Removal of Organic Matter and Nutrient in Swine Wastewater Using a Membrane System

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Abstract − Swine wastewater was treated using a unique sequence of ion exchange membrane bed system (IEBR). Organic matter and nutrient in swine wastewater was pre-treated by electron beam irradiation. The optimal dose for solubilization of organic matter in swine wastewater ranged from 20 kGy to 75 kGy. The carbohydrates, proteins, and lipids were investigated as the solubilized organic fraction of swine wastewater and proteins and lipids mainly contained of the solubilized organic matter. The solubilization of organic matter in swine wastewater was affected by the combination effect of temperature and a dose. The average chemical oxygen demand (COD) removal efficiency under room temperature conditions was 67.1%, while that under psychrophilic conditions was 54.6%. For removal of ammonia, the removal efficiency decreased from 63.6% at 23˚C to 33.5% 16.8˚C. On the other hand, the removal of phosphorus was not a function of temperature. Struvite was one of main mechanisms in anaerobic condition.

Key words : Electron beam, Irradiation, Swine wastewater, Organic matter, Nitrogen, Phosphorus

INTRODUCTION

Swine production has increased on robust global demand with accordance to the intensive growth of husbandry industry. Specifically, swine production has been obvious in Asia. For instance, China consumes one half of pigs generated in the world. South Asia became one of the biggest exporters because of continuous demand of red meats in the developed countries. According to the Food and Agricultural Organization, swine produced in Korea was 9,880,630 head in 2010 (FAO 2010). Considering population in Korea was approx. 49,000,000 in 2010, swine wastewater has been highlighted as one of high-strength organic wastewaters in Korea (Statics Korea 2010). Organic matter and nutrient in swine wastewater are required to be treated before released into the water systems because of the ammonia toxicity and deterioration of environmental quality caused by high organic loading (Park *et al.* 2011). Generally, swine manure requires long hydraulic retention time (HRT) to stabilize organic matter in a continuously stirred tank reactor due to the high-strength of organic loading and solids (Chynoweth *et al.* 1999). Andreadakis (1992) stated that approximately 40% of total organic matter is non-biodegradable in swine waste. However, several investigators showed that swine wastewater can be successfully treated using high-rate anaerobic processes such as an anaerobic filter, an upflow anaerobic sludge blanket, an anaerobic baffled reactor, and a static granule bed reactor (Yang and Chou 1985; Lo *et al.* 1994; Lim and Fox 2011a, b). Nitrogen in swine wastewater commonly occurs in ammonium because the ionic fraction of ammonia is predominant at pH 7. Although ammonium is less toxic than free ammonia in aqueous phase, it must be removed to prevent the inhibition of human health or the ecosystems. In addition, the effluent phosphorus contained in swine wastewater should be stringently res-

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trained because it mainly causes eutrophication in fresh/coastal waters (Luo *et al.* 2002). The actual nutrient in swine wastewater considerably differs from a particular operation of manure collection, housing, and humidity. For instance, there is a considerable loss of nitrogen to air, a potential run-off, or leaching to ground water when manure is exposed to an open lot (Zhang and Felmann 1997).

Several investigators stated that biological processes can be feasible to treat swine waste because of low cost (Lim *et al.* in press; Lucas *et al.* 2010). On the other hand, advanced oxidation processes (AOPs) using radicals, ozone, and irradiation can be alternative for mineralize refractory solving nortorious problems. Ye *et al.* (2009) reported the deodorinization of swine wastewater using horseradish peroxidase and peroxides. Getoff (2002) stated that "The radiation chemistry helps to solve environmental problems very efficiently, especially in the degradation of water pollutants". Several investigators reported that the solubility and biodegradibility of organic matter were enhanced after the electron beam irradiation. Park *et al.* (2009) showed the enhancement of gas production and biodegradability of sewage sludge using electron beam irradiation. In this study, temperature decreased from room temperature conditions to psychrophilic temperature conditions during the study period. Swine wastewater was biologically treated after the pre-treated by electron beam irradiation. The combined effects of temperature and dose on organic matter and nutrient removal were investigated using an ion exchange biological reactor (IEBR), which was developed by Park *et al.* (2011).

MATERIALS AND METHODS

1. Configuration of the IEBR

A schematic diagram of the IEBR used in this study is shown in Fig. 1. This system consisted of three chambers, which separated by a cation exchange membrane (CEM; ASTOM Co., Tokyo, Japan) and an anion exchange membrane (AEM; ASTOM Co., Tokyo, Japan). The active volume of each chamber was 2 l. The dimension of each membrane installed in this system was 100 mm × 16.80 mm. A CEM and an AEM used in this study was specified to design exchanging monovalent cations or monovalent anions. The influent flowed into chamber A followed by chamber C, whereas chamber B was hydraulically closed. The degree of ammonium transportation was determined by the ammonium flux via a CEM between chamber A and chamber B. The transportation of nitrate was performed caused by the concentration gradient via an AEM between chamber B and chamber C (Park *et al.* 2009, 2011). While both chamber A and C were stirred using magnetic bars, chamber B was continuously aerated with air. The air flow rate was 5 l min^{-1}. Each chamber was considered as a continuous stirred tank reactor due to the completely mixing conditions. Monovalent

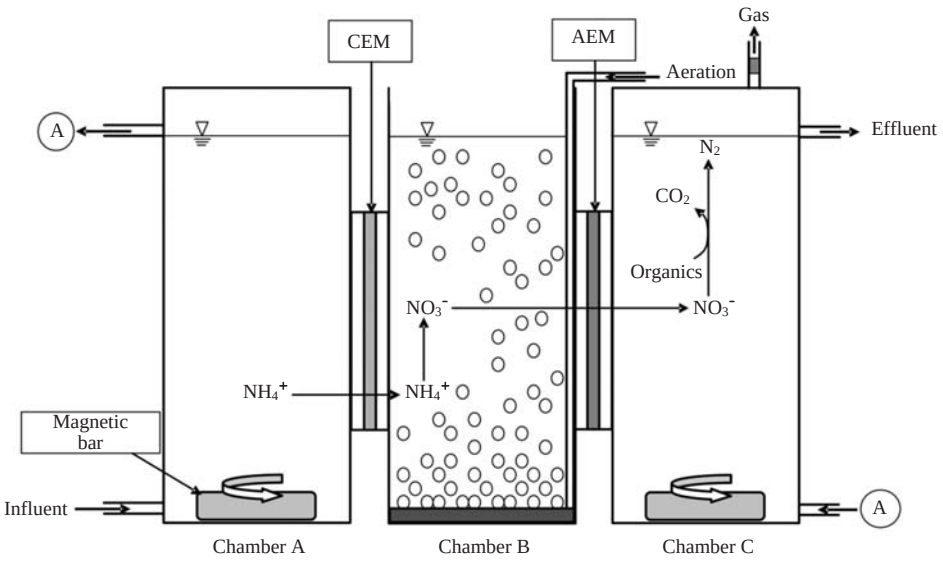

Fig. 1. A schematic diagram of the IEBR.

cations including ammonium in swine wastewater were ion-exchanged between chamber A and chamber B via a CEM. The ion-exchanged ammonium was nitrified to nitrate. The inoculated activated sludge for chamber B was taken from a livestock WWTP located at Gongju, Korea. Activated sludge was washed three times with purified water. The washed activated sludge with 50 mM phosphate buffer solution was inoculated to chamber B. The concentration of inoculated activated sludge at chamber B was 3240 ± 20 mg l^{-1}. The pH of chamber B was adjusted to 7.0 to 7.5 using 1.0 N NaOH or 1.0 N H_2SO_4. Monovalent anions such as nitrite and nitrate in chamber B were ion-exchanged between chambers B and C via an AEM due to the concentration gradient. $NaHCO_3$ were added to study nitrification (2.0 g l^{-1}). Also, the concentration of dissolved oxygen (DO) was monitored. Organic matter (electron donor) in swine wastewater flowed into chamber C and was used for denitrification.

2. Characteristics of swine wastewater

The concentration of swine wastewater is commonly affected by several parameters such as pig age and the diet of hogs, temperature, humidity of a building, housing or confinement, waste removal, and pre-processing (Andreadakis 1992; USDA 1992; Zhang and Felmann 1997; Day and Funk 1998). Among parameters, dilution, storage, and separation play important roles in determining the characteristics of swine wastewater (Chynoweth *et al.* 1999). Swine wastewater is solid waste which has some liquid, while municipal or industrial wastewater is usually liquid waste which has some solids (Andreadakis 1992). Total solids (TS) of swine excrete is approximately 10% and it is usually diluted with urine. It is necessary that, in order to obtain a representative sample, swine wastewater be sampled at a livestock WWTP. The swine wastewater used in this study was collected from a livestock WWTP located at Gongju, Korea. The collected sample was stored at 4°C until use. The characteristics of swine wastewater used in this study are shown in Table 1. The COD/N ratio of swine wastewater was 6.54. US EPA (1993) recommended that greater than 6 of COD/N ratio enhances the denitrification efficiency.

Table 1. Characteristics of swine wastewater used in this study

Parameter	Value (Ave. ± Std.)[a]
pH	8.6 ~ 9.1 (8.7 ± 0.1)
ORP (mV)	−402.0 ~ −20.9 (−162.8 ± 184.7)
DO (mg l^{-1})	0.6 ~ 1.6 (0.4 ± 0.6)
Alkalinity (mg $CaCO_3$ l^{-1})	7603.6 ~ 9200.0 (8410.7 ± 706.2)
TCOD (mg l^{-1})	7893.0 ~ 10074.0 (8898.9 ± 766.5)
SCOD (mg l^{-1})	7293.0 ~ 9553.0 (8151.1 ± 690.9)
Carbohydrates (mg l^{-1})	346.2 ~ 541.2 (464.3 ± 77.7)
Proteins (mg l^{-1})	1576.3 ~ 4134.2 (3520.0 ± 1091.8)
Lipids (mg l^{-1})	1100.0 ~ 3630.0 (2370.0 ± 959.6)
TS (mg l^{-1})	11860.0 ~ 9440.0 (11024.0 ± 754.2)
VS (mg l^{-1})	7580.0 ~ 5980.0 (6467.1 ± 494.2)
T-N (mg l^{-1})	2582.0 ~ 2005.0 (2302.5 ± 177.5)
NH_3-N (mg l^{-1})	2399.0 ~ 1565.0 (216.86.0 ± 268.3)
NO_2-N (mg l^{-1})	0.0 ~ 31.2 (16.7 ± 16.8.9)
NO_3-N (mg l^{-1})	0.0 ~ 3.5 (1.1 ± 1.6)
T-P (mg l^{-1})	200.0 ~ 415.0 (245.9 ± 58.2)
PO_4-P (mg l^{-1})	0.0 ~ 33.9 (9.0 ± 16.6)

[a]: minimum-maximum (average ± standard deviation)

3. Operating condition of the IEBR

The IEBR was operated for 110 days at a room temperature. The nominal HRT in the IEBR was 10 ± 0.03 days. In order to compare the solubilization of influent, the swine wastewater was irradiated by electron beam. The doses used in this study were 20, 50, 75, and 100 kGy, respectively. Electron beam radiation was carried out by an electron accelerator (1 MeV, 40 kW; ELV-4, EB-Tech Co., Korea). The absorbed dose was measured with dichromate solution according to the method described by Han *et al.* (2002). The operating condition in the IEBR is shown in Table 2. Since chamber B was hydraulically closed the inoculation of activated sludge was necessity. The average concentration of mixed liquor suspended sludge (MLSS) at chamber B during the study period was 2950 ± 20 mg l^{-1}. Sodium bicarbonate was periodically added at chamber B to prevent the inhibition of nitrification. Organic matter and the ion-exchanged nitrate were simultaneously removed at anoxic chamber C. As shown in Table 2, the organic loading rate (OLR), nitrogen loading rate (NLR), and phosphorus loading rate (PLR) at chamber A were approximately 2.1 kg TCOD m^{-3} day^{-1} and 0.5 kg T-N m^{-3} day^{-1}, and 0.1 kg T-P m^{-3} day^{-1} irrespectively.

4. Analytical methods

The pH, ORP, DO, temperature, and flow rate were measured daily. The pH and ORP were measured using a pH/ORP meter (Orion 3 Star, Thermo Fisher Scientific Inc., Beverly, MA, USA) equipped with a pH probe (Orion 8157 BNUMD pH/ATC Triodes, Thermo Fisher Scientific Inc., Beverly,

Table 2. Operating condition of the IEBR during the study period

Parameter	Chamber		
	A	B	C
Volume (L)	2	2	2
HRT (day)	5	n.a.	5
Operational mode	Continuous	Hydraulically closed	Continuous
OLR (kg TCOD m^{-3} day^{-1})[a]	2.1±0.4	n.a.	n.a.
OLR (kg SCOD m^{-3} day^{-1})[a]	1.9±0.4	n.a.	n.a.
NLR (kg T-N m^{-3} day^{-1})[a]	0.5±0.1	n.a.	n.a.
NLR (kg NH_3-N m^{-3} day^{-1})[a]	0.5±0.1	n.a.	n.a.
PLR (kg T-P m^{-3} day^{-1})[a]	0.1±0.0	n.a.	n.a.
PLR (kg PO_4-P m^{-3} day^{-1})[a]	0.0±0.0	n.a.	n.a.
pH[b]	8.7±0.1	9.9±0.3	9.0±0.1

[a]: loading rate at chamber A, [b]: results from monitoring at each chamber during the study, n.a.: not available

MA, USA) or an ORP probe (Orion 9179 BNMD, Thermo Fisher Scientific Inc., Beverly, MA, USA). The DO concentration was measured using a pH/DO meter (Orion 4 Star, Thermo Fisher Scientific Inc., Beverly, MA, USA) equipped with a DO probe (Orion 081010MD, Thermo Fisher Scientific Inc., Beverly, MA, USA). Samples for COD, solids, nitrogen, and phosphorus were taken biweekly. After the quasi steady-state was reached, each sample was obtained. All analyses were done according to Standard Methods for the Examination of Water and Wastewater (APHA 1998). Carbohydrates were measured by Bubois method (Bubois *et al.* 1956), proteins by the Layne method (1957), and lipids by the Bligh method (Bligh and Dyer 1959). Ions were measured with an ion chromatography (ICS-2000, Dionex Co., Sunnyvale, USA) equipped with a cation exchange column (IonPac CS18 Cation-Exchange Column, Dionex Co., Sunnyvale, USA) and an anion exchange column (IonPac AS18 Anion-Exchange Column, Dionex Co., Sunnyvale, USA). Crystals formed at chamber A or chamber C were characterized by scanning electron microscopy (SEM; JSM-6390, JEOL, Japan).

RESULTS AND DISCUSSION

1. Removal of organic matter

The variation of TCOD removal at different doses is shown in Fig. 2. The average TCOD removal efficiencies at a dose of 0 kGy, 20 kGy, 50 kGy, 75 kGy, and 100 kGy were 67.1%, 61.0%, 58.7%, 59.3%, and 40.9%, respectively. It seems that removal of organic matter was not a function of electron beam pre-treatment. However, at a dose of 0 kGy, the TCOD removal efficiency at 16.8.0±2.8°C was comparable to that at 23.0±2.3°C. The average TCOD removal efficiency at a dose of 0 kGy under room temperature conditions (23.0°C) was 67.1%, whereas that under psychrophilic conditions (16.3°C) was 54.1%. With increasing dose, the influent TCOD concentration increased. On the other hand, the effluent concentration (TCOD concentration at chamber C) did not significantly fluctuated because the effluent TCOD concentration was highly dependent upon the MLSS concentration at chamber C. Such the SCOD concentration was (data not shown). This implies that swine wastewater was oxidized by several radicals and partly transformed to biodegradable organic matter (Auslender *et al.* 2002; Getoff 2002; Kim *et al.* 2007). Wagner *et al.* (2009) reported that 19～72% of TCOD removal efficiency in swine wastewater to produce hydrogen using a microbial electrolysis cell. Park *et al.* (2011)

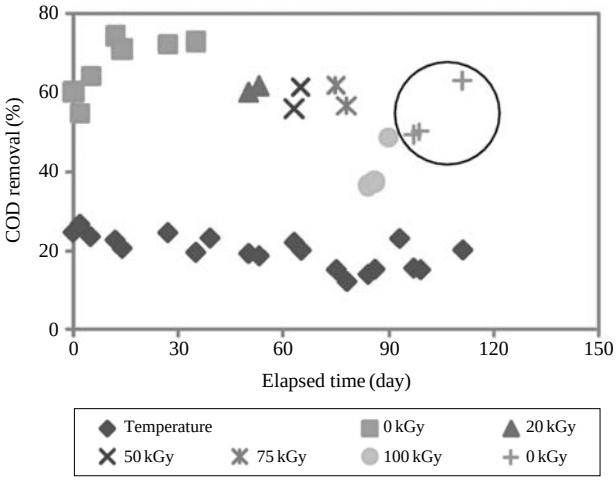

Fig. 2. Variation of COD removal at different doses (sqare: 0 kGy at 23.0±2.3°C, cross: 0 kGy at 16.8.0±2.8°C).

showed that 99.5% of SCOD was removed from acetate using the IEBR. Acetate is a well-known substrate that one of the most easily biodegradable organic matter (carbon source). However, swine wastewater generally requires long HRT to be sufficiently treated since swine manure consists of recalcitrant organic matter such as cellulose or lignin (Shin *et al.* 2005; Lim and Fox 2011a, b). Some of transformed organic matter were solubilized and supplied for electron donors. The degree of solubilization of carbohydrates, proteins, and lipids in swine wastewater is shown in Fig. 3. The solubilization of organic matter in the influent/at each chamber can be defined as the following equation.

$$\text{Solubilization of a substance (mg l}^{-1}) = \text{concentration}_{\text{-before}} - \text{concentration}_{\text{_after}} \quad (1)$$

where, $\text{concentration}_{\text{-before}}$: influent concentration of a substance before electron beam irradiation or influent concentration at each chamber (mg l^{-1})

$\text{concentration}_{\text{_after}}$: effluent concentration of a substance after electron beam irradiation or effluent concentration at each chamber (mg l^{-1})

As shown in Fig. 3, most organic matter was solubilized by electron beam irradiation. Subsequently, the biodegradable organic matter such as short chain fatty acids was used as electron donor for denitrification. As shown in Fig. 3, while the amount of solubilized carbohydrates in the influent was small, those of proteins and lipids were high. This is because the initial carbohydrates concentration was relatively low and because carbohydrates in swine wastewater usually contains of refractory organic matter (Andreadakis 1992). The optimal dose for solubilization of organic matter in swine wastewater (influent) ranged from 20 kGy to 75 kGy.

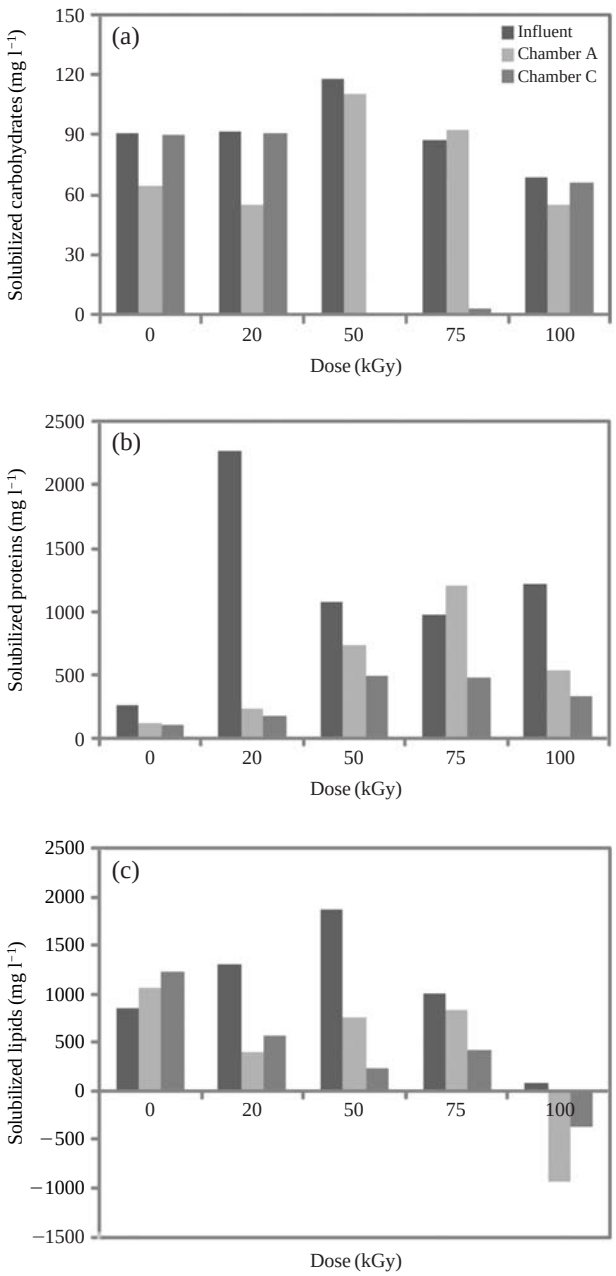

Fig. 3. Solubilization of organic matter in swine wastewater different doses ((a) carbohydrates, (b) proteins, (c) lipids).

2. Characteristics of nutrient removal in the IEBR

The average ammonia removal efficiency during the study period is shown in Fig. 4. Removal of ammonia was significantly affected by temperature and solubilized organic matter. Ammonium in swine wastewater is usually removed via

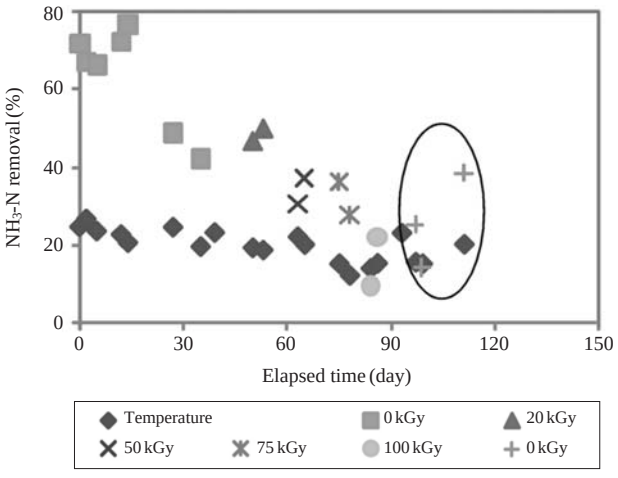

Fig. 4. Variation of ammonia removal at different doses (sqare: 0 kGy at 23.0±2.3°C, cross: 0 kGy at 16.8.0±2.8°C).

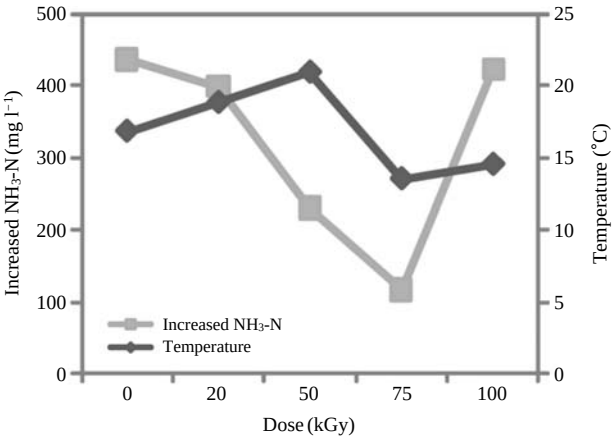

Fig. 5. Effects of temperature and dose on increased ammonia in the influent.

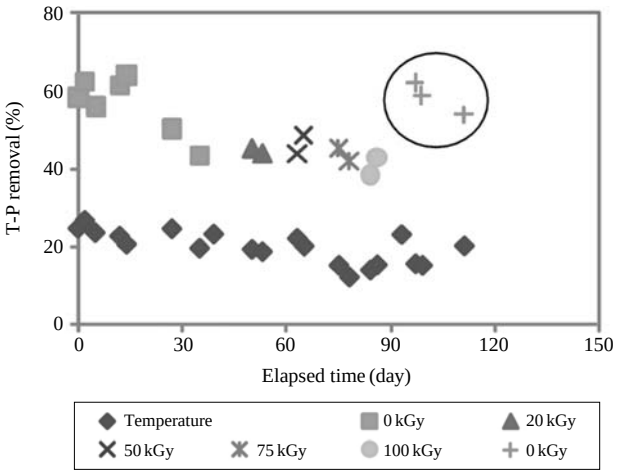

Fig. 6. Variation of T-P removal at different doses (sqare: 0 kGy at 23.0±2.3°C, cross: 0 kGy at 16.8.0±2.8°C).

biochemical oxidation/reduction processes. In these processes, many autotrophic/heterotrophic bacteria are involved. Specifically, since the activity of autotrophic bacteria enzymes is highly dependent upon temperature, the effect of temperature on ammonia removal in the IEBR was greater than that of bonding rupture by a dose (Madigan *et al.* 2006). Fig. 5 shows that the effects of temperature on the increment of ammonia in the influent. As shown in Fig. 5, the increase of ammonia concentration in the influent was a fuction of temperature, rather than a dose. This implies that the solubilization of proteins in swine wastewater was affected by the combination effect of temperature and a dose. The concentration of increased ammonium in the influent was high at a dose of 20 kGy and 100 kGy. This is the same result from solubilized proteins (Fig. 3b). On the other hand, unlike TCOD removal, comparing the ammonia removal efficiency at a dose of 0 kGy under room temperature conditions (23°C) to psychrophilic conditions (16.8°C), the ammonia removal efficiency at psychrophilc conditions was not recovered to that at 23°C. The average ammonia removal efficiency under room temperature conditions was 63.6%, while that under psychrophilic conditions was 26.0%. It is because the activity of autotrophs is more sensitive than that of heterotrophs (US EPA 1993). Swine manure mainly contains proteins and lipids. As shown in Table 1, the average concentrations of carbohydrates, proteins and lipids were 464.3 mg l^{-1}, 3520.0 mg l^{-1} and 2370.0 mg l^{-1}, respectively. Ammonium at chamber A was ion-exchanged via a CEM to chamber B and oxidized to nitrate. Subsequently, considering the pKa of ammonia at 25°C is 9.23 and the amount of ammonium for synthesizing

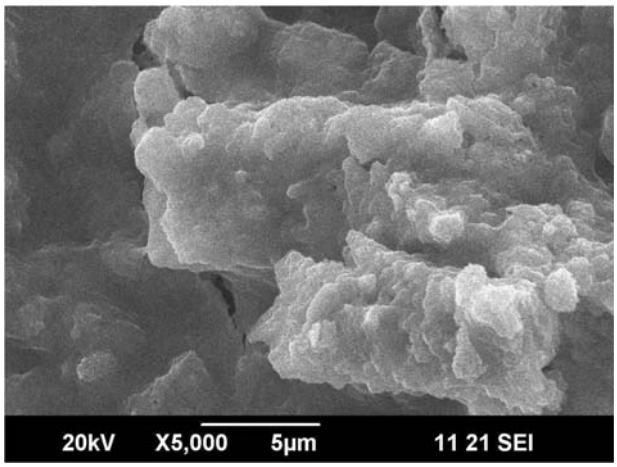

Fig. 7. Scanning electron micrograph of struvite crystals precipitated at a dose of 0 kGy at chamber C under psychrophilic conditions.

nitrifying bacteria is small, most oxidized nitrate was ion-exchanged via an AEM to chamber C due to the concentration gradient. In other words, removal of organic matter in swine wastewater in the IEBR is highly dependent upon nitrification at chamber B and denitrification chamber C. Park *et al.* (2009, 2011) showed that the rate-limiting step in the IEBR is the ammonium flux via a CEM. According to their research, the ammonium flux was 0.83 mg m^{-2} sec^{-1}. As shown in Figs. 3 and 4, the relationship between ammonia removal efficiency and solubilized organic matter in this investigation is significant at a dose rage of 20 kGy to 100 kGy. Solubilized proteins and lipids were efficiently used as electron donors, not carbohydrates. It is a similar result from

Lim *et al.* (2009). This evidently supports that the optimal dose exists for oxidizing swine wastewater. The T-P removal efficiency during the study period is shown in Fig. 6. The T-P removal efficiency under room temperature conditions was 38.4～56.5 (45.1±5.1%). Unlike the ammonia removal efficiency, the T-P removal at psychrophilic conditions (16.8 ˚C) was 58.2%. Nonetheless, the concentration of phosphorus was not increased significantly (data not shown). Hanhoun *et al.* (2011) presented that pK_{sp} of struvite ranged 13.29 at 15˚C to 13.00 at 30˚C. This implies that removal of phosphorus in the IEBR was not significantly affected by temperature. A CEM/an AEM and sludege had contained white crystals, indicating the struvite formed during the study period. Struvite is one of phosphate removal mechanisms in anaerobic/anoxic reactor. Fig. 7 shows the SEM of struvite crystals precipitated at a dose of 0 kGy at chamber C under psychrophilic conditions.

CONCLUSION

The results of this research have yielded the following conclusions.

- The optimal dose for solubilization of organic matter in swine wastewater ranged from 20 kGy to 75 kGy.
- The solubilization of organic matter in swine wastewater was affected by the combination effect of temperature and a dose.
- Removal of nitrogen removal was significantly affected by temperature. On the other hand, the removal efficiencies of organic matter and phosphorus at a dose of 0 kGy under psychrophilic conditions were comparable to those under room temperature conditions.

ACKNOWLEDGMENT

This research was supported by the Nuclear R&D Program and Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology.

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Manuscript Received: February 15, 2012
Revised: February 29, 2012
Revision Accepted: March 5, 2012