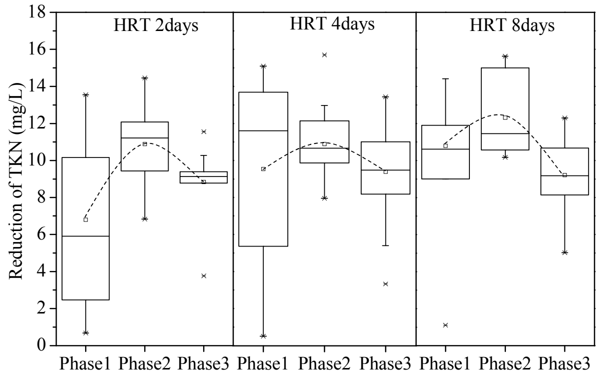

Fig. 6. Reduction of TKN during different phases with different HRT

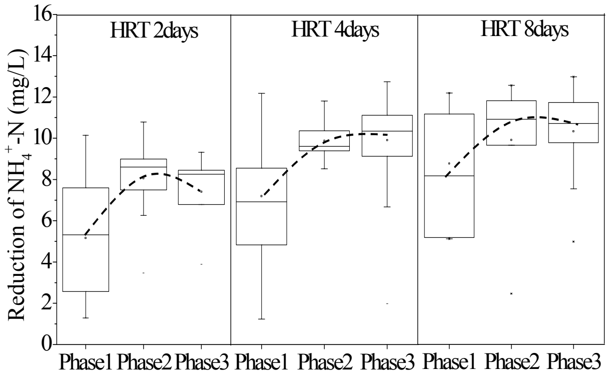

Fig. 7. Reduction of NH_4^+-N at different phases with different HRT

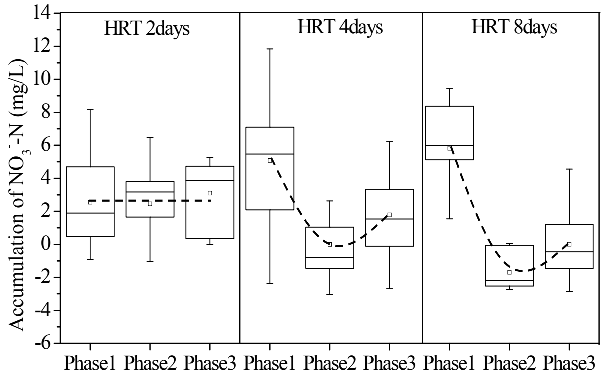

Fig. 8. Accumulation of nitrate during different phases with different HRT

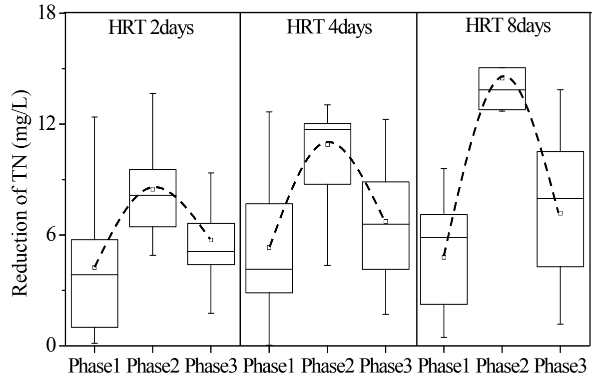

Fig. 9. Reduction of TN during different phases with different HRT

Taking all of these findings together, the removal of organic and ammonia nitrogen was facilitated with all three operational modes during Phase 2, the distinct period. However, the enhanced removal of organic nitrogen could not be maintained and it dropped during Phase 3. Meanwhile, most of the nitrogen removed during Phase 3 was ammonia nitrogen except, with 2 days of HRT.

As calculated, the total biologically transformed nitrogen showed fairly higher values than the total detained nitrogen. This implies that the predominant nitrogen removal process was biological nitrogen removal was achieved principally through nitrification and denitrification.

3.4 Nitrification and denitrification rates during different phases

After an acclimatization period during Phase 1, significant enhancement of the nitrification rate during Phase 2 was observed, and then a continuous high rate was maintained during Phase 3, except for that with 2 days of HRT (Fig. 10). In addition, the nitrification rates decreased with the increase of HRT.

Fig 11 clearly illustrates that after an acclimatization period during Phase 1, significant enhancement during Phase 2 was observed, but then a significant decrease was found during Phase 3. In addition, the denitrification rates decreased with the increase of HRT.

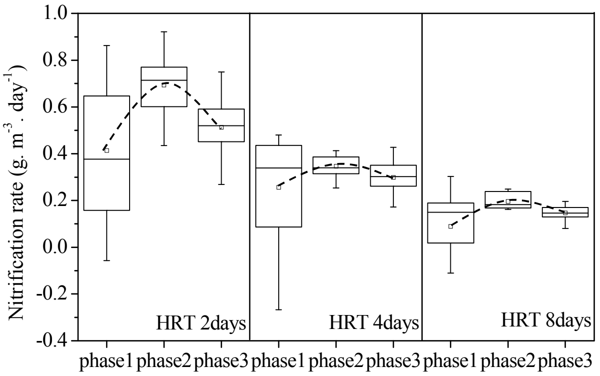

Fig. 10. Nitrification rate during different phases with different HRT

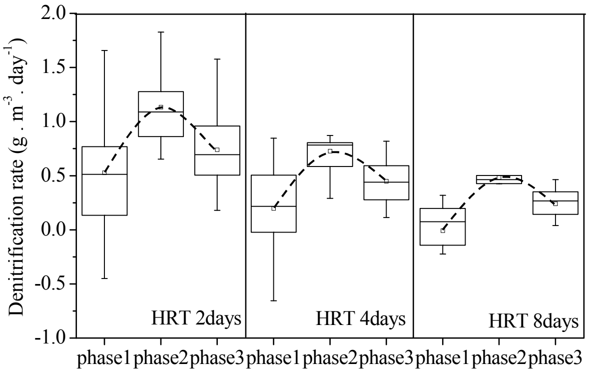

Fig. 11. Denitrification rate during different phases with different HRT

Both nitrification and denitrification rates increased significantly during Phase 2 (the distinct period). This distinct period overlapped the period when the COD concentration increased accompanying the appearance of foams. Therefore, foaming, the increase of COD concentration, or the release of organic matter must be significantly related to the nitrogen removal or transformation.

3.5 Origin of the foams accompanying the increase of COD concentration

Fig. 12 shows the concentration of piggery water boiled with four separated parts of the synthetic fiber for 2 and 4 hours. As illustrated, the COD concentration of the piggery water boiled with the blue part for 2 hours increased significantly (by about two times). However, when boiled with longer time (4 hours), the COD concentration reversely decreased a little. Meanwhile, strong foams were observed in the piggery water boiled with the blue and white parts. Actually, the COD concentration of the piggery stormwater increased as well when the synthetic fiber was put into piggery stormwater without boiling, even within one day. If the foams in the fiber are the same as that in the boiled piggery water, the increase of COD concentration in the wetland could also be due to the release of organic matter from the polypropylene fiber fed with piggery water, and then they formed foams.

Fig. 13 shows the COD concentration of the separated foams from the samples and the boiled piggery water with different separation times. As illustrated, both of the samples displayed high COD concentration, suggesting that they were the same foams. Indeed, the existence of organic surfactant is essential for foams to be produced. The difference of COD concentration between them only indicates the intensity difference. Thus, stronger foams were produced in the wetland. This was due to the larger amount of fiber in the wetland. In conclusion, the increase of COD concentration and the appearance of foams

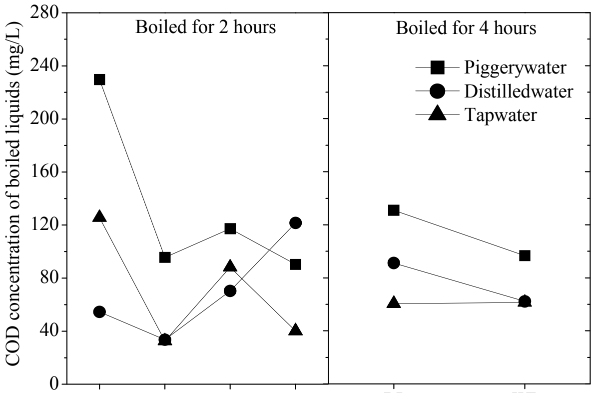

Fig. 12. COD concentrations on the different parts of fiber

Table 3. Pearson correlation coefficients between denitrification/nitrification and TCOD/SCOD

Parameter	HRT(day)	Nitrification rate			Denitrification rate		
		2	4	8	2	4	8
TCOD	r	0.343	0.183	0.214	0.408	0.476	0.494
	p	0.000	0.115	0.196	0.000	0.000	0.002
SCOD	r	0.359	0.202	0.201	0.426	0.509	0.483
	p	0.000	0.080	0.225	0.000	0.000	0.002

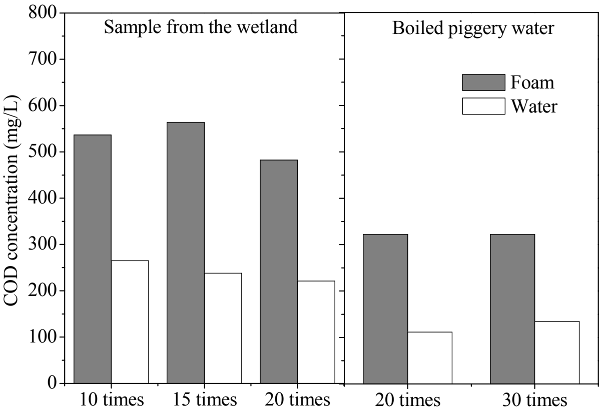

Fig. 13. Comparison of the COD concentration of foams separated from the sample and the boiled piggery water

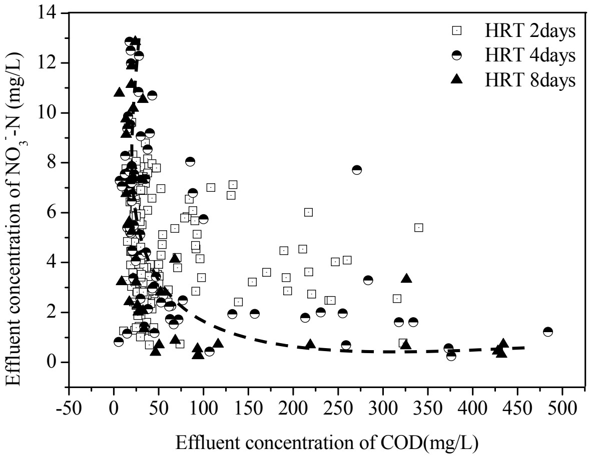

Fig. 14. Effluent concentration of NO_3—N varying with effluent concentration of COD with different HRT

in the effluent were due to the release of organic matters from the polypropylene synthetic fiber fed with piggery stormwater in the wetland.

3.6 Effect of foaming incident on the changes of nitrogen species

In order to investigate the effect of foaming on the changes of nitrogen species, a statistical test (*Pearson correlation analysis*) was conducted, and the results are shown in Table 3. It clearly shows that the COD concentration exerted a significant positive correlation to the nitrification rate with HRT of 2 days, whereas no obvious relation was found with HRTs of 4 and 8 days. This is due to the more frequent recirculation with HRTs of 4 and 8 days, which have already promoted nitrogen removal to a high level. Thus, recirculation enhanced nitrification more than foaming. Differently, the COD concentration was found to show a significantly positive correlation to the denitrification rate, regardless of HRT. The existence of strong, persistent, but rupturable foams during Phase 2 indicates more air hold-up inside the column. The air trapped inside the surface of fibers could then be served for nitrification. Thus, foaming promoted oxygen transfer, which could be the reason for the enhancement of nitrification during Phase 2.

On the other hand, the released organic matter served as a carbon source, which then facilitated denitrification and thereby removed more nitrogen. This can be verified by Fig. 14, which depicts that higher effluent concentration of COD and the release of organic matter corresponded to a lower effluent concentration of nitrate. In addition, a statistical test (*Pearson correlation analysis*) was also carried out between the other parameters and nitrification/denitrification rates. Only temperature and pH were found to show positive relations to nitrification/denitrification rates (Table 4). It should be noted that pH and temperature during the distinct period were not highly variable, so they were not key influencing factors. Therefore, only the release of COD is concluded to be a primary

Table 4. Pearson correlation coefficients between denitrification/nitrification rate and pH/temperature

Parameter	HRT(day)	Nitrification rate			Denitrification rate		
		2	4	8	2	4	8
pH	r	0.356	0.319	0.639	0.333	0.193	0.564
	p	0.000	0.005	0.000	0.000	0.094	0.000
Temperature	r	0.178	0.216	0.338	0.286	0.429	0.492
	p	0.029	0.061	0.038	0.000	0.000	0.002

factor, which functioned to enhance the nitrogen removal during the distinct period.

3.7 Effect of foams on oxygen transfer

Materials having porous structure with a hydrophobic property can easily form foams with an organic surfactant (Kornev et al., 1999). The hydrophobic polypropylene fiber donuts with highly porous structure could work as a sparger (Tschentscher et al., 2011; Veera et al., 2001), and piggery stormwater with high COD concentration (organic solvent) could function as a foaming agent. Then, foams were produced continuously during the distinct period (phase 2). With the presence of foams, gas hold-up and oxygen transfer were enhanced.

Fig. 15 depicts the gas hold-up inside the VSF fiber column wetland in this study. Fig. 15 (a) and (b) show the feeding and discharge processes, respectively. Oxygen renewal was obtained by intermittent feeding, internal recirculation, and drainage. Fig. 15 (c) and (d) depict the difference between non-foaming and foaming water as inflow in terms of air hold-up. The increased water level indicates the volume of the air hold-up inside the column, with which more oxygen could be utilized for a longer time.

As illustrated in Fig. 16, foams with surfactants construct a resistance interface, which may hinder oxygen transfer. However, on the other hand, bubbles tend to become smaller as surfactants decrease surface tension. Thus, the oxygen transfer coefficient increases due to the increase of the specific gas-liquid interfacial area. In addition, the oxygen solubility in the surfactant is larger than that in water, which may promote the oxygen transfer through the interface (Asgharpour et al., 2010). Furthermore, as the oxygen is continuously consumed by nitrification, the greater requirement for oxygen also helps to increase the oxygen transfer rate to some extent. Foams in this study are easy to burst in a short time, with which they release oxygen again after holding up oxygen for some time.

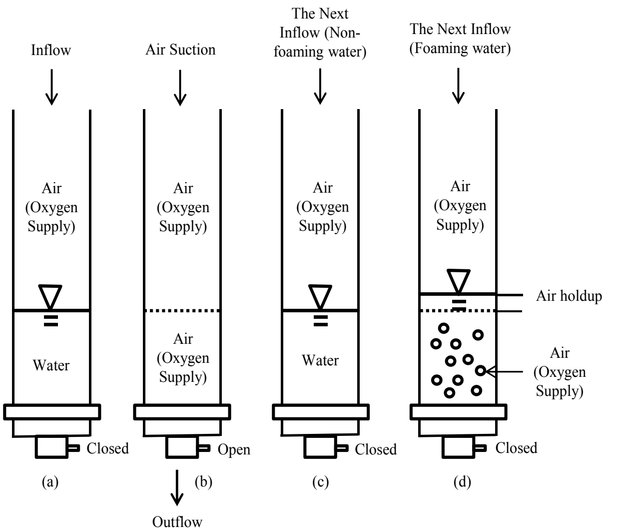

Fig. 15. Air hold-up with foaming water in the fiber column wetland

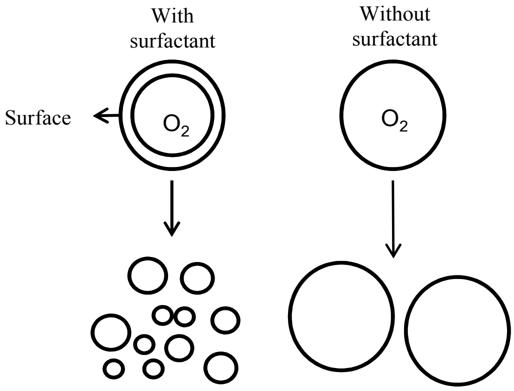

Fig. 16. Comparison of oxygen transfer between foams and bubbles

Therefore, the appearance of foams tended to hold up more air inside the column and promoted the oxygen transfer overall. In this study, the observation of high life forms such as nematodes and other protozoa validated that the high oxygen level was virtually sustained inside the wetland column.

4. Conclusions

A lab-scale vertical subsurface flow (VSF) wetland with polypropylene fiber as a main substrate was fed with diluted piggery stormwater with HRT 2, 4, and 8 days and coupled with daily recirculation. Common water quality monitoring was carried out. Unexpectedly, COD concentration increased gradually and then decreased again during a distinct period. Meanwhile, foams were observed to appear and then disappear accompanying the increase and decrease of COD concentration. Hence, additional experiments were conducted to investigate these accidental incidents. Finally, a special effect on nitrogen removal was found.

With piggery stormwater fed continuously, degradation of some parts of the polypropylene fiber was started. Then, the degraded organic matter increased COD concentration and produced foams at the same time. Foams produced during the degradation of polypropylene displayed high COD concentration, and are able to capture oxygen and then release oxygen when bursting. With the presence of foams and high oxygen level achieved, nitrification was promoted. Meanwhile, the released organic matter served as carbon sources and then enhanced denitrification significantly. After the distinct period, COD concentration decreased again, and foams disappeared as well, indicating the end of the possible release of the degradation products of polypropylene fiber. Meanwhile, nitrification and denitrification rates returned to the previous level.

Different HRTs exerted different effects on nitrogen removal. In terms of removal efficiency, HRTs of 4 and 8 days showed better removal than an HRT of 2 days. Temperature and pH are also important factors affecting nitrification and denitrification, so relatively higher temperature and pH around 7 are favorable for nitrogen removal.

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